SIGNIFICANCE OF TESTS AND PROPERTIES OF Concrete & Concrete-Making Materials

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Significance of Tests and Properties of Concrete and Concrete-Making Materials

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Foreword

THIS PUBLICATION is a revision and expansion of *Significance of Tests and Properties of Concrete and Concrete-Making Materials (STP 169C)* published in 1994. That publication in turn replaced editions published in 1956, 1966, and 1978. The present publication includes a number of new materials and test methods that have been developed, or materials that have increased in importance since the 1994 edition. Two most useful additions are the chapters on slag as a cementitious material and self-consolidating concrete.

As in the previous editions, chapters have been authored by individuals selected on the basis of their knowledge of their subject areas, and in most cases because of their participation in the development of pertinent specifications and test methods by ASTM Committee C09 on Concrete and Concrete Aggregates and, in some cases, ASTM Committee C01 on Cement. The authors developed their chapters in conformance with general guidelines only. Each chapter has been reviewed and, where necessary, coordinated with chapters in which overlap of subject matter might occur.

This latest edition has been developed under the direction of the Executive Committee of ASTM Committee C09 by coeditors Joseph F. Lamond, Consulting Engineer, and James H. Pielert, Consultant, both members of Committee C09.

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Introduction

Joseph F. Lamond[1] *and James H. Pielert*[2]

***ASTM STP 169C, SIGNIFICANCE OF TESTS AND** Properties of Concrete and Concrete-Making Materials,* was published in 1994. ASTM Committee C9 on Concrete and Concrete Aggregates has once again decided the time was appropriate to update and revise this useful publication to reflect changes in the technology of concrete and concrete-making materials that have taken place since that time. New materials have appeared on the scene, along with a greater appreciation of the capabilities of concrete as a basic construction material. Committee C9 and its subcommittees have made significant changes in many of its specifications and test methods to reflect these changes. New specifications and testing techniques have been developed to provide for informed use of new materials and new uses for concrete.

Hydraulic cement concrete is a product composed of many materials and produced in many forms. The quality of concrete is dependent on the quality of the constituent materials and related manufacturing, testing, and installation processes. Since 1914, ASTM Committee C9 has played a vital role in promoting the quality of concrete by developing specifications, testing methods, and practices for concrete and concrete-making materials. This has been possible through the dedication and commitment of its volunteer members over the years.

Committee C9 first published *Report on Significance of Tests of Concrete and Concrete Aggregates, ASTM STP 22*, in 1935, with an updated report published in 1943. *ASTM STP 169* was published in 1956, followed by *ASTM STP 169A* in 1966, *ASTM STP 169B* in 1978, and *ASTM STP 169C* in 1994.

Following this brief introduction, this special publication is organized into six parts: General, Freshly Mixed Concrete, Hardened Concrete, Concrete Aggregates, Concrete-Making Materials Other than Aggregates, and Specialized Concretes, with revised and new chapters.

In Part I, the chapters consist of general subjects on the nature of concrete, sampling, variability, and testing laboratories. A new chapter deals with modeling cement and concrete properties.

Part II deals with the properties of freshly mixed concrete.

Part III concerns itself with the properties of hardened concrete.

Part IV deals with concrete aggregates. The order of the chapters has been revised. They are now presented in the order that most concerns concrete users: grading, density, soundness, degradation resistance, petrographic examination, reactivity, and thermal properties. Some of the chapter titles have changed and the previous chapter on pore systems has been included in the chapter on density.

Part V includes materials other than aggregates. The title of the chapter on curing materials was changed to reflect current technology of materials applied to new concrete surfaces. The chapter on mineral admixtures has been separated into two chapters, one on supplementary cementitious materials and the other on ground slag.

Part VI, on specialized concretes, contains one new chapter on self-consolidating concrete. The subcommittee structure of Committee C9 has been modified to accommodate this need.

The editors, along with ASTM Committee C9 on Concrete and Concrete Aggregates, believe this new edition will serve the concrete industry well. The editors selected authors and their chapters were reviewed in accordance with ASTM's peer review procedures. C9 subcommittees having jurisdiction over the subjects for pertinent chapters participated informally in the review process. The editors appreciate the help and guidance of these people and the cooperation of ASTM Committee C1 on Cement in providing authors for the two chapters on cement. Some of the authors in *ASTM 169C* are no longer active in Committee C9. The co-editors and Committee C9 members wish to dedicate this edition to those authors who have died since *ASTM STP 169C* was published. They are Paul Klieger, Ed Abdur-Nur, Bill Dolch, Jack Scanlon, Bob Philleo, Bill DePuy, Bryant Mather, Ron Mills, and Owen Brown.

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PART I
General

2

The Nature of Concrete

Richard A. Helmuth[1] *and Rachel J. Detwiler*[2]

Preface

T. C. POWERS AUTHORED THE FIRST VERSION OF this chapter, which was published in *ASTM STP 169A* in 1966. His chapter was reprinted without revision in *ASTM STP 169B* in 1978. In *ASTM STP 169C* (1994), Richard A. Helmuth condensed some of that work and included more recent material. The present version relies on the framework established by the earlier authors, while updating and adding to it.

Introduction

For thousands of years, mankind has explored the versatility of materials that can be molded or cast while in a plastic state and then hardened into strong, durable products [1]. As with ceramics and gypsum plasters, lime mortars and pozzolanic concretes provided engineers with economical materials for production of diverse utilitarian and aesthetically pleasing structures. Modern concretes preserve these ancient virtues while greatly extending the range of technically achievable goals.

Concrete-Making Materials—Definitions

Concrete is defined in ASTM Terminology Relating to Concrete and Concrete Aggregates (C 125) as "a composite material that consists essentially of a binding medium within which are embedded particles or fragments of aggregate; in hydraulic-cement concrete, the binder is formed from a mixture of hydraulic cement and water." Hydraulic-cement concretes are those most widely used in the United States and worldwide. Hydraulic cement is defined in ASTM Terminology Related to Hydraulic Cement (C 219) as "a cement that sets and hardens by chemical interaction with water and that is capable of doing so under water." Portland cement is the most important hydraulic cement. It is produced by pulverizing portland cement clinker, consisting essentially of hydraulic calcium silicates, usually by intergrinding with small amounts of calcium sulfate compounds to control reaction rates. It may be used in combination with one or more supplementary cementitious materials, such as fly ash, ground granulated blast furnace slag (referred to as "slag" in the remainder of this chapter), silica fume, or calcined clay.

Aggregate is defined in ASTM C 125 as "granular material, such as sand, gravel, crushed stone, or iron blast-furnace slag, used with a cementing medium to form hydraulic-cement concrete or mortar." Detailed descriptions of these and other materials for making concrete and their effects on concrete properties are given in other chapters in this work.

Typical hydraulic-cement concretes have volume fractions of aggregate that range approximately from 0.7 to 0.8. The remaining volume is occupied initially by a matrix of fresh cement paste consisting of water, cementitious materials, and chemical admixtures, that also encloses air voids. While the aggregates occupy most of the volume, they are relatively inert and intended to be stable. It is the cement paste matrix that undergoes the remarkable transformation from nearly-fluid paste to rock-hard solid, transforms plastic concrete into an apparent monolith, and controls many important engineering properties of hardened concretes.

Scope

Hydraulic-cement concretes may be designed to provide properties required for widely varying applications at low life-cycle cost. If not properly designed or produced, or if exposed to service conditions not understood or unanticipated, premature failures may result. Successful use depends on understanding the nature of concrete.

The scope of this examination of the materials science of concrete is mainly confined to concretes made with portland cements, with or without supplementary cementitious materials and chemical admixtures. The focus is mainly on how we understand concrete performance in ordinary construction practice. That understanding is based on knowledge of its constituents, and their physical and chemical interactions in different environments.

Freshly-Mixed Cement Paste and Concrete

Water in Concrete

The properties of fresh cement paste and concrete depend on the structure and properties of ordinary water, which are unusual for a substance of such low molecular weight. Each molecule has a permanent dipole moment, which contributes to the strong forces of attraction between water molecules and results in unusually high melting and boiling points, heats of fusion and vaporization, viscosity, and surface tension [2].

In addition to dipole interactions, hydrogen bonding between water molecules and thermal agitation affect the structure of water and aqueous solutions. Hydrogen bonding favors formation of clusters of molecules, while thermal

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agitation, including translational, rotational, and vibrational motions, tends to disrupt the structure.

In the liquid state, the molecules are easily oriented in an electric field so that water has a high dielectric constant (78.6 at 25°C). This orientation, as well as molecular polarization, means that the electric field strength and the forces between charged particles, such as ions in solution, are reduced to 1/78.6 relative to that in vacuum (or air). Because of its exceptionally high dielectric constant, water is an excellent solvent for salts: the energy of separation of two ions in solution is an inverse function of the dielectric constant of the solvent. Ions in solution are not separate entities but are surrounded by water molecules attracted to them by ion-dipole forces.

A few minutes after mixing begins, about half of the cement alkalies are dissolved so that the concentration of the alkali and hydroxide ions may commonly be 0.1 to 0.4 mol/L, depending mainly on the water-to-cement ratio and the cement alkali content [3]. At 0.3 mol/L, each ion would be separated from like ions, on the average, by about 1.7 nm, or about five water molecules.

Interparticle Forces

Atoms near the surface of solids are distorted and shifted relative to their positions in the interior because of the unsatisfied atomic bonds at the surface. These distortions of the surface produce net positive or negative surface charge, and elastic excess surface free energy. In aqueous solutions, solid surfaces may preferentially adsorb certain ions [4]. Particles with surface charges of the same sign repel each other in suspensions and tend to remain dispersed. Particles of opposite sign attract each other and flocculate [5].

In addition to these electrostatic forces, which can be attractive as well as repulsive, there are forces among adjacent surfaces of solids, atoms, and molecules that are always attractive. These van der Waals, or dispersion, forces exist because even neutral bodies constitute systems of oscillating charges that induce polarization and oscillating dipole interactions [5]. The combined action of the different forces causes sorption of water molecules and ions from solution, which can neutralize surface charge and establish separation distances of minimum potential energy between solid particles [6]. The mechanical properties of fresh and hardened cement pastes and concretes depend on these forces.

Structure of Fresh Cement Paste

Modern portland cements have mass median particle sizes that are about 12 to 15 μm (diameter of an equivalent sphere), almost all particles being smaller than 45 μm, and very little of the cement being finer than 0.5 μm. During grinding, calcium sulfates grind faster and usually become much finer than the clinker. After mixing with water, the solid surfaces become covered by adsorbed ions and oriented water molecules forming a layer of solution of different composition and properties from those of the bulk aqueous phase; the layer extends out to a distance at least several times the diameter of a water molecule. These surface layers have the effects of simultaneously separating and weakly binding the particles into a flocculated structure.

In fresh cement pastes and concretes made with high doses of water-reducing admixtures, cement particles may become almost completely dispersed (deflocculated) because large organic molecules are adsorbed on their surfaces, displacing water films, and greatly reducing attractive forces between cement particles. Supplementary cementitious materials that contain small percentages of ultrafine (submicron) particles may also aid in dispersing cement particles by adsorption of the ultrafine particles on the surfaces of the larger particles. This specific kind of fine-particle effect is responsible for the improved flow of many portland cement/fly ash mixtures [7,8].

The average thickness of films of water separating dispersed particles in the paste depends on the water-to-cement ratio (w/c) and the cement fineness. A first approximation of the average thickness of these films is given by the hydraulic radius: the volume of water divided by the specific surface. If it is assumed that the films are thin compared with the particle sizes, the calculated thickness is 1.2 μm for cement of specific surface of 430 m^2/kg, mixed at 0.5 w/c [9]. Since the assumption is not valid for the finer fractions and much of the fine fraction in portland cement is composed of calcium sulfates and other phases that dissolve within minutes after mixing begins, the average film thickness for the larger particles in that paste is probably about 2 μm. For flocculated particles, the films are much thinner between adjacent particles, so that much of the water is forced into relatively large cavities or capillary-like channels.

Cement Hydration and Structure Formation

Early Hydration Reactions

It is convenient to divide the process of cement hydration into the early (within the first 3 h), middle, and late (after 24 h) periods. Soon after mixing cement with water, a gel layer forms on the surfaces of the cement grains. Taylor [10] characterized this layer as ". . . probably amorphous, colloidal and rich in alumina and silica, but also containing significant amounts of calcium and sulfate . . ." Within about ten minutes, stubby rods of calcium aluminoferrite trisulfate hydrate (AFt) begin to form. They appear to nucleate in the solution and on the outer surface of the gel layer.

During the middle period of hydration approximately 30 % of the cement reacts. The rapid formation of calcium silicate hydrate (C-S-H) and calcium hydroxide (CH) is accompanied by significant evolution of heat. The CH forms massive crystals in the originally water-filled space. The C-S-H forms a thickening layer around the cement grains. As the shells grow outward, they begin to coalesce at about 12 h, a time coinciding with the maximum rate of heat evolution (Fig. 1) and corresponding approximately to the completion of setting. The shells are apparently sufficiently porous to allow the passage of water in and dissolved cement minerals out. A gap begins to appear between the hydration shell and the surface of the cement grain. Toward the end of the middle period the growth of AFt crystals resumes; however, this time they are distinctly more acicular in shape. Their formation coincides with a shoulder on the heat evolution curve [10].

Like most chemical reactions, cement hydration proceeds more rapidly with increasing temperature. Verbeck and Helmuth [11] postulated that because of the low solubility and low diffusivity, the ions forming the cement hydration products would not have time to diffuse any significant distance from the cement grain, thus forming a highly nonuniform distribution of solid phases. They believed that the dense hydration shells would serve as diffusion barriers, hindering further hydration. A consequence of the uneven distribution of the solid phases is a coarser pore structure. Skalny and Odler [12] found that C_3S pastes of a given w/c hydrated at temperatures of 50 to 100°C had a coarser structure and greater volume of large pores than

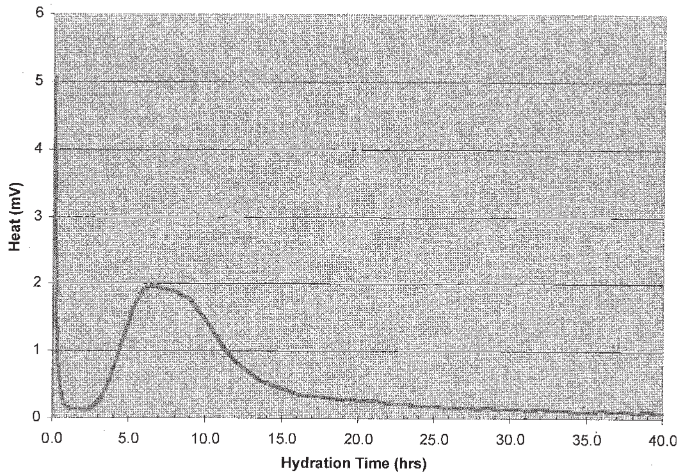

Fig. 1—Heat evolution of Type I/II portland cement paste as measured by conduction calorimetry. The first heat peak is associated with the initial hydrolysis of the C3S, the hydration of the C3A in the presence of gypsum to form ettringite, and rehydration of the hemihydrate to form gypsum. It is normally completed within 15 min. Deposition of ettringite on the surface hinders further hydration of the C3A. The first peak is followed by a dormant period of 2 to 4 h, during which the paste remains in the plastic state as the C3S continues to dissolve slowly. The acceleration period begins with the renewed evolution of heat (beginning of the second peak) as the initial hydration products of the C3S begin to form. Initial set coincides with the beginning of the acceleration period. The CH crystallizes from solution, while the C-S-H deposits on the surface of the C3S, creating a diffusion barrier. Eventually the rate of hydration slows due to the difficulty of diffusion of water and ions through this barrier [20]. Final set takes place just before the maximum point of the second peak. The "shoulder" of the second peak, which in this figure appears at about 9 h, is associated with the renewed formation of ettringite [10]. Further hydration of the cement continues at a much slower rate, asymptotically approaching 100 % [20]. (Image courtesy of E. Shkolnik.)

those hydrated at 25°C. Kjellsen et al. [13] found that in cement paste hydrated at 50°C to a degree of hydration of approximately 30 % the hydration shells were already sufficiently dense to act as diffusion barriers to sulfate ions. The morphology of the CH crystals was dependent on the hydration temperature, being more elongated in cement paste hydrated at 5°C and more compact in cement paste hydrated at 50°C.

During the first hours of hydration, very fine supplementary cementitious materials such as silica fume and fine fly ash have some important physical effects on the development of the microstructure. According to Bache [14], the principal physical effect of silica fume in concrete is an enhancement of particle packing because the silica fume particles can fit into spaces between cement grains in the same way that sand fills the interstices between particles of coarse aggregate and cement grains fill the interstices between sand grains. This analogy is valid only when there is sufficient superplasticizer to counteract the effects of attractive surface forces. As the cement begins to hydrate, the fine particles of silica fume or fly ash serve as nucleation sites for the hydration products. Asaga et al. [15] found that fly ash, silica fume, and slag all increase the rate of early hydration of C_3S, even though the supplementary cementitious materials do not themselves react at this stage.

Before setting, two, and sometimes three, kinds of volume changes occur. Sedimentation causes subsidence of the floc structure and collection of bleeding water on the top surface, if evaporation is not excessive. If the surface becomes partly dried, capillary tension in the water can cause plastic shrinkage and cracks. Chemical shrinkage is the volume change that results from formation of hydrates that have less solid volume than the volume of water and solids reacted. While the paste is plastic, the entire volume of paste undergoes chemical shrinkage. After setting, the external dimensions remain essentially fixed and additional water must be imbibed to keep the pores saturated with water. If sufficient water is not imbibed, the paste undergoes self-desiccation.

Autogenous shrinkage is the volume change that results when there is no moisture loss to the surrounding environment. It is most significant for concrete in which the water-to-cement ratio is less than about 0.42. Before the concrete sets, autogenous shrinkage is equivalent to chemical shrinkage and manifests itself as plastic settlement. Once a continuous structure begins to form, the chemical shrinkage is restrained to some degree. As the internal structure of the cement paste matrix becomes more rigid, autogenous shrinkage is influenced less by chemical shrinkage and more by self-desiccation [16]. Tazawa and Miyazawa [17] found that the amount of autogenous shrinkage increases with increasing C_3A and C_4AF contents of the cement and decreasing water-to-cement ratio. Bentz and Geiker [18] found that the effects of self-desiccation, and thus the autogenous shrinkage, can be mitigated by the use of water-saturated low-density fine aggregates or of superabsorbent polymer particles to provide an internal source of water, or by the use of shrinkage-reducing admixtures.

Gypsum and other sulfate-bearing materials are normally interground with portland cement clinker in the production of cement to control the hydration of the aluminates. If little or no gypsum is present, the cement experiences "flash set" or "quick set," in which the cement sets rapidly with much evolution of heat. Plasticity is not regained with further mixing, and the subsequent development of strength is poor. It is associated with the rapid hydration of the aluminate and ferrite phases to form plates of low-sulfate AFm phases such as C_4AH_{13} and C_4AH_{19} throughout the paste. Some cements low in aluminate phase do not flash set even without gypsum [10].

Flash set and quick set may be caused by insufficient sulfates in the cement, or by the presence of the wrong form(s) of sulfate. During milling, gypsum can dehydrate to form hemihydrate or so-called "soluble anhydrite" (γ-$CaSO_4$). Some byproducts from various industrial processes contain calcium sulfite ($CaSO_3$), known as hannebachite, rather than gypsum, and are much less soluble than gypsum. Byproduct gypsum from flue gas desulfurization is often difficult to dispense uniformly due to its high moisture content; thus the quantity of gypsum in the cement can be variable. The form of sulfate has a direct bearing on the availability of sulfates in solution at the right time, since the solubility and rate of solution vary considerably from one form to another. Very finely ground cement requires more sulfate to control the reactivity of the aluminates.

Incompatibility among the various concrete ingredients may also contribute to flash set or quick set. Lignosulfonates in water-reducing admixtures limit the solubility of sulfate and calcium ions. Triethanolamine (TEA) in water-reducing admixtures makes the aluminate phases react faster while retarding the hydration of the calcium silicates. Some Class C fly ashes

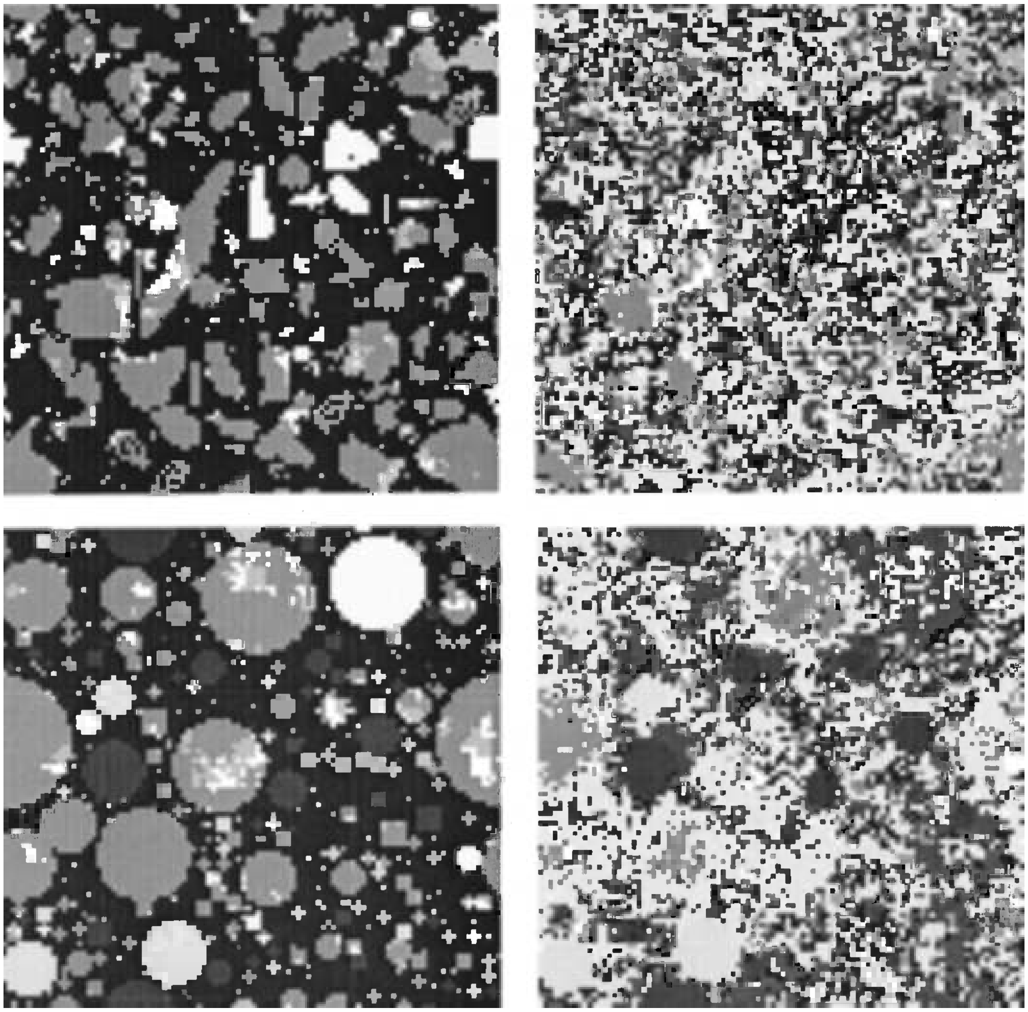

Fig. 2—Computer-based model of the development of microstructure in hydrating portland cement pastes: (upper left) portland cement paste before hydration begins; (upper right) portland cement paste after 47 % hydration; (lower left) unhydrated portland cement paste with fly ash; (lower right) partially hydrated portland cement paste with fly ash [images courtesy of E. J. Garboczi]. All images 100 μm × 100 μm.

contain sufficient quantities of reactive aluminate phases to upset the balance between aluminates and sulfates unless additional gypsum is added to the system. If either the cement or the fly ash has a high alkali content, it will increase the rate of hydration. Hot weather accelerates the hydration of the aluminates and reduces the solubility of the sulfates [19].

Another type of rapid set is called "false set," in which little heat is evolved. False set is associated with the rehydration of calcium sulfate hemihydrate to form secondary gypsum. The interlocking and intergrowth of the gypsum crystals results in the stiffening observed. If the quantity of secondary gypsum is not too great, it redissolves on further mixing, and the hydration reactions proceed normally. During milling, gypsum can dehydrate to hemihydrate; hemihydrate is sometimes added directly to the clinker when a more reactive form of sulfate is desired [10]. Grinding at a higher temperature or a lower relative humidity can often achieve the same result.

Hardening Reactions and Microstructure

After the first 24 h, the hydration shells surrounding the cement grains become less permeable, and C-S-H begins to form inside them. The availability of sulfate ions is very limited within the hydration shells, and calcium aluminoferrite monosulfate hydrate (AFm) forms, replacing AFt as the aluminate phases continue to hydrate. After the spaces between the hydration shells and the cement grains fill with hydration products, further hydration is slow and appears to occur by a topochemical rather than a through-solution mechanism [10].

Larger clinker particles in cement hydrate partly by dissolution and partly by in situ reactions so that a pseudomorph of inner products is formed within the boundaries of the original grain. The depth of reaction increases with time, but at decreasing rates so that large particles may have unhydrated cores even after years of moist curing. The dissolved portion forms outer products in the water-filled space near the grains. The calcium silicates produce crystalline calcium hydroxide and nearly amorphous calcium silicate hydrate (C-S-H gel) that engulf crystalline phases formed by the early reactions. Capillary pores remaining in mature cement paste increase in size with water-to-cement ratio and have diameters ranging from 10 nm to 10 μm [20].

Powers designated the hydration products of portland cements as "cement gel," recognizing that they contained both C-S-H gel and crystalline products, and micropores [21]. He found that typical cement gels had minimum porosities of about 30 %, and specific surface areas of about 200 m^2/g, as calculated by Brunauer, Emmett, Teller (BET) theory [1] from water-vapor adsorption data obtained after first drying to remove all of the evaporable water. These studies also showed that at 0.38 water-to-cement ratio all of the capillary pore space was just filled by maximum density gel when all the cement was hydrated. Mixtures made with water-to-cement ratios less than 0.38 cannot be completely hydrated; the amount of cement that can hydrate is less because hydration virtually stops when the capillary space is filled with gel of minimum porosity. Saturated, fully hydrated cement pastes made at water-to-cement ratios above 0.38 have remaining capillary pore space (by definition) equal to the excess above 0.38. Partially hydrated mixtures have proportionately less gel and more capillary space. Cements of different compositions behave similarly, with similar values for the constants.

Supplementary cementitious materials can alter the developing microstructure of hydrating cement paste by means of the pozzolanic reaction, in which silica reacts with CH and water to form additional C-S-H. The composition of the C-S-H formed by pozzolanic reaction differs somewhat from that of C-S-H formed by hydration of cement in that the former has a lower Ca/Si ratio and a higher Al/Ca ratio [10]. In the hydration of cement pastes containing slag, Al substitutes for Si in the silicate chains [22]. With any supplementary cementitious material, the pozzolanic reaction improves both the later-age strength and the durability of concrete because it consumes the relatively weak, soluble CH and generates more C-S-H, which further subdivides the larger pores and increases the strength of the cement paste. The microstructure of cement pastes hydrated at elevated temperatures is marked by uneven distribution of hydration products and, consequently, a coarser pore system. Goto and Roy found that the total porosities of pastes hydrated at 60°C were greater than those of comparable pastes hydrated at 27°C for the same length of time. They attributed the difference in porosity largely to the difference in volume of pores of radius 75–230 nm [23]. It is the larger pores that contribute most to the permeability; Mehta and Manmohan proposed a pore radius of 50 nm as a somewhat arbitrary dividing point between "large" pores which contribute most to permeability and "small" pores which are much less significant [24]. For cement pastes hydrated at low temperatures, the hydration products are more evenly distributed and the pores fine and discontinuous. Hydration shells around the cement grains are not apparent. For cement pastes hydrated at elevated temperatures, dense hydration shells are readily apparent and the pores are coarse and interconnected [13].

Supplementary cementitious materials mitigate the deleterious effects of elevated-temperature curing. Cao and Detwiler [25] found that both silica fume and slag were effective in homogenizing the distribution of the hydrates of cement pastes cured at 70°C. While the total pore volume remained essentially the same for pastes with and without silica fume or slag, the average pore size was significantly reduced.

Drying and rewetting alter the microstructure, and different adsorbates measure different surface areas. The sheet-like crystallites are imperfectly stacked and separated by interlayer-adsorbed water at relative humidities down to 11 %. Before drying or aging, cement gels have specific surfaces of C-S-H monomolecular sheets, about 700 m^2/g, as measured by small-angle X-ray scattering [26]. Because of the large internal surface area, the distances between solid surfaces of the pores in the gel approach the size of water molecules; most of the gel water is close to the surfaces. In such systems, it is not certain how much of the volume change of chemical shrinkage should be attributed to the reaction itself, and how much to the possible change of density of water in pores as it is adsorbed on newly created surfaces. If it is assumed [27] that the adsorbed solution has the same density as that in large pores, the apparent specific volumes of the nonevaporable (hydrate) water and solids were found to be 0.74 and 0.398 cm^3/g, respectively, and the minimum porosity of the gel to be 30 %. The amount of chemical shrinkage is expressed in terms of the change in the apparent specific volume of the reacted water, from 0.99 to 0.74 cm^3/g, and the amount, w_n, of nonevaporable water: 0.25 w_n.

Hydration of each unit volume of cement produces about 2.2 volumes of gel. This value does not depend on the assumption concerning specific volumes. Although chemical shrinkage slightly reduces the space filling by solid hydrates, cement gel is an even more effective filler of the capillary space than the solid hydrates because of the 30 % porosity of the gel.

Effects of Drying

Loss of moisture due to self-desiccation or evaporation partially empties the largest capillaries at exposed surfaces. Adsorbed water remains on capillary walls as concave menisci form and progress into smaller interconnected pores. Meniscus curvature and capillary tension in the remaining water are increased as the relative humidity is decreased down to about 45 % RH, below which sorption effects prevail. Reductions in relative humidity slow the hydration rate; at 80 % RH, hydration is insignificant. Drying causes shrinkage of hardened cement paste and major alterations of the gel microstructure. Shrinkage and stabilization of cement paste by drying are complex and partially irreversible processes involving capillary, sorption, and dehydration effects.

Capillary tension in the pore water increases as the relative humidity decreases below the vapor pressure of the pore solution. For dilute solutions, tensions increase to about 97 MPa (14 000 psi) at 50 % RH. At lower relative humidities, 40 to 45 % RH, the tension exceeds the cohesive strength of water in capillaries and menisci can no longer exist [21]. Above 45 % RH, capillary tension in the water must be balanced by compressive stresses in the solid structure, in which stress concentrations can produce irreversible effects. When the pores are nearly water-filled, the average stress is that produced by surface tension in the cross-sectional area that is pore water; the resulting strain in the solid structure is the beginning of the drying shrinkage. As the capillaries empty the cross sections of the remaining water-filled pores are smaller, but the capillary tension increases and causes local collapse of less dense regions of the outer product, and enlargement of large pores. Desorption causes shrinkage both by permitting solids to come together, and by increasing solid surface tension. Well-crystallized AFm and AFt hydrated phases also dehydrate, decreasing lattice spacings, so that elastic restraint of the shrinking C-S-H gel is reduced.

Cement paste cured for six months before drying at 47 % RH for the first time shows both reversible and irreversible water loss and shrinkage [28]. Increased drying time causes increased water loss, shrinkage, and greatly reduced internal surface area. Rewetting causes sorption and swelling that only

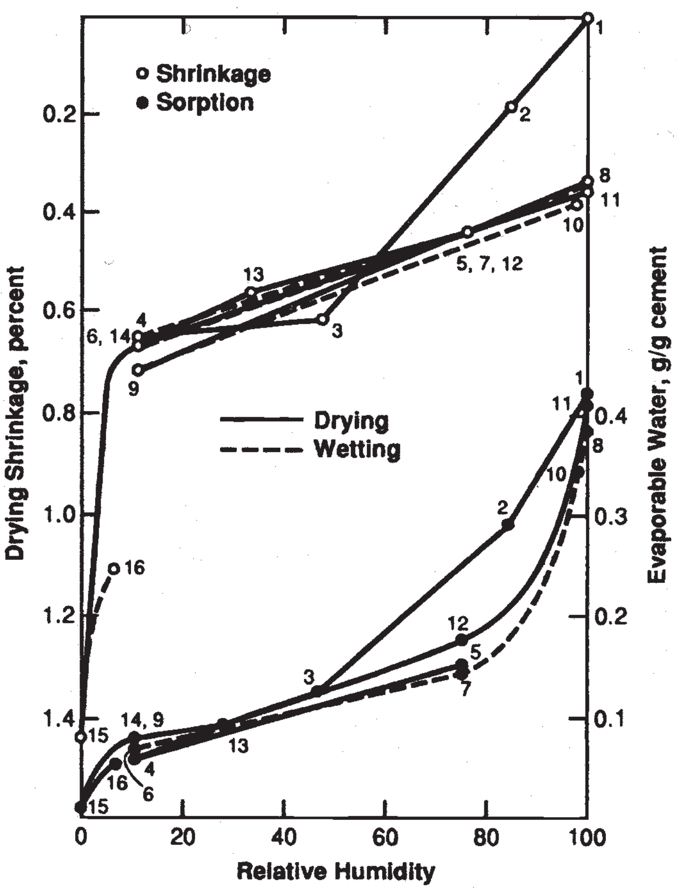

Fig. 3—Drying shrinkage, rewetting swelling, and evaporable water content of a 0.6 water-to-cement ratio hardened portland cement paste. Numbers indicate sequence of measurements [28]. Reprinted with permission of the Portland Cement Association.

partly reverses the first water loss and shrinkage (Fig. 3). The irreversible component of shrinkage increases with water-cement ratio (0.4 to 0.6) from 0.2 to 0.4 %, whereas the reversible component (after stabilization by drying) is only 0.2 % and nearly independent of porosity. Even without drying, long-term aging in moist conditions causes age stabilization at water-cement ratios above 0.4 so that even the irreversible shrinkage tends to become porosity-independent at about 0.2 %. The irreversible shrinkage volume is only about half of the volume of the irreversibly lost water, if we assume its specific volume to be 0.99, which suggests that some pores emptied during drying become closed off and are not accessible during rewetting [29], or have reduced capacity. Below 11 % RH, loss of interlayer water is accompanied by large partially irreversible shrinkage and water loss effects [30].

Cement Paste Structure—Property Relationships

Rheology of Fresh Cement Paste

When cements are mixed with sufficient water and sufficient intensity, dry agglomerates of fine particles are first dispersed and then tend to form a floc structure, which is continuously broken down by mixing if the early reactions are well controlled. When mixing is stopped, the floc structure reforms until it becomes an essentially continuous floc. Cement pastes in this condition are actually weak solids with measurable shear stress yield values that depend on water-to-cement ratio, cement fineness, and other factors. Typical values for portland cement pastes without admixtures range roughly from 10 to 100 Pa (0.0014 to 0.014 psi) for w/c from 0.6 to 0.35 [9]. Prior to yield, the pastes are elastic and shear deformations can reach about 20 deg, indicating a rubber-like elasticity.[3] They are also plastic solids with typical values for plastic viscosity that range roughly from 10 to 100 mPa · s (centipoise). High water-to-cement ratio pastes seem to be liquid and may be poured easily because their yield stress values are so low. At low water-to-cement ratios, the pastes are obviously plastic and can be deformed by moderate forces. Standard test pastes made at normal consistency have w/c about 0.25 and yield stress values of about 2000 Pa (0.29 psi) [9].

At ordinary temperatures, portland cement hydration reactions cause progressive stiffening and setting during the first few hours. Yield stress values increase to 2×10^4 Pa (2.9 psi) at initial set and 1×10^5 Pa (14 psi) at final set [9].

Increasing the water-to-cement ratio of a cement paste reduces both the yield stress and the plastic viscosity. Water reducers and superplasticizers increase the dispersion of the agglomerated particles of cementitious materials by adsorption on the particle surfaces, thus reducing the amount of water needed to produce a given flow. Taylor [10] discussed three mechanisms by which these admixtures cause dispersion: (1) an increase in the zeta-potential between the first adsorbed layer and the bulk solution, so that like surface charges of sufficient magnitude cause the particles to repel one another; (2) an increase in the solid-liquid affinity, so that the particles are more attracted to the mix water than to one another; and (3) steric hindrance, the oriented adsorption of a nonionic polymer that weakens the attraction between particles. Thus the addition of water reducers and superplasticizers reduces the yield stress of the fresh paste. The addition of air-entraining admixture, on the other hand, stabilizes the air-water interface of the air voids [1] and reduces the surface potential of the particles of cementitious material, reducing the plastic viscosity of the paste [31]. In the presence of sufficient superplasticizer to overcome the effects of surface forces, small quantities of silica fume (less than approximately 5 % by mass of cement) reduce the plastic viscosity; larger dosages of silica fume increase both yield stress and plastic viscosity, making the paste sticky [31]. The effect at small dosages was attributed by Bache [14] to the displacement of water due to more efficient packing of the particles when minute particles of silica fume can fill the interstices between cement grains; at larger dosages, the particle packing is no longer efficient. Another possibility is that, like the finest fraction of fly ash particles, the silica fume particles adhere to the surfaces of the cement grains, causing the cement grains to disperse [7].

Elasticity and Creep

Hardened cement pastes are not perfectly elastic, but are viscoelastic solids. Internal friction, creep, and stress relaxation are useful in dissipating vibrational energy, and preventing excessive stress concentrations in concrete. They are a result of redistribution of moisture, viscous flow of gel, and dissolution of solids under stress and recrystallization in pores. These processes, and slow growth of cracks, are thermally activated

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[3] Unpublished work done at Construction Technology Laboratories for the Portland Cement Association under Project HR 7190.

processes in which random thermal motions provide sufficient energy, in addition to the applied stress, at sites of adsorbed water molecules, or solid-solid bonds, to exceed the bond energy. Short-term loading tests of water-saturated cement paste show that creep and creep recovery versus time curves are bimodal and consist of a component with retardation times ranging from 0.2 to 2 s and a slower component that ranges over weeks. The short-time component was associated with redistribution of water in capillary pores [32]. Diffusion of strongly adsorbed and hydrate water, recrystallization, and other irreversible changes are believed to contribute to the slower processes. Long-term creep of cement paste can be several times the elastic deformation.

Elastic moduli can be measured precisely by dynamic methods and are found to vary with porosity, ε, according to

$$E = E_0(1 - \varepsilon)^3$$

in which E_0 is the modulus at zero porosity [33]. If the capillary porosity is used, E_0 is the modulus of the cement gel, about 34 GPa (5×10^6 psi) for Young's modulus of water-saturated pastes. If the total porosity (including that of the gel) is used, E_0 is an average modulus for the solids, about 76 GPa (11×10^6 psi). Equations of the same form apply for the shear and bulk moduli. Drying significantly reduces Poisson's ratio, from about 0.3 to 0.18, and the bulk modulus; stresses are carried by at least some of the water in pores.

Elastic moduli of saturated pastes increase moderately as the temperature is decreased to 0°C. At temperatures in the freezing range down to about −60°C, ice formation in capillaries increases the moduli as ice contents increase. At still lower temperatures, the moduli increase more rapidly; internal friction reaches a peak at about −90°C as the gel water viscosity increases as it approaches its glass transition temperature at −119°C [34]. The gel water (adsorbed solution) does not freeze to ice, but becomes a glassy solid.

Compressive Strength

The fraction, X, of the available space that is filled by cement gel at any stage of hydration is called the gel/space ratio. It can be calculated from the water-to-cement ratio, the fraction of the cement that has hydrated, and the volume of gel produced. For fully hydrated cement pastes, it may be expressed as $(1 - \varepsilon)$, where ε is the capillary porosity. Compressive strength, f_c, at different water-to-cement ratios and ages can be simply expressed as

$$f_c = f_{cg}X^n$$

in which f_{cg} is the intrinsic strength of the gel (at $X = 1$), and n has a value of about 3. Use of this equation by Powers indicated intrinsic strengths of cement gels ranging from 90 to 130 MPa (13 000 to 18 500 psi) in mortars made with five different cements [21]. However, mortars probably do not provide accurate measures of intrinsic strengths of pastes because of transition zones at aggregate surfaces. Tests of cement pastes yielded higher strengths at gel/space ratios calculated to be equal to those of mortars made with the same cement.

Testing of pastes made with both normally ground and controlled-particle-size-distribution portland cements has shown that intrinsic strengths of the gel do not depend on cement particle size distributions over the range investigated, although rates of strength development do. However, paste strengths at several ages defined different straight lines for each water-to-cement ratio when plotted against X^3, and indicated intrinsic strengths of 134 and 97 MPa (19 400 and 14 000 psi) at 0.36 and 0.54 w/c, respectively [35]. This result indicates that the intrinsic strength of the gel formed at water-to-cement ratios above about 0.38 decreases with increasing water-to-cement ratio, in contrast with Powers's mortar data.

If we consider fresh cement pastes to have strengths equal to their yield stress values, typically 10 to 100 Pa, and ultimately harden to compressive strengths of 10 to 100 MPa, the increase is about six orders of magnitude.

Permeability and Diffusivity

The transport properties of cement paste and concrete largely determine its durability in most environments. Water itself may be harmful because of its ability to leach CH from the cement paste, and because ice crystals that grow on freezing generate tensile stresses that may cause cracking [36]. Water may also carry harmful dissolved species such as chlorides or acids into the concrete. Cement and concrete are also permeable to gases such as oxygen and carbon dioxide, which contributes to the corrosion of steel reinforcement. D'Arcy's law describes the flow of water through saturated cement paste

$$\mathrm{d}q/\mathrm{d}t = \mathrm{K}_1 \cdot A \cdot \Delta h/l$$

where $\mathrm{d}q/\mathrm{d}t$ is the flow rate, K_1 is the permeability, A is the cross-sectional area, Δh is the hydraulic head, and l is the thickness of the specimen [37]. Thus water-saturated flow is proportional to hydraulic pressure differences, if corrections are made for osmotic effects; concentration gradients cause osmotic flow to higher concentrations, and diffusion of ions to lower concentrations. When the cement paste is partly dried, relative humidity and moisture gradients cause flow because of capillary tension and diffusion along surfaces and in the vapor phase. The changes of microstructure that cause great changes of elastic moduli and strength of cement pastes during hardening cause reductions of permeability and diffusivity. The presence of supplementary cementitious materials can reduce the permeability by several orders of magnitude as the pozzolanic reaction reduces the continuity of the pore system [10].

Permeability coefficients of fresh portland cement pastes of 0.5 and 0.7 water-to-cement ratio, calculated from bleeding data, range from 5.7 to 20×10^{-5} m^3/(s · m^2 · MPa/m) [6.1 to 22×10^{-4} in.^{3}/(s · in.^{2} · psi/in.)], respectively. These coefficients for hardened pastes of the same water-cement ratios after prolonged moist curing, determined with machined samples, were reduced to ultimate values of 4.5 to 60×10^{-12} m^3/(s · m^2 · MPa/m) [4.8 to 65×10^{-11} in.^{3}/(s · in.^{2} · psi/in.)] [37]. Permeability coefficients of fresh pastes are about ten million times as great as when fully hydrated.

The diffusion of ions in cement paste is described by Fick's second law once steady-state conditions have been reached:

$$\delta c/\delta t = D_c \cdot \delta^2 c/\delta x^2$$

where c is the concentration of the ion at distance x from the surface after time t and D_c is the diffusion coefficient. In practice, the transport of ions may also be driven electrically or by convection (in the case of partial saturation). Diffusion coefficients for Na^+ are on the order of 10^{-11} to 10^{-13} m^2/s and for Cl^- on the order of 10^{-11} to 10^{-12} m^2/s [10]. Diffusion coefficients increase with increasing temperature and water-to-cementitious materials ratio and decrease with degree of hydration.

Specimens dried step-wise to 79 % RH and carefully resaturated so as to avoid cracking had permeability coefficients about 70 times those of comparable specimens that were continuously moist cured. Such changes indicate enlargement of large pores by partial drying.

Thermal Expansion

Coefficients of thermal expansion of concretes are determined mainly by those of their aggregates. However, thermal expansions of cement paste depend strongly on their moisture contents because retention of water by surface forces in the gel decreases as temperatures increase, and vice versa. When cooled without access to additional water, slightly dried cement gel has a linear coefficient of thermal expansion of about $27 \times 10^{-6}/^{\circ}C$. When cement gel is cooled in contact with sufficient capillary water in cement paste or external water, moisture flows into the gel; the resulting expansion (during or after cooling) produces a net coefficient of thermal expansion of about $11.6 \times 10^{-6}/^{\circ}C$ [38]. Mature saturated cement pastes of low (< 0.45) water-to-cement ratio show transient effects caused by the relatively slow movement of moisture from capillary to gel pores during cooling and vice versa during warming. At low relative humidities, coefficients of thermal expansion decrease to about the same value as for saturated pastes. Such differences between coefficients of thermal expansion of pastes and aggregates may cause excessive local stresses in concretes unless relieved by creep.

Concrete Aggregates

The major constituents of ordinary concretes are crushed rocks or gravels used as coarse aggregates and sands used as fine aggregates. Materials used in concrete usually need to be processed to be of proper size grading and relatively free of such deleterious substances as clay, salts, and organic matter.

Specific Gravity and Porosity

It is useful to classify aggregates by specific gravity and porosity into lightweight, ordinary, and heavyweight materials (ASTM C 125). Lightweight aggregates are used to reduce dead loads and stresses, especially in tall structures, and to provide thermal insulation. Heavyweight aggregates are used mainly for radiation shielding. Ordinary aggregates, such as sandstone, quartz, granite, limestone, or dolomite, have specific gravities that range from about 2.2 to 3.0. Densities of ordinary concretes range from about 2.24 to 2.4 Mg/m^3 (140 to 150 lb/ft^3).

Porosity reduces the weight, elastic modulus, thermal conductivity, and strength of aggregates and concretes, although the effect on strength may be significant only in high-strength concrete. Porosity increases permeability to fluids and diffusivity to ions in pore solutions, especially if the pores are open (interconnected) rather than closed. Freezing of water in pores in aggregate particles in moist concrete can cause surface popouts or D-cracking in concrete pavements [20].

Strength of Aggregate Particles

Strength test results of individual samples of rock from any one source show wide variations that are caused by planes of weakness, and their different orientations, in some of the samples. Such weaknesses in the rock samples may not be significant once the rock has been crushed to the sizes used in concrete so that the higher, or at least average, strengths may be more significant. Ten different common types of rock tested at the Bureau of Public Roads had average compressive strengths that ranged from 117 MPa (16 900 psi) for marble to 324 MPa (47 000 psi) for felsite [39]. A good average value for concrete aggregates is about 200 MPa (30 000 psi), but many excellent aggregates range in strength down to 80 MPa (12 000 psi) [40]. These values are generally above strengths of ordinary concretes.

Permeability

Measurements of coefficients of permeability to water of selected small (25-mm (1-in.) diameter) pieces of rock, free of visible imperfections, yielded values several orders of magnitude smaller than for larger specimens, which probably contained flaws [32]. Values for the small specimens ranged from $3.5 \times 10^{-13}\ m^3/(s \cdot m^2 \cdot MPa/m)$ [3.8×10^{-12} in.^{3}/(s · in.^{2} · psi/in.)], for a dense trap rock, to $2.2 \times 10^{-9}\ m^3/(s \cdot m^2 \cdot MPa/m)$ [2.4×10^{-8} in.^{3}/(s · in.^{2} · psi/in.)] for a granite. These values are equal to those measured for mature hardened portland cement pastes made at water-to-cement ratios of 0.38 and 0.71, respectively, despite the low (less than 1 %) porosities of these rocks.

Concrete Proportioning, Structure, and Properties

Proportioning and Consistency

Two basically different kinds of concrete mixtures must be distinguished. Nonplastic mixtures made with relatively small amounts of water show considerable bulking as water is added, and after compaction have sufficient strength to support their own weight. The concrete block industry employs such nonplastic but cohesive mixtures. Void space in such mixtures is relatively high and filled mostly by air. The strength of the cohesive mixture results from capillary tension under menisci bounding the water films on and between the solid particles, and solid surface forces. Strength and bulking increase to a maximum as water is added, and then decrease as the void space nearly fills with water and capillary tension is diminished. With sufficient water, the mixture is wetted so that surface menisci and capillary tension disappear, void contents reach a minimum, and limited plastic deformation becomes possible. The remaining cohesive force is a result of interparticle attraction between closely spaced fine particles. This minimum void space contains about 12 % air when such mixtures are compacted by ordinary means, and the cement content is not below a certain limit. The water content at minimum voids content is called the “basic water content” [6].

The consistency of cement paste at its basic water content is nearly the same as the normal consistency as defined in ASTM standards. Normal consistency pastes, and mortar or concrete mixtures made with different aggregates at their basic water contents, have slump values of about 42 mm (1.7 in.) in the standard test. Such concretes are much stiffer than the plastic mixtures commonly used in American practice that usually contain chemical admixtures and higher water contents. Further additions of water increase void volume, reduce interparticle forces, and increase the capacity for plastic deformation.

The main effect of adding increments of aggregate to paste is to reduce the volume of voids and cement per unit volume. The total effect is not just that of volume displacement, because the cement paste void space is increased by the added aggregate surfaces, as described in the next section. Also, when aggregate is introduced, plastic strains in the paste during compaction are necessarily greater and the mixture is stiffer than the paste. If such additions are begun using cement paste of the standard normal consistency, and if the same compacting force or energy is applied to the mixtures as to the paste, that consistency can be maintained constant if increments of water are added with each increment of aggregate. The ratio of the volume of water plus air to the total volume of solids (voids ratio) decreases with added aggregate, but not as much as without the added water, until a minimum voids content is reached, and then increases.

Consistency of concrete depends on consistency of cement paste as well as on dispersion of aggregate by sufficient paste volume for each particular aggregate. Although concrete yield stress values can be calculated from slump values, there is as yet no valid method of calculation of concrete slumps from paste yield stress values for concretes made with different aggregates and proportions. For fixed proportions, the stiffer the paste, the stiffer the concrete. In mixtures that are relatively rich in cement and paste volume, adding increments of aggregate does not greatly increase water requirements for flow. In leaner mixtures, particularly those with aggregate contents above those at minimum voids ratios, but below those very lean mixtures that require excess amounts of entrapped air, the water requirement is proportional to the volume fraction of aggregate in the total solids [6]. This range comprises much of the concrete made for ordinary use.

Structure

For concrete to possess plasticity, the aggregate must be dispersed by a sufficient volume of cement paste to permit deformation under shear stress. For any aggregate size grading, the minimum voids ratio indicates the volume required to fill the voids in compacted (dry-rodded) aggregate. If the concrete is plastic, it must contain a volume of paste and air above the minimum to disperse the aggregate, that is, to provide some separation between particles that would otherwise be in contact. Fine aggregate disperses coarse aggregate but also reduces average paste film thicknesses. For concretes made with nearly the same voids ratios (about 0.20), at 75 to 100 mm (3 to 4 in.) slump, and different aggregate finenesses, Powers calculated the minimum separation distances between aggregate particles from the excess paste volumes, by two different methods with dissimilar results [6]. The average values by the two different methods ranged from 26 μm to 121 μm for lean to rich mixtures, respectively, the latter having the highest percentage of fine aggregate (43 %) and being close to the minimum voids ratio. Such results indicate that many concretes, especially very lean mixtures, suffer from poor workability because of particle interference to flow by the larger (≥ 30 μm) cement particles. This indication has been confirmed by recent research. Although cement pastes made with cements of 30-μm maximum particle size were stiffer than those made with ordinary cements, improved flow was obtained using cements with controlled particle size distributions in standard mortars and ordinary (not lean) concretes [41]. Particle interference by large particles is also one of the reasons that some fly ashes increase water requirements of concretes [8].

The presence of the aggregates creates an anomaly in the structure of hardened concrete known as the transition zone between the cement paste and the aggregate. Cordon and Gillespie [42] noted that for concrete with a water-to-cement ratio of 0.40, the larger the maximum size of the aggregate the lower the strength of the concrete. They explained these results in terms of the strength of the bond between paste and aggregate: for concrete with a high water-to-cement ratio, failure is controlled by the properties of the paste; as the quality of the paste improves, the strength of the paste-aggregate bond controls the strength of the concrete; with sufficient improvements in the strength of the paste-aggregate bond, the strength of the aggregate becomes the controlling factor. Hadley [43] found that the first hydration product to form on the aggregate surface is a thin film of calcium hydroxide. While this film is still forming, calcium silicate hydrate gel begins to appear on the film. Small crystals of calcium hydroxide begin to form on the surfaces of the cement grains. These crystals also have a preferred orientation parallel to the aggregate interface. With increasing distance from the interface, the orientation of the crystals become more random.

Figure 4 [44] illustrates the microstructural characteristics of the transition zone as compared to the bulk cement paste: higher void content, higher contents of CH and ettringite, reduced content of C-S-H, and larger crystals of CH strongly oriented parallel to the aggregate surface. Factors contributing to the nature of the transition zone include bleeding, which creates pockets of water-filled space beneath aggregate particles; less efficient packing of particles of cementitious materials in the vicinity of a surface (the "wall effect"); and the "one-sided growth effect" of dissolved cementitious materials and hydration products diffusing in from the bulk cement paste (but not from the aggregate) [45]. As the cementitious materials hydrate, the transition zone fills preferentially with hydration products that form through solution (that is, CH and ettringite). Because of the relatively open space, the crystals can grow large.

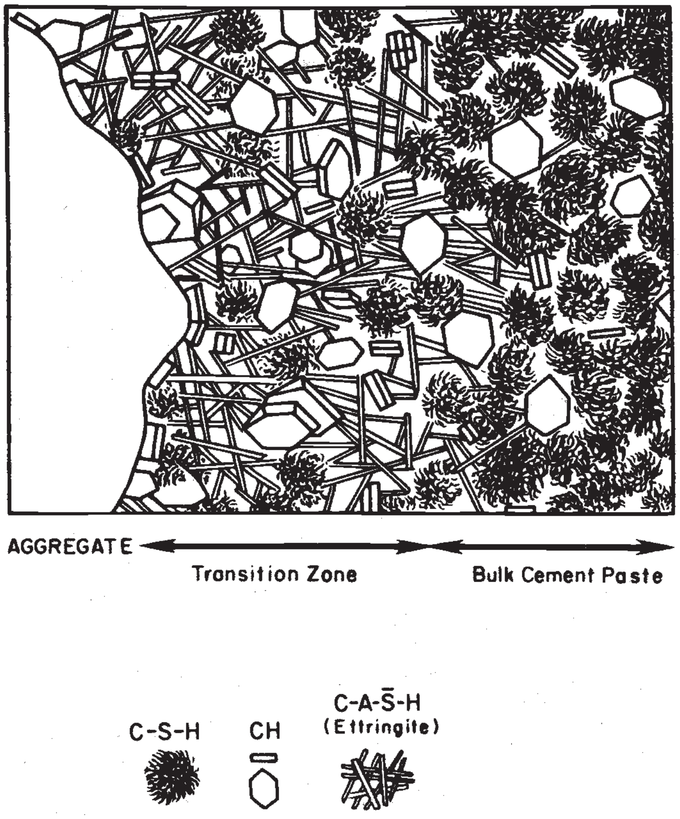

Fig. 4—Representation of the transition zone at a paste/aggregate interface in concrete, showing a more coarsely crystalline and porous microstructure than that of the bulk cement paste [44]. Reprinted with permission of P. K. Mehta.

Mathematical modeling of the microstructure of concrete has improved our understanding of the mechanisms by which the microstructure develops and by which it affects the properties of the concrete. By developing models and verifying them against experimental results, Bentz et al. [45] showed that silica fume particles both reduce the initial thickness of the transition zone and react to convert CH to C-S-H. Thus the transition zone is thinner and more closely resembles the bulk cement paste. Fly ash at the same dosage is less beneficial due to its larger particle size and lower pozzolanic activity; thus it is used at higher dosages.

Because of the greater porosity and the connectivity of the pores in the transition zone, the effect on the transport properties of the concrete is significant, particularly if the individual transition zones interconnect with one another to percolate through the concrete [45]. The structure provides continuous pathways for the transport of fluids or ions. Similarly, the presence of the relatively weak transition zone affects the mechanical properties of concrete. The propagation of cracks preferentially through transition zones reduces both the strength and the modulus of elasticity of concrete.

Properties of Hardened Concrete

Other chapters in this publication give comprehensive treatments of many properties of hardened concrete. Some details are noted here to relate previous sections to specific properties of the cement paste/aggregate composite.

In short-term loading tests for compressive strength, stress/strain relationships for aggregates and cement paste can be sensibly linear up to near the compressive strength of the paste, while those for concrete exhibit curvature with increasing strain and pseudo-plasticity at stresses above about 0.4 times strength; microcracking at paste-aggregate interfaces develops progressively with increasing strain [40]. For high strength concrete, however, there is little cracking at the interface until the concrete reaches about 0.8 times its ultimate strength.

Creep of many concretes, except possibly those loaded at early ages, is proportional to stress/strength ratios up to 0.3 to 0.6; microcracking also begins in about this same range, depending on the heterogeneity of the mixture. Mortars, for example, exhibit proportionality up to about 0.85. In concretes, stress/strength ratios near this value produce failure in time [40].

Durability of concrete depends strongly on exposure and service conditions, concrete properties–especially porosity and pore structure–and structural detailing. Exposure to acidic or neutral waters causes leaching of calcium hydroxide from the paste, increasing its porosity and permeability, and can eventually soften even the much less soluble C-S-H. Sulfates in fresh waters can penetrate into concrete to cause sulfate attack by reaction with aluminate phases in the cement paste to produce ettringite, which can be destructively expansive if there is insufficient space to accommodate the volume expansion of that highly hydrated reaction product. Calcium and sodium chloride solutions react with aluminates to form Friedel's salt and other complex salts; some of these reactions are also expansive under some conditions. Seawater causes leaching and contains sodium, chloride, magnesium, and sulfate ions in amounts sufficient to cause significant reactions, but the main effect is that of erosion or loss of constituents [1]. Ion exchange, such as the replacement of calcium with alkalies or magnesium, can also damage the concrete.

Moisture and freezing temperatures can cause damage by ice formation in large pores in the paste and in some aggregates. Ice formation at frozen surfaces is propagated through capillaries large enough for the contained water to freeze. Because of ice/water interfacial tension, smaller capillaries require lower temperatures to be penetrated by the growing tips of ice crystals. These crystals in frozen capillaries, and also those in entrained air voids, grow by osmotic accretion of ice by diffusion of water from the gel pores. If entrained air voids are closely spaced, so that their void spacing factors are less than about 0.20 mm (0.008 in.), diffusion of moisture from both capillary and gel pores to ice in the air voids dries the paste and prevents excessive expansions, otherwise caused by ice formation in capillary pores [46].

Concluding Discussion

The materials science of concrete provides a foundation for understanding, predicting, and possibly controlling its behavior. Our understanding of the fundamental relationships between structure and properties has been furthered by computer-based models simulating the development of the microstructure of hydrating cement paste and concrete [45]. These models have advanced sufficiently to allow the prediction of transport and mechanical properties and even provide insights into the reasons for the behavior observed.

The increasing emphasis on high performance concrete provides an incentive for further development of performance-based specifications. To make performance specifications truly practical, and to allow us to use them with confidence, we need to continue to improve the test methods that measure the performance and correlate them with field performance. Ideally, these correlations would not be merely statistical, but would be based on the underlying physical and chemical mechanisms. Some of the models used to predict field performance, for example for the purpose of comparing life-cycle costs of various alternative designs, are not solidly based on empirical data. Thus the use of such models is no better than extrapolation. Further work is needed to provide the necessary data.

Another implication of the use of high performance concrete is that, as we seek to increase the strength or durability of the concrete, or the rate of construction, we observe seemingly new problems: a greater tendency to crack, for example. Although the basics of good concrete practice had been long established when the first edition of this volume was published in 1966, these basics are often slighted in the interests of meeting the constraints of schedule and budget. Concrete is a remarkably forgiving material, performing amazingly well even under the less-than-ideal conditions that prevail in the field. However, high performance concrete is much less "abuser-friendly"; it requires due attention to proper design and craftsmanship. Understanding the science behind the practice enables us to specify and enforce the right criteria to ensure the desired performance.

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3

Techniques, Procedures, and Practices of Sampling of Concrete and Concrete-Making Materials

Toy S. Poole[1]

Preface

THE SUBJECT COVERED IN THIS CHAPTER WAS previously covered by C. E. Proudley in *STP 169*, by E. A. Abdun-Nur in *STP 169A* and *169B*, and by Abdun-Nur and Poole in *STP 169C*. The chapter in *169C* was largely written by Ed Abdun-Nur and contained an extensive bibliography on sampling. The chapter in this edition focuses more on the details of sampling as they are represented in current standards. The reader is referred to the bibliography in *169C* as an excellent source for references on general sampling theory and practice.

Introduction

The purpose of this chapter is to discuss general sampling concepts as they apply to concrete and concrete-making materials, to discuss details critical to specific sampling problems, and to outline the major features of sampling practices described in current ASTM standards.

It is not difficult to make the argument that the details of how, when, and where samples are taken can often be of critical importance in determining the meaning of the test results representing that sample. However, sampling is often a neglected part of the testing process. At least two things contribute to this. One is that details of sampling for various materials are seldom obvious, and therefore require knowledge and training of the individuals involved, who may already be under severe time constraints. Another reason may be that adhering to the details of standard guidance is time consuming and expensive to execute in some situations. It is at least important for people responsible for the quality of construction to have some grasp of the important details of sampling so that, as a minimum, they can develop an understanding of the limits of interpretation of test results when sampling history is unknown.

Sampling Concepts

The two major problems sampling protocols seek to address are variation in a material or concrete structure and bias in the taking of the sample. If sampling is inadequate, and either or both of these problems exist in a material source or in hardened concrete, then test results may poorly represent the material under examination.

Variation is a normal part of the production stream of all concrete-making materials and of all finished concrete. The challenge for sampling is to either capture the nature of this variation, or to smooth out this variation, depending on the purposes for which the sampling and testing is being employed. In some instances the purpose of the work is to describe the variation. This information on uniformity is often needed for monitoring material supplies to help control concrete uniformity and is often part of the description of the hardened concrete properties in a structure. In other cases, the variation is not of particular interest, but rather the average property of the material is the object of the testing. This would plausibly be the case for some materials sources in which considerable blending of material will occur prior to making concrete. In this case, it is important for sampling to cause test results to represent the average property of the material.

Bias in the taking of a sample is particularly a problem in sampling heterogeneous materials, which include to some degree most concrete-making materials. A major source of bias in sampling is segregation within the lot of material being sampled. The intention of the sampling protocol is to avoid misrepresenting a material source due to the sampling of a segregated part of the material. An exception to this would be the case where the sampling and testing is to verify whether or not a stockpile is segregated to the point of causing a problem with uniformity of concrete production.

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So the importance of problems of variation and bias varies with the purposes of the testing program, which explains much of the differences in the details of sampling requirements that are found in the various ASTM standards.

Several terms and concepts consistently appear in sampling sections of ASTM standards. These are the lot, the sample unit (or sometimes unit), the grab sample (or sometimes increment), the composite sample, and the number of samples. The guidance for each sampling problem uses these concepts to guide the user towards solving or avoiding the problems with uniformity and segregation problems typical of that material.

A lot is the fundamental unit of material subject to sampling. Definitions vary among standards, but the phrase ". . . amount of material offered for sale. . ." features prominently in most definitions. Some standards are more specific. In practice the definition of a lot is often determined by the user for his own purposes. It is typically the amount of material in which a user is interested in determining the properties by sampling and testing. For acceptance purposes this is often the amount of material for which a financial payment is agreed on by contract. A lot might reasonably be the amount of product delivered to a project over a day, a week, a month, etc., or it may be defined as some discrete quantity, such as a barge load or a train load. It may also be defined by the user as the total amount of aggregate to be used in a construction project.

A sample unit (sometimes called simply a unit) is a subdivision of a lot. Again, its exact definition is often determined by the user for his own purposes, but typically a lot will be subdivided into a relatively large number of sample units, with a relatively small number of units being actually sampled and tested. A sampling unit may have some meaning with respect to the production of concrete. For example, it may be a truck load of material, the amount used to produce a single batch of concrete, be based on some time interval of production, or the elements of a grid superimposed on a structure. Some standards will define a sampling unit, while others leave it to the user to define. It is normally required that the sample units to be sampled and tested be selected at random.

The simplest type of sample is the grab sample. Grab samples are defined in various ways, but all definitions contain the concept that they are samples taken as a single effort, and that each grab sample represents a relatively small fraction of the material being sampled. Terms such as "one pass" or "one scoop," etc., are often used. The term increment is synonymous with grab sample (as in D 75). It is usually required that grab samples be taken at random locations within a sampling unit. An important concept about grab sampling is that test results among grab samples will tend to show the maximum variation in the material being tested, since there is essentially no averaging in the process.

The composite sample is put together by blending two or more grab samples. Depending on the amount of such compositing directed in a sampling program, composite samples may represent quite a bit of averaging of properties. Standards covering specific materials are usually rather specific as to the amount of compositing allowed or required.

As mentioned above, grab samples show the maximum variation in a lot or production stream of material. This is often unrealistic because the material is rarely or never used in the small amounts represented by a grab sample. In the case of concrete production, the smallest amount of material typically used is the amount that goes into a single batch of concrete. For example, in commercial production a batch of concrete might reasonably contain 1 to 3 metric tons (Mg) of cement, and fine and coarse aggregate amounts might each range from 2 to 10 metric tons of material. So it would be a reasonable practice to composite grab samples over a total amount of material approximating this range with the expectation that tests would then better correlate with performance of a batch of concrete. However, for reasonably uniform materials, this may be an unnecessary detail.

The number of samples required to adequately represent a lot of material varies considerably among testing standards, usually depending on the amount of variation expected in a material or structure being sampled and the confidence interval on the test. This can be a relatively complicated statistical problem. ASTM standards that cover acceptance testing and uniformity testing vary quite a bit as to the number of grab samples required or allowed to make a composite sample and as to the number of composite samples that need to be analyzed to make a determination.

Mostly for purposes of economy, some standards vary the amount of sampling or the amount of testing per sample depending on the purposes of the testing. For example C 183 uses a normal and reduced testing concept, depending on the knowledge of the quality history of a cement source. C 494 uses the concept of quality and uniformity testing. The former is used when the full properties of a lot of material are unknown. In the latter only those tests that are good indicators of uniformity are run.

Acceptance Testing of Concrete Materials

Aggregate

Practice D 75 covers sampling of aggregates. The major problem associated with sampling aggregate is segregation, which can lead to a biased sample. All concrete aggregate is a mixture of a wide range of particle sizes, which tend to easily segregate during handling. The practice covers sampling of aggregate production from flowing discharges, conveyor belts, stockpiles, transportation units, and from in-place roadway bases. Appendices are included that give guidance on practices for sampling potential aggregate sources–quarries and gravel deposits.

The practice strongly favors sampling aggregate from a flowing discharge or from a conveyor belt because of the relative absence of segregation at these locations. The practice discourages sampling coarse-aggregate and mixed coarse-aggregate and fine-aggregate from stockpiles and transportation units, particularly if grading is one of the critical properties to be tested, because of the ease with which segregation can occur and the difficulty in dealing with it in these cases. An appendix to the practice does give some recommendations on practices to use if sampling these is required.

The problem with stockpiles is the tendency for the larger particles to become segregated at the bottom of the pile. Recommended sampling involves using power equipment to dig into the stockpile and taking samples from many locations in hopes of overcoming the possible segregation effect.

The problem with sampling conveyances is that it is difficult to get to various locations in the shipment unless power equipment is available, so that if some segregation has occurred in the loading of the conveyance, it may be difficult to capture this with a series of grab samples.

The definition of what constitutes a lot and what constitutes a sample unit (called a unit in D 75) is left to the user. Units are selected at random. A test sample is defined as a composite of three increment samples (grab samples). The number of samples needed to test a lot is left undefined and must be determined by the user. The number of samples is intentionally left undefined, as explained in the practice, because the number needed to characterize a lot depends on the variability of the material in the lot and the criticality of the test result. If it is relatively uniform or the criticality of test results is low, then a small number of samples is adequate, but if the material is quite variable or the criticality is high, then more samples must be taken to capture this property. Practice E 122 is designed to assist in determining the number of samples required to develop the required resolution.

Cementitious Materials

The major problem sampling schemes for cementitious materials must overcome is manufacturing variation. Standards covering these materials typically give rather specific instructions on when and where grab samples can be taken, on how many grab samples can be composited for a test sample, and also relatively specific instructions on the number of test samples required to adequately represent a lot of production. Segregation is not heavily covered, but many standards do caution about sampling from the surface of a storage or transportation unit because of the layer of fine cementitious dust that can settle there as a result of the loading or transfer process. The procedure is typically to remove several inches of material before taking a grab sample.

Cementitious materials are sometimes carried in trucks or rail cars used for hauling other materials. Small residues of these other materials may reside in the bottom of these containers. This may constitute a relatively minor contamination considering the amount of material in the load, but this relatively minor contamination may show up as a significant contamination if a grab sample is taken from the first material taken from the container. A common result of such a sample (due to, for example, fly ash and cement being transported in the same trucks or stored in close proximity such that contamination can occur) is that the insoluble residue of the cement will exceed normal levels or fail to meet requirements.

Sampling of hydraulic cement is covered by Practice C 183. This the oldest (1944) standard covering the sampling of cementitious materials and the most detailed. This practice contains considerable detail on the sampling of hydraulic cement for purposes of determining conformance with purchase specifications. A number of configurations exist by which hydraulic cement is stored, transferred among storage units, and shipped to a site; therefore, this amount of detail is required to cover all scenarios.

Two standard methods of sampling for acceptance testing purposes are allowed under all circumstances. The methods are:

1. from a conveyor (from process stream) to bulk storage;
2. during transfer between storage bins.

Each method directs that a specific number of grab samples be taken. The standard gives instructions on compositing these into a test sample.

Four optional methods of sampling for acceptance testing purposes are allowed contingent upon approval by the purchaser. These methods are:

1. from bulk storage at point of discharge;
2. from bulk storage and bulk shipment by means of a slotted tube sampler;
3. from bags or packages;
4. from bulk shipment of rail car or truck.

As with the two standard methods, a specific number of grab samples are prescribed for each of these optional methods, with additional instructions in the standard on compositing into a test sample.

The practice distinguishes between sources of cement that have a documented testing history and undocumented sources. Procedures are given for calculating control limits on documented sources. These procedures cause the sampling and testing frequency to be adjusted according to the uniformity of the source and the proximity of a material's properties to the specification limit. A reduced sampling and testing requirement is allowed for these sources relative to an undocumented source.

Hydraulic cement sources typically issue mill certificates listing test data for the cements offered for sale. The user should be aware that the sampling and testing frequency described in C 183 is probably different from the sampling procedures used by manufacturers for quality control and for generating mill certificate data. Cement plants typically take grab samples every hour during production, then make a 24 h composite sample for full testing. The mill certificate typically reflects values from this composite sample. Therefore, mill certificate data probably represent at least a 24-h average. Some may represent a longer period of averaging. Sampling and testing for acceptance typically involves considerably less compositing, so test values are more likely to reflect more of the manufacturing variation in the cement. Therefore these test data may differ from the mill certificate data, depending on the amount of this variation.

Lack of agreement between a user's test data and mill certificate values can be a serious source of contention for properties that typically fall near the specification limit, such as alkali (Na_2O_e) and sulfate content. Manufacturing variation will sometimes cause grab samples to slightly exceed the specification limit, while larger composites will show the average to comply with the limit. Recognizing this problem, some cement plants will sample and test for these properties at a higher frequency, such as every hour during production.

There are also considerable differences among cement companies in the frequency with which they produce mill certificates for distribution to customers. In some cases, the certificate actually represents reasonably closely the cement being sold. In other cases the mill certificate is only a representation of typical values one can expect in the product. In these cases, it is not uncommon that the same mill certificate will be issued with shipments for quite a long time.

Sampling fly ash and natural pozzolans for acceptance testing is covered by Method C 311. Information is given on types of samples, size of samples, and on frequency of samples required for acceptance testing.

Like hydraulic cement, pozzolan is stored and shipped in a number of configurations. Three standard sampling procedures are allowed:

1. from bulk storage at point of discharge, from rail cars, and from trucks (road tankers);
2. from bags;
3. from conveyor delivering to bulk storage.

This practice prescribes different frequencies and different levels of compositing for testing of different properties. Moisture content, loss on ignition, and fineness are believed to require daily (or at least every 360 Mg) testing, while other physical tests and chemical analyses are recommended only on monthly composites. The standard also distinguishes between new and established sources, the latter requiring six months of quality assurance records.

Sampling of ground-granulated-blast-furnace slag is covered in Specification C 989, though not in extensive detail. Sampling is allowed to be either by grab sample or by a composite sample. Each sample is represented by no more that 115 Mg of material. This detail is currently under consideration for revision. The standard is not exactly clear on this, but apparently this mass of material represents the maximum size of a sampling unit. Sampling units are chosen for testing such that, on the average, one sample is collected for each 2300 Mg of material purchased.

Sampling of silica fume is covered in Specification C 1240. As with slag, sampling of silica fume is not covered in great detail. The standard relies heavily on Practice C 183 for guidance, although sampling frequencies are somewhat higher than for hydraulic cements.

Chemical Admixtures

Sampling of chemical admixtures is covered in Specifications C 494 and C 1017. The specification recognizes two purposes for sampling. One is for determining properties for acceptance testing and the other is for determining uniformity.

A major concern in sampling liquid products is segregation on storage due to settling or floating of one or more of the components. This is particularly a problem if the liquid is not a true solution, but rather a suspension or an emulsion of solid particles in a liquid carrier. The solids may either settle or float on prolonged storage. True solutions do not segregate on standing.

The specification directs that containers be agitated or stirred prior to sampling. In the case of large storage tanks that cannot be stirred, sampling at different levels of the tank using a special sampling bottle is required.

Solid-phase admixtures (called non-liquid admixtures in the specification) are sampled using the same grab-sample and composite-sample concepts as for liquid admixtures, although the physical acquisition of the samples more resembles the techniques used for cementitious materials.

Acceptance testing is performed on composite samples, each required to be made up as a composite of at least three grab samples. There is no specific guidance as to the location of these grab samples, but the general guidance is offered that they should be distributed to insure that the composite sample is representative of the lot under test. No definition of lot is offered.

Uniformity may be determined using either grab or composite sampling. If the uniformity within a lot or sampling unit is required, the analysis should be on grab samples. If uniformity among lots or sampling units is required, then the analysis should be on composite samples taken from each.

Sampling of air-entraining admixtures is covered in Specification C 260. The sampling guidance is adapted from Specification C 494, and does not differ substantially.

Curing Compounds

The sampling of curing compounds is covered in Specifications C 309 and C 1315, which contain identical guidance. Segregation is the major source of problems in sampling curing compound. On standing, the solid component of many curing compounds will either float to the surface or settle to the bottom of the container. Therefore, agitating and sampling from the top and bottom of containers is required. These standards contain specific guidance for determining the number of containers to be sampled from a lot. The number is the cube root of the total number of containers in the lot, rounded to the next largest whole number. For example, if there are 100 containers in a lot, then five (4.6 rounded to 5) would be sampled for testing.

Sampling for Determination of Uniformity

Uniformity testing takes two forms. One is testing to determine mixer performance as measured by uniformity within a batch of concrete. The other is testing to determine production stream uniformity, as in uniformity in freshly made concrete or in concrete-making materials.

Mixer uniformity is covered in Specification C 94 on ready-mixed concrete. Mixer-uniformity tests are normally run to determine the minimum mixing time required per batch. There are two important requirements in sampling for mixer uniformity. One is that samples be taken from either the extreme ends of a mixer, if concrete is sampled directly from the mixer, or from the first and last 15 % of the batch, when concrete is sampled on discharge from the mixer. These are apparently the locations in a mixer most likely to show the effects of poor mixing. The other requirement is that, when samples are taken from the discharge stream, an entire cross section of the discharge stream be collected and that nothing be done to impede the flow of concrete from the mixer, such as partially closing the gate. The purpose of these precautions is to prevent getting a segregated sample due to separation of coarse aggregate from mortar.

Determining uniformity of concrete-making materials is covered by Practice C 1451. Method C 917 is a similar standard specific to hydraulic cement. C 917 predates C 1451 and is the model on which the latter was developed. Current specifications on concrete-making materials do not have uniformity limits. The practice is based on analysis of grab samples, so results show the maximum variation in a material. Since it is a practice and not a specification, the exact structure of the sampling scheme is not defined, but left to the discretion of the user. A materials supplier might determine uniformity for its own purposes using a practice of regular sampling, while a user might want to determine uniformity of the materials according to the schedue on which they are received, or for other specific uses. These two sampling schemes could easily result in different calculated levels of uniformity.

Analysis of uniformity among concrete batches is covered by ACI 214 and 318. These standards use compressive strength as the metric for the analysis and determination of compliance. ACI 214 gives sampling and analysis procedures for determining a concrete producer's performance criteria. ACI 318 gives minimum sampling frequencies for quality assurance purposes.

Sampling Fresh Concrete

Sampling from a single batch of fresh concrete is covered by Practice C 172. The principal precautions are to avoid sampling the very first or the very last material delivered from a mixer and to avoid practices that will cause the concrete to segregate during sampling. The first precaution is the exact opposite of the guidance given in C 94 on mixer uniformity, which is to intentionally sample the first and last part of the batch. The apparent purpose in C 172 is to capture the average properties of the batch of concrete and not let irregularities in the fringes bias that determination. Segregation of the concrete can occur when the gate on the delivery stream is partially closed or when only part of the delivery stream is collected, so guidance is given to avoid these conditions.

Specific instructions are given for sampling from stationary mixers, paving mixers, revolving-drum or agitating trucks, and open-top, non-agitating trucks. Instructions are also given for sampling concrete containing large nominal maximum-sized aggregate by wet screening.

Sampling Hardened Concrete

Sampling concrete from structures for purposes of determining in-place properties is covered by Practice C 823. The practice distinguishes two types of problems, each requiring different approaches to sampling. One is sampling for purposes of identifying the cause of some kind of concrete problem, and the other is sampling for purposes of describing the average and distribution of properties of concrete in a structure.

Sampling for the purpose of analysis of a concrete problem is relatively simple if the location of the problem is known. Samples of problem concrete and similar concrete that does not appear to have a problem, if such exists, are usually taken using judgment as to number and location to insure that the full expression of the problem or problems is represented.

Sampling to determine the range and distribution of properties, particularly when property variation is not obvious on visual inspection, is more complicated. If sampling is not done properly, an incorrect description of the state of a structure may result. The practice recognizes two conditions. One is when all of the concrete appears to be of similar condition (called Situation 1). The other is when the concrete appears to be in two or more categories with respect to composition or quality (called Situation 2).

For Situation 1, any random or systematic sampling scheme is allowed, as long as there is no bias in the selection of sampling sites. As shown in a later paragraph, the practice actually recommends using random sampling methods, which are preferable since systematic sampling can sometimes cause the analyst to miss or to overrepresent regularly occurring features in a structure.

The term Probability Sampling is sometimes used in this context. The practice recommends dividing the structure or parts of the structure under investigation into a number of equal sections. The individual sections are sample units. Determining the number of sample units requires some experience. The practice recommends at least ten, ". . . but not less than the number of separate areas that are included in the investigation." The meaning of this phrase is unclear.

Sampling units are chosen by some random selection method. The practice comments on the issue of the number of samples taken, but no guidance is offered on how to determine this number. Practices E 105 and E 122 are cited for this purpose (see below). Determining an adequate number of samples requires some experience and judgment as to the purposes and economic limitations of the analysis. Clearly, the more samples taken, the more detail will be developed in the description of the properties of the structure. A useful approach, if possible, is to take a preliminary round of samples to determine whether there is cause to believe that there are problems in the concrete, then, if such are found, design a more comprehensive plan to determine the extent of the problem in more detail. Practice E 141 gives guidance on how to properly sample for cases likely to be part of a legal dispute.

Paragraph 11.3, on sampling for Situation 2, appears to be very confusing. The paragraph reads, "In Situation 2, samples may be taken for comparison with respect to several categories of performance by using the Chi-Square test (5)." The (5) is a reference to a standard statistics text. The Chi-Square test is usually applied to the analysis of count or frequency data to determine whether the number of events in one category occur significantly more or less frequently than the number of events in one or several other categories. Such a test might be applied in the analysis of the data obtained from samples of a concrete of mixed types or conditions to determine the statistical significance of the frequencies of each type of concrete found (if samples were taken at random). No other guidance is given specific to Situation 2. This section of the practice probably needs to be revised.

Other Guidance

Practice D 3665

This practice was developed to assist in random sampling of road and paving materials. The practice gives examples of how to use a random number table to determine which sublots (sampling units) should be sampled from a lot of material. The practice also gives information on how to determine the number and size of sublots. The practice contains a large random-number table.

Practice E 105

This practice describes recommended rules for setting up a random sampling plan, but is not specific to concrete and concrete-making materials. It also discusses some problems encountered in executing probability-sampling plans.

Practice E 122

This practice gives guidance on how to determine the number of samples needed to give an estimate of a material property to the desired level of precision. It is not specific to concrete or concrete-making materials. The user must determine the desired level of precision. This type of calculation also requires some knowledge of the variation in the material and testing process. The practice gives information on how to estimate this from other sources if there is no such information in the actual testing program.

Practice E 141

This practice gives recommendations for rules for setting up a sampling plan that will be suitable for evidentiary purposes. It is not specific to concrete or concrete-making materials. The practice is particularly designed to assist in one-of-a-kind studies, where there is no previous experience to rely on. It uses terminology that is unconventional relative to that commonly found in concrete standards, and so requires some study to follow. It gives rules for accepting or rejecting evidence based on a sample and for data collection procedures for legal purposes. The practice covers topics on auditing the sampling plan, reporting formats, sample sizes, probability sampling rules, and procedures for determining unbiased estimates of experimental or measurement errors.

ACI 214

This standard gives guidance on sampling, calculating, and using among-batch variation in a concrete production for determining project specifications on strength.

Building Code ACI 318

This is the Building Code. It gives minimum sampling frequencies for concrete for quality assurance and acceptance purposes, as well as sampling guidance for hardened concrete in cases of disputes over strength.

Referenced Documents

ACI 214-77 (Reapproved 1997). Practice for Evaluating Strength Results of Concrete.

ACI 318-99. Building Code Requirements for Structural Concrete.

ASTM C 42-03. Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete.

ASTM C 94-03a. Specification for Ready-Mixed Concrete.

ASTM C 172-99. Practice for Sampling Freshly Mixed Concrete.

ASTM C 183-02. Practice for Sampling and the Amount of Testing of Hydraulic Cement.

ASTM C 260-01. Specification for Air-Entraining Admixtures for Concrete.

ASTM C 309-03. Specification for Liquid Membrane-Forming Compounds for Curing Concrete.

ASTM C 311-02. Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete.

ASTM C 494-99a. Specification for Chemical Admixtures for Concrete.

ASTM C 823-00. Practice for Examination and Sampling of Hardened Concrete in Constructions.

ASTM C 917-98. Test Method for Evaluation of Cement Strength Uniformity from a Single Source.

ASTM C 989-99. Specification for Ground Granulated Blast-Furnace Slage for Use in Concrete and Mortars.

ASTM C 1017-03. Specification for Chemical Admixtures for Use in Producing Flowing Concrete.

ASTM C 1240-03a. Specification for Use of Silica Fume for Use as a Mineral Admixture in Hydraulic Cement Concrete.

ASTM C 1315-03. Specification for Liquid Membrane-forming Compounds Having Special Properties for Curing and Sealing Concrete.

ASTM C 1451-99. Practice for Determining Uniformity of Ingredients of Concrete from a Single Source.

ASTM D 75-03. Practice for Sampling Aggregates.

ASTM D 3665-02. Practice for Random Sampling of Construction Materials.

ASTM E 105-58 (Reapproved 1996). Practice for Probability Sampling of Materials.

ASTM E 122-00. Practice for Calculating Sample Size to Estimate, with a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process.

ASTM E 141-91 (Reapproved 2003). Practice for Acceptance of Evidence Based on the Results of Probability Sampling.

4

Statistical Considerations in Sampling and Testing

Garland W. Steele[1]

Preface

THE APPLICATION OF STATISTICAL CONSIDERA-tions to the sampling and testing of concrete and concrete-making materials has been addressed by chapters in each of the four previous editions of *ASTM STP 169*. The first edition, published in 1956, contained the chapter entitled "Size and Number of Samples and Statistical Considerations in Sampling," by W. A. Cordon. The second edition, published in 1966, contained the chapter entitled "Evaluation of Data," by J. F. McLaughlin and S. J. Hanna. The second edition also contained the chapter by W. A. Cordon that first appeared in the 1956 edition. The third edition, published in 1978, contained the chapter entitled "Statistical Considerations in Sampling and Testing," by H. T. Arni. The fourth edition, published in 1994, contained the chapter entitled "Statistical Considerations in Sampling and Testing," by the author of this chapter. The contributions of previous authors regarding the practical application of statistical probability to the field of concrete and concrete-making materials are hereby acknowledged. This edition will review and update topics as addressed by the previous authors, introduce new clarification of the concepts presented, and include appropriate references.

Introduction

The use of statistical methods to assess test data derived for the purpose of determining the characteristics of concrete and concrete-making materials is now established industry practice. The use of probability-based acceptance criteria founded upon statistically assessed test data applicable to concrete and concrete-making materials is now an accepted industry practice in many areas. Subsequent to the pioneering work by Walker [23], much research and development work was done in and around the decade of the 1960s that aided in establishing the rational foundation for current practices. Many papers documenting the work were published and are still available in various records. One of the most succinct papers, by Abdun-Nur, explaining the need for probability-based concrete strength specifications was published in 1962 [1]. The purpose of this chapter is to provide suggestions regarding the use of statistical applications that are practical, valuable, and appropriate when used in the sampling, testing, and evaluation of concrete and concrete-making materials. If needed, detailed texts on statistical methods and procedures are available from many sources. A few are listed in the references.

General Considerations

Statistical Parameters [2–5]

If the characteristics of concrete or of a material used in the concrete are to be determined with a known probability of meeting the user's specifications, a plan is required. Such plans are commonly called acceptance plans or other similar names that appeal to the designer of the plan. Two types of risks that are often determined for acceptance plans are the seller's risk (α), or supplier's/contractor's risk, and the buyers risk (β), or owner's/specifying authority's risk. When a material is exactly at the acceptable quality level (AQL) as set forth in the acceptance plan for a contract, the probability of rejecting that material is the seller's risk. When a material is exactly at the rejectable quality level (RQL) as set forth in the acceptance plan for a contract, the probability of accepting that material is the buyer's risk. Since it is possible for the quality level to vary over the entire range between the AQL and the RQL, an operating characteristics (OC) curve can be constructed to show the probability of acceptance (or rejection) at any quality level for the acceptance plan, if the plan specifies accept/reject decisions based on specified numbers of tests. If the acceptance plan incorporates pay adjustment factors for various quality levels, determination of the α and β risks is a more complex task. Construction of OC curves for each of the various pay factors is one requirement necessary for further evaluation of the risks. Other tools that are helpful include computer simulation programs to be used in the development of percent within limit (or percent defective) acceptance plans tailored to provide a predetermined buyer's (or seller's) risk, and expected pay (EP) curves that are developed to show the expected average pay for given levels of quality.

When a plan is designed to obtain the desired information through a process called inspection by attributes, each item or group of items will usually be classified only as satisfactory or unsatisfactory so that the relevant parameter is percent satisfactory or percent unsatisfactory. The number of each is recorded for use in decisions concerning the use or other disposition of the item(s). Attributes inspection is occasionally used on precast concrete items.

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The majority of plans designed for concrete and concrete-making materials use a process called inspection by variables. Inspection by variables is done by measuring the selected characteristic of a material or product and recording the measured value. The values are used to calculate fundamental statistical parameters needed to describe the characteristic. Such descriptions are used in decisions concerning the use or other disposition of the material or product.

The fundamental statistics derived from variables inspection that are most useful in making decisions concerning concrete and concrete-making materials are the mean and the standard deviation. The mean (arithmetic), when derived from data gathered by a properly designed plan, conveys, in one value, the average value (central tendency) of the measured characteristic of the material or product. It also indicates that approximately one half of the measured values of the characteristic will have values greater than, and approximately one half will have values smaller than the average value, when measurements are made. Although the mean is a very important statistic, it does not show how far or in what way the greater or lesser values may be distributed from the mean. This information is conveyed by the standard deviation, a powerful statistic that measures variation about the mean, or dispersion. The standard deviation indicates, in one figure, how far above or below the mean other values will be and how many values will likely be found at any distance from the mean.

Other statistics occasionally derived from variables inspection of concrete and concrete-making materials include skewness and kurtosis. Skewness indicates whether the distribution of values tend to be grouped unequally above or below the mean (nonsymmetrical) rather than equally occurring on each side (symmetrical). Kurtosis indicates whether the frequency of occurrence of values at any distance from the mean is greater or less than the frequency expected from a normal distribution.

Other statistics representing central tendency, such as geometric mean, mode, etc., are seldom calculated for concrete and concrete-making materials characteristics. Likewise, other statistics concerning dispersion, such as the average deviation, are usually not calculated. However, in addition to the standard deviation, two statistics concerning dispersion are frequently calculated. These are the range and the coefficient of variation (CV). The range conveys the difference between the largest and smallest value of the measured characteristic. It is not uncommon to see the range used in decisions concerning the use of materials or products. The coefficient of variation is the ratio of the standard deviation to the mean of a group of values, usually expressed as a percentage. This statistic is frequently calculated for measured characteristics of concrete and concrete-making materials.

Generally, as previously noted, the most useful statistic concerning the central tendency of data collected during the measurement of concrete and concrete-making materials characteristics is the arithmetic mean. The most useful statistic concerning the dispersion of said data is less straightforward. The simplest of the three statistics that are commonly encountered is the range. The range is also the least powerful and its use is normally limited to specific applications (for example, control charts). The range is also used occasionally to estimate the standard deviation. However, since many pocket calculators and spread sheet software used in construction activities have keys for direct calculation of the standard deviation, it is preferable not to use the range for this purpose. The standard deviation is the preferred statistic, particularly when it is constant over the range of values collected for the measured characteristic. When the standard deviation is not reasonably constant for the range of values encountered, the coefficient of variation should be considered. If the coefficient of variation is constant, it would normally be used. If neither statistic is constant over the range of values obtained, either statistic may be used. However, the standard deviation is preferred in such cases unless standard practice dictates otherwise. Note that it is acceptable practice to apply either statistic to subset ranges of the overall set of values when said statistic is constant for the subset.

When the behavior of one variable is to be compared to the behavior of another variable, a correlation coefficient may be determined. A good, or significant, positive correlation {A} implies that as one of the variables being correlated increases, the other tends to increase also.[2] Alternatively, the second variable may tend to decrease as the first increases (negative correlation). All that is indicated by a good correlation is that the trend exhibited by the data when one variable is plotted against the other is sufficiently well defined to permit rejection of the hypothesis that there is no relationship between the two variables–in other words, that the relationship is completely random.

Other statistical terms will be encountered when an owner/specifying authority requires comparison of test data derived by said authority's representative with test data derived by the supplier/contractor's representative. The purpose of such comparisons will usually be twofold. First, to determine whether the two datasets are from the same population, and second, to determine whether the material and completed work comply with the governing specifications for the contract. The additional terms encountered may include some or all of the following: difference two sigma limit (d2S limit), difference two sigma percent limit (d2S % limit), t-test, F-test.

The d2S limit is the maximum acceptable difference between two test results obtained on a split sample of the same material using the same process followed in deriving the limit value. (Additional discussion of this limit and the d2S % limit is in the Precision and Bias section of this chapter.) Since d2S limits appear in many ASTM standards, the comparison process is easily followed. When more than one pair of split sample test results are to be compared, a paired t-test may be used. The calculated t-value, based on differences within pairs, is compared to a critical t-value at the selected significance level. Both of these comparisons provide information only on test procedure and equipment variability, as they are designed to do.

A more powerful comparison process involves independent random samples obtained by representatives of the owner/specifying authority and representatives of the supplier/contractor. First, the F-test is used to compare the variances of the two independent sets of data. The ratio of the variances is calculated and compared to a critical F value at the selected significance level. If the null hypothesis (i.e., the hypothesis that the two independent sets of data are from the same population) is upheld for the variances, a t-test is then used to compare the means of the two independent datasets. The calculated t-value is compared to a critical t-value at the selected significance level. If the null hypothesis is upheld for the means, the two sets of data are generally considered to be from the same population. This comparison provides information on test procedure and equipment variability, materials variability, and process variability.

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[2] Letters in braces refer to the notes attached to the end of this chapter.

Regression Lines

The standard method of using paired data as a source in developing a process for predicting one variable from another is by calculating a linear regression line using the method of least squares [6–10]. This method is very useful in situations that involve actual calibration of a measuring device and in which the plotted points approximate the calculated regression line very closely with small scatter {B}. When a coefficient of determination, r^2 (i.e., the square of the coefficient of correlation, r), is calculated for an assumed linear relationship, consideration should be given to selecting a minimum acceptable r^2. However, note that an r^2 value that is closer to the value of 0 than the selected minimum only means that there is little linear correlation between the variables. There may or may not be a high nonlinear correlation between the variables.

When two separate measurements, each obtained from different test methods, are statistically related to each other and one of the test methods is used to obtain measurements that are then used to predict measurements of the other type, there may be a large variation in the prediction obtained from the measured value. Note C and the references contained therein illustrate this point. While both sets of measurements are affected by changes in the property of interest, each is actually measuring two different quantities and each is affected by different sets of influences extraneous to the property of interest. This is usually accounted for by recognizing that many measurements are needed to derive a reliable line.

If regression lines showing the relationship between data from two measurement systems are to be calculated and used for purposes other than illustrating the relationship existing in the data, several points should be considered.

- Derivation of a linear regression line involves determination of two parameters, the slope of the line, b_1, and the Y intercept of the line, b_0. Due to the departures of the data points from the line, each of these parameters has an estimated variance and corresponding standard error (the square root of the variance): s_{b1}^2 for the slope and s_{b0}^2 for the intercept. In addition, there is an estimated variance, s_y^2, and corresponding standard deviation, s_y, calculated from the sum of the squares of the deviations of the measured Ys above and below the fitted line. The three measures of variation (variances or their corresponding square roots) should always be given whenever a regression line is reported. The standard errors of the slope and intercept indicate the significance of the relationship {D}. In addition, the number of pairs of data from which the regression was calculated and the upper and lower limits of the data of both X and Y values should be reported along with the other parameters.
- A confidence interval for the fitted line should be shown on the graph of the line {E} (Fig. 1).
- There are three kinds of confidence intervals that can be calculated for a fitted regression line: the line as a whole, a point on the line, or a future value of Y corresponding to a given value of X {F}.
- When appropriate confidence limits are correctly calculated and reported, proper use requires a practical understanding of the confidence interval given {G}.

It should be reiterated when using regression lines that new measurements are probably subject to the same degree of variation that characterized the original data.

In general, then, if any system of measurements is valid, "the system must have validity within itself." The use of a regression equation to convert measurements obtained in one system to measurements that might have been obtained by some other system introduces an additional degree of uncertainty into the process. Extrapolation beyond the limits of the data from which the equation was derived increases the degree of uncertainty in the derivation. Further, the use of relationships other than linear equations will, in appropriate situations, provide more reliable data {H}.

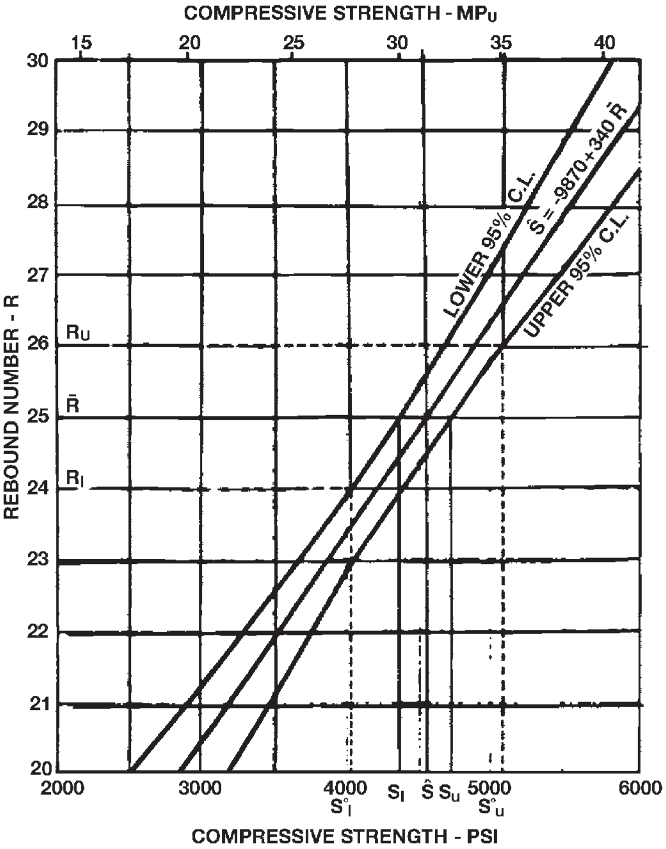

Fig. 1—Regression curve and confidence limits for compressive strength versus rebound numbers [12].

Sampling

The goal of sampling concrete and concrete-making materials is to obtain samples from which an unbiased estimate of the characteristic of interest of a lot of material or product can be obtained. Therefore, when a sample is used to evaluate a characteristic of a lot (quantity) of material, all material in the lot should have an equal chance of being included in the sample. Alternatively, if the probability of inclusion is not the same for all parts of the lot, this fact should be considered when evaluating data derived from the sample.

Given the preceding goal, the simplistic answer may seem to be a requirement that all material in the lot should have an equal chance of being included in any sample obtained from the lot. However, in many situations this may not be practical. Consider, for example, a 100-yd^3 (76-m^3) concrete placement.

- Using an appropriate plan, the fresh concrete could be sampled during the placement process in a manner that would assure that all concrete had an equal chance of being included in the sampling operation.

- In contrast, using an appropriate plan for sampling hardened concrete, it is likely that in most situations the hardened concrete in different segments of the placement would have an unequal chance of being included in the sampling operation (or even no chance). Final geometry, critical stresses, reinforcement, and lack of accessibility all contribute to the situation.

It is, therefore, very important that the circumstances in each sampling situation be recognized and receive competent consideration.

The term "representative sample" has been used in many different ways in the construction industry. The most useful definition would paraphrase the goal of sampling previously noted; that is, a representative sample is a sample that is obtained from a lot or quantity of material, using procedures that will allow an unbiased estimate of a particular characteristic of the material to be derived from the sample. If the stated definition is to be operant, the sample must be selected randomly. ASTM Practice for Random Sampling of Construction Materials (D 3665) contains a random number table that can assist the sampler in obtaining random samples.

The definition should also assist in a better understanding of composite samples. If, for example, a particular characteristic of several randomly selected 2000-lb (900-kg) units of aggregate is to be used to estimate that characteristic in 10 000 tons (9000 metric tons) of material, the characteristic can be determined from testing each of the 2000-lb (900-kg) units in total. Alternatively, five (or some other suitably determined number) randomly selected subsamples may be selected from each of the 2000-lb (900-kg) units for compositing. The five randomly selected subsamples from each of the 2000-lb (900-kg) units may then be combined to form a single sample obtained from each 2000-lb unit randomly selected for sampling. This is called a composite sample. Results from the single composited samples may then be used to estimate the characteristic of the 10 000-ton (9000-metric ton) lot of aggregate. Obviously, this alternative provides a possible advantage in that much smaller quantities of aggregate are handled in the final sample. However, a possible disadvantage to be considered is that variations in the characteristic of interest that exist in the 2000-lb (900-kg) unit of aggregate will remain unknown. A second alternative for compositing is available that provides the information needed for estimating the variations within the 2000-lb (900-kg) units of aggregate for the characteristic of interest. The second alternative requires that each subsample be individually retained and used for testing. The data derived from each subsample in a 2000-lb (900-kg) unit may then be averaged and used in the same manner as the data derived from the composited sample in the first alternative.

Similar types of examples could be presented for cement, other concrete-making materials, and concrete, either fresh or hardened.

The number of samples needed to provide the information necessary for estimating a particular characteristic of a lot or quantity of material with a given confidence in the results can be easily determined. ASTM Practice for Calculating Sample Size to Estimate with a Specified Tolerable Error, the Average for Characteristic of a Lot or Process (E 122) contains equations for calculating sample size. Note that "sample size" as used in ASTM E 122 is equivalent to the "number of samples" as commonly used in the construction industry.

A useful concept when sampling concrete and concrete materials is "stratified random sampling." This concept can be easily implemented by dividing the lot or quantity of material into a number of sublots or subquantities. The most convenient number is usually equal to the number of samples to be obtained. Each sample is then randomly obtained from one sublot or subquantity. The purpose of stratified random sampling is to prevent the possibility that several samples would be obtained from one segment of the lot or quantity of material being sampled.

An adequate plan for evaluating specific characteristics of a material (or process) will provide sampling direction. This should include the type, frequency, method, and location of sampling. Furthermore, the quantity and handling of samples subsequent to selection should be clearly detailed [19].

Testing

The reason for testing concrete and concrete materials is to produce data from which unbiased estimates of certain characteristics of the material can be derived. The reliability of these estimates improves as the number of test results increases. Also, although depending somewhat on the purpose for which the estimates will be used, the reliability tends to increase as the quantity of material undergoing test increases. To illustrate, consider the 2000-lb (900-kg) unit of aggregate example set forth in the previous section on sampling. Without resorting to mathematical proof (which could be done), it is intuitively seen that the performance of a test on the entire 2000-lb (900-kg) unit will likely yield a better estimate of the characteristic being determined than would the performance of a test on a 10-lb (4 1/2-kg) subsample obtained from the 2000-lb (900-kg) unit. Likewise, estimates based on ten 10-lb (4 1/2-kg) subsamples obtained from the 2000-lb (900-kg) unit will be more reliable than the estimate derived from one 10-lb (4 1/2-kg) subsample. Then, as the number of subsamples is increased, the reliability of estimates based thereon will approach that of the 2000-lb (900-kg) unit. Finally, when the number of subsamples obtained from the 2000-lb (900-kg) unit equals 200, the reliability of the estimates will be equal. However, as noted in the original example, the information concerning the characteristic that can be derived from the 200 subsamples will be far greater than that which can be derived from treating the 2000-lb (900-kg) unit as a single sample.

It is also intuitively evident from the previous example that testing additional samples or samples of greater size increases the cost of testing. It is necessary, therefore, to establish the reliability required in each case commensurate with the resources that are to be made available and with how much information it is necessary to obtain. These decisions should also be based on a determination of the consequences of inadvertently accepting defective material due to a low level of reliability in the sampling and testing process.

Similar examples could be presented for cement, other concrete-making materials, and concrete, either fresh or hardened.

Although it is seldom articulated, it may be better to make no tests than to make tests with poor samples that do not portray the actual properties of the materials. An engineer, representing the owner or specifying authority, who must rely on samples or tests that do not provide reasonably unbiased estimates of the properties of interest for materials or structures could probably make more appropriate decisions if there were no samples or tests available. Such decisions would likely be based on conservative assumptions, with large safety factors, rather than reliance on fallacious information with consequent unknown risks. While neither of these situations will generally yield optimum cost effectiveness, the greater risks inherent in using faulty data are obvious. Optimum cost effectiveness minimizes the risk that the owner will accept faulty material and the risk that the supplier will have acceptable material rejected. These risks were previously defined and discussed in the Statistical Parameters section of this chapter.

Evaluation

Test data, to be useful after they have been obtained, must be evaluated with respect to a standard or potential standard such as contract documents, specifications, design criteria, or other previously determined concepts. Any evaluation of concrete or concrete-making materials will be more practical and informative when commonly used statistical procedures are applied. Typical guidance can be obtained in numerous publications. ASTM Practice for Sampling and the Amount of Testing of Hydraulic Cement (C 183) utilizes control charts {I} and quality history to determine the frequency of testing and the evaluation of hydraulic cement. The versatile concept of moving averages is demonstrated in the evaluation of cement strength uniformity as set forth in ASTM Test Method for Evaluation of Cement Strength Uniformity from a Single Source (C 917). ACI 214, Recommended Practice for Evaluation of Strength Test Results of Concrete {J}, provides detailed procedures for the evaluation of concrete strength tests.

The principles contained in the three cited standards above can be extended to many other concrete and concrete-making materials tests. However, for a few nonparametric {K} tests this is not the case. Examples are ASTM Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals (C 672) that uses an ordinal scale of measurement and ASTM Test Method for Organic Impurities in Fine Aggregates for Concrete (C 40) that uses a nominal or classification scale. The median can be used to indicate central tendency for such tests. Reference [26] should be consulted for other procedures applicable to these types of test data.

Another consideration that may be encountered when evaluating values derived from test data is whether retesting is appropriate. Generally, when the level of quality of a material or process is estimated from the evaluation of one set of test results that have been obtained in accordance with appropriate standards, said estimate will be improved by including another set of test results, obtained in accordance with appropriate standards, in the evaluation procedure. The practice of retesting is fully acceptable if all test results, both original and retests, are 1) obtained and tested in accordance with appropriate standards, 2) included in the final evaluation, and 3) the final evaluation recognizes the increased number of test results existing for that particular process or lot of material.

Precision and Bias Statements

One of the most important factors concerning a test method that is used to determine acceptance and rejection of materials and construction in a buyer-seller relationship is the information contained in the precision and bias statement.

Numerical limits, based on standard test data, which are included in specifications to govern acceptance decisions, should be fully compatible with the precision and bias information contained in the test standard used for generating the data. Reference [27] provides additional information concerning this point. Test standards that contain no information concerning precision are of limited value in making acceptance or rejection decisions.

Standards {L} best suited for the determination of precision and bias of tests for concrete and concrete-making materials are ASTM Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials (C 670) and ASTM Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials (C 802). The indices of precision used in these standards are, in order of preference, the "difference two-sigma limit (d2S) and the difference two-sigma limit in percent (d2S %). The indices are determined by multiplying the factor $2(2)^{1/2} = 2.828$ by the standard deviation of test results or the coefficient of variation of test results, respectively. This is the standard deviation or coefficient of variation obtained from a properly planned interlaboratory study. The d2S or d2S % limit published in a test standard will generally have descriptive terms preceding the value listed that will define the process followed in deriving the value. Typical descriptive terms include the following: "single laboratory," "within laboratory," "multi-laboratory," "between laboratory," "single operator," "within test," etc. When one of these indices is provided in a test standard, the user of the standard can presume that only about one time in 20 will the difference between two test results {M} on split samples, obtained under approximately the same conditions noted in the precision information, exceed the value indicated by the index. If more than two results are to be compared, different multipliers are required to determine the index. ASTM C 670 contains a table of multipliers for determining indices for use in comparing up to ten test results.

When test values from independently obtained samples are to be compared, the F-test and t-test should be used as previously discussed in the Statistical Parameters section of this chapter. Test values derived from independently obtained samples contain additional components of variance not present in test values derived from split samples. The following items are very important in the maintenance of valid precision information in standard test methods:

- Requirements for the designated number of test results (one or more) that constitute a valid test should be stated in a test standard.
- Criteria used in deriving the precision information should be outlined in the standard.
- When a standard test is revised, other than editorially, the precision information should be reviewed to determine whether a new precision index should be derived.

Although other indices based on different criteria can be derived and used, those noted earlier have been recommended as the most appropriate for test standards developed by ASTM Committees C1, C9, D4, and D18.

Other Useful Standards

ASTM Practice for Conducting A Ruggedness or Screening Program for Test Methods for Construction Materials (C 1067) provides an economical procedure for the detection and reduction of variations in test methods before initiating a complete interlaboratory study.

Procedures for determination of precision based on values from other related tests are presented in ASTM Practice for

Calculating Precision Limits Where Values are Calculated from Other Test Methods (D 4460).

Procedures for deriving the variation of properties of concrete-making materials from a single source and appropriate comparisons of such derivations to precision statement data are presented in ASTM Practice for Determining Uniformity of Ingredients of Concrete from a Single Source (C 1451).

ASTM Practice for Inclusion of Precision Statement Variation in Specification Limits (D 6607) presents guidance concerning the appropriate inclusion of test method precision values in specifications containing limits that are based on data obtained by use of said test method.

Notes

{A} For example, many in-place tests on concrete tend to show increases in the measurements obtained as concrete strength increases. A good correlation (or high correlation coefficient) between the results of any test that exhibits such behavior and the results of standard strength tests does not in itself constitute evidence that the relationship is sufficiently close to permit the use of one type of test as a means of predicting what will happen in another type of test. In fact, the correlation coefficient has limited use in the field of analyzing engineering data.

{B} An example is the calibration of proving rings that are used for calibrating testing machines. This use is discussed by Hockersmith and Ku [11].

{C} Even when the existence of this uncertainty is recognized, its size and affect on the derived measurements may be underestimated. This point is illustrated in the case concerning prediction of compressive strength measurements from penetration and rebound tests in Refs [12] and [13].

{D} In general, if the slope is not at least twice as large as its standard error, this is insufficient evidence to conclude that the true slope is other than zero (that is, the relationship is random).

{E} Calculation of the quantities needed to plot the confidence interval is described elsewhere [6]. It should be noted that the upper and lower confidence limits are represented by two branches of the hyperbola that are closest together at the point where $X = \bar{X}$, the average of all the X values used in calculating the relationship. Thus uncertainty of the predicted Y increases as the X value departs from $\bar{X}$ in either direction. Occasionally, confidence intervals have been plotted by multiplying the S_Y (standard error of the Y estimate) by the t value for the number of points used and drawing parallel straight lines above and below the regression line. This is not correct and gives an optimistic picture of the uncertainty of estimated Y values.

{F} Reference 6 describes how to calculate these three confidence intervals. It is important to note that only the confidence interval for the location of the line as a whole is appropriate to use if the calculated line is to be used repeatedly for predicting future values of Y from future observed values of X.

{G} Figure 1 (taken from Ref [12]) illustrates the point. The regression line shown is based on 16 plotted points relating strengths of 28-day cylinders to the average of 20 Swiss hammer rebound numbers obtained on slabs made from the same batches of concrete as the cylinders. The figure also shows the hyperbolic curves representing the upper and lower 95 % confidence limits for location of the line referred to earlier.

For a hypothetical rebound number of 25, this figure indicates a calculated average compression strength of 31.2 MPa (4530 psi), with the 95 % confidence interval extending from 30.0 to 32.5 MPa (4350 to 4710 psi). Since the rebound numbers themselves have a distribution with a characteristic scatter, illustrated in this case by a standard deviation of 0.50 for averages of 20 rebound numbers, the 95 % confidence interval for the average rebound measurement is from 24 to 26 (i.e., $<25 \pm [2(0.50)]\}$. These figures combined with the confidence interval for the line give an approximately 90 % confidence interval for the predicted compressive strength of 27.6 to 35.1 MPa (4010 to 5070 psi).

Unfortunately, this is not the limit of the final uncertainty of the predicted result. A 95 % confidence limit is often interpreted as meaning that 95 % of future results will be within the limits given. What it actually means, however, is that if the experiment is repeated a large number of times, each with the same materials and conditions and with the same number of determinations, and each time the 95 % confidence interval is calculated, then 95 % of the intervals so calculated will include the true average. This does not mean that any particular one of the intervals will actually contain the true average in its exact center. For any given determination of the line and its confidence interval, it is highly unlikely that the calculated line will coincide exactly with the true line.

{H} One example that illustrates the successful use of nonlinear equations for determining the relationship between certain variables in concrete is contained in ASTM Test Method for Developing Early-Age Compressive Strength and Projecting Later-Age Strength (C 918). This standard involves the application of the maturity concept. The concept goes back a long way, but detailed work was done on its application to evaluation of concrete strengths in the 1970s [15–18].

The standard involves establishing a relationship between the strength and the logarithm of a quantity called maturity of the concrete that is defined as the product of temperature at which the curing is taking place and the curing time in hours (degree-hours).

{I} *Monitoring Production-Continuous Evaluation (adapted from Ref [20])*—One of the most effective means of maintaining the quality of a manufactured product is by continuously monitoring the quality by means of regularly performed tests throughout the process of production. The best tool for doing this is by means of control charts.

The control chart became a well-established technique in production quality control during the World War II era. The control chart is a combination of both graphical and analytical procedures. The basis of the theory arises from the fact that the variation of a process may be divided into two general categories. One portion of the variation can be described as random or chance variation of the process and the other as the variation due to assignable causes. A process that is operating with only chance variation should result in some distribution of the measured characteristics, and one should be able to predict a range within which a certain percentage of the data should fall. If some assignable cause (such as an increased water-cement ratio) results in a change in the distribution, then the values of the measured characteristic could fall outside the predicted range.

Three types of control charts that are frequently used are control charts for averages (or moving averages), control charts for standard deviations (or moving standard deviations), and control charts for ranges (or moving ranges). Also, control charts for other measures, such as percent defective (or percent within limits), may be useful. Detailed treatment of this subject and tables of control chart constants for determining upper and lower control limits are presented in texts on statistical quality control [14,19,21]. Table 27 in Ref [22] contains the control chart constants for averages, standard deviations, and ranges.

{J} *Evaluation of Strength Tests*—One of the earliest and most widely used applications of statistics in the concrete field has been in the area of evaluation of strength tests both of mortar cubes for the testing of cement strength and, more extensively, for the analysis of strengths of concrete specimens, usually, in the United States, in the form of 6 by 12-in. cylinders. The chief pioneer in this effort was Walker who published his study in 1944 [23]. In 1946, largely at the instigation of Walker, the American Concrete Institute (ACI) began work on statistical evaluation of compression tests that eventually resulted in the publication of ACI Standard Recommended Practice for Evaluation of Strength Test Results of Concrete (ACI 214-03). First published as a standard in 1957, this document has undergone a number of revisions.

In 1971 a symposium was conducted at the ACI Fall Convention on the subject "Realism in the Application of ACI Standard 214-65." This symposium presented valuable information on the meaning and use of ACI 214 and resulted in a symposium volume that included seven papers presented at the symposium, a reprint of ACI 214-65, and reprints of two earlier papers dealing with evaluation of concrete strengths [24].

{K} *Nonparametric Tests*—There are some test methods that do not provide numbers for which the customary processes of calculating means, standard deviation, (d2S) limits, and other so called parametric statistics are applicable. Such tests measure on a nominal or classification scale, or on an ordinal or ranking scale [25]. Test methods of the latter type sometimes cause problems because of the fact that numbers are assigned to the different levels of quality of performance in the method, and then the numbers are treated as though they represented measurement on an interval scale, which is the type of measurement scale appropriate to most concrete or concrete-making materials test methods. Because of the numbers derived, there is a temptation to average results of several specimens and even to calculate standard deviations to indicate scatter. Such calculations are inappropriate when the magnitudes of the numbers indicate only order or rank and not measurements of quantities. When lengths are measured, for instance, the difference between an object that measures 5 cm and one that measures 6 cm is a length of 1 cm. The same difference applies to two objects that measure 9 and 10 cm, respectively. The increment of one between scaling ratings of one and two, however, is not necessarily the same increment as that between three and four. Adding ranking numbers of this type and dividing by the number of measurements may have little significance. Central tendency and scatter can be indicated by giving the median and the range.

A test that provides measurement on a nominal or classification scale is one in which results merely fall into different categories without any judgment being made that one category is higher or lower than another; for example, ASTM C 40. In one procedure, a solution from the test sample is compared to a reference solution and judged to be lighter, darker, or the same. In another procedure, five color standards may be used. The latter may be treated as an ordinal scale if one end of the scales is judged to be better than the other end and the stages in between represent progression from one level to another.

{L} As a result of concern about problems connected with precision statements and how to develop and use them, a joint task group of ASTM Committee C1 on Cement, C9, D4 on Road and Paving Materials, and D18 on Soil and Rock for Engineering Purposes developed two practices: ASTM Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials (C 670) and ASTM Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods for Construction Materials (C 802). ASTM C 670 gives direction and a recommended form for writing precision statements when the necessary estimates (usually standard deviations) for precision and/or bias are in hand. ASTM C 802 describes a recommended method for conducting an interlaboratory study and analyzing the results in order to obtain the necessary estimates. Both of these standards should be studied and followed closely by any task group that is charged with writing a precision and bias statement for construction materials.

{M} If two results differ by more than the (d2S) limit, a number of interpretations are possible. Which interpretation is most appropriate depends on various circumstances connected with the situation, and in most cases a degree of judgment is involved.

The limit in a precision statement is to provide a criterion for judging when something is wrong with the results. Thus the failure of a pair of results to meet the (d2S) criterion causes concern that the conditions surrounding the two tests may not be the same as those existing when the precision index was derived or that the samples used in the two tests are not unbiased samples from the same type of material. The appropriate action to take depends on how serious the consequences of failure are. In most cases, a single isolated failure to meet the criterion is not cause for alarm, but an indication that the process under consideration should be watched to see if the failure persists. If appropriate, the tests can be repeated, and usually the procedures of the laboratory(s) involved should be examined to make sure that the test is performed in accordance with the standard from which the precision statement was developed.

Failure to meet a multilaboratory precision limit may entail more serious consequences than those connected with failure to meet a single-operator criterion. The latter is sometimes used to check the results and procedures of a single operator in a laboratory, and failure to meet the criterion leads to reexamination of the materials and procedures. If the test is being used to determine compliance with a specification, the single-operator (d2S) limit should be used to check whether or not the results obtained are a valid test for the purpose. The former may occur in situations where there is a dispute about acceptance of materials. In these cases, both laboratories should obtain two results by the same operator who was used in the multilaboratory tests, and use the single-operator difference as a check on proper performance of the test method within the laboratories.

Also note that conditions, materials, apparatus, operators, etc., change with time. In many cases, the subcommittee responsible for the test method can obtain proficiency sample data from the Cement and Concrete Reference Laboratory or the AASHTO Materials Reference Laboratory from which appropriate revisions to update a precision statement can be drafted as shown in the ASTM C 670 appendix.

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5

Uniformity of Concrete-Making Materials

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Preface

THE SUBJECT OF VARIABILITY OF CONCRETE-making materials was first covered in *ASTM STP 169C.* It was derived from the work of the joint ASTM C01/C09 Task Group on Variability of Concrete-Making Materials. The Task Group provided the impetus for development of guidelines on determining uniformity of concrete-making materials, which eventually became ASTM Standard Practice for Determining Uniformity of Ingredients of Concrete from a Single Source (C 1451).

Introduction

The goal of the concrete supplier is to provide a material that consistently meets requirements set out by the buyer, whether these are defined in the form of prescriptive or performance specifications. The question is, "How do we define and assure uniformity of concrete?" To answer this question, it is necessary to consider those factors that affect concrete properties and performance.

The steps to obtaining concrete performance are conceptually illustrated in Fig. 1. The process starts with a mix design and specification developed for the particular application. It is followed by selection and acquisition of constituent materials and processing of those materials in accordance with the specifications. Presumably, if the design, selection, and implementation steps are properly conducted, the concrete properties and performance will meet job requirements. However, it is naive to assume that the steps to obtaining properties and performance can be achieved without accommodating variations. But what level of variation can be accepted without detrimental impact on performance? This chapter will address the variability of concrete-making (constituent) materials and their effects on performance.

With improvements in concrete technology, concrete has become more versatile, but also more complex in that the number of mixture constituents has increased. It is rare to encounter concrete that consists only of cement, fine aggregate, coarse aggregate, and water. Today, most mixtures also contain chemical admixtures or mineral admixtures (supplementary cementitious materials) or both. To minimize the variability of concrete, it is necessary to control the uniformity of constituent materials as well as the uniformity of batching, mixing, transporting, placing, and curing. Uniformity of properties may be as important to the concrete supplier as the individual properties themselves. Within relatively broad limits, the supplier can adapt concrete mixtures to accommodate individual properties of constituent materials. However, once that is done, it is essential to maintain uniformity to assure consistent concrete properties and performance. For example, once a mixture has been developed for a specific cement, water reducer, retarder, air-entraining admixture, aggregates, and batch water, unanticipated changes in critical properties of individual components can cause problems with fresh or hardened concrete performance.

The ASTM C01/C09 Uniformity Task Group identified important properties of constituent materials, how much variation is acceptable, and how uniformity from a single source of these materials can be controlled.

Properties of Constituent Materials that Affect Concrete Performance

In 1988, members of ASTM Committee C1 on Cement and Committee C9 on Concrete and Concrete Aggregates were surveyed to obtain their impressions on the relative importance of concrete-making materials. Members were asked to rank major constituent materials in their order of importance relative to variability, and also in the context of field practices for three categories of construction (residential, low-rise commercial, and high tech/high strength). In addition, each constituent material was rated relative to its own properties and attributes.

Twenty-eight members responded to the survey. Since no attempt was made to scientifically select the sample population, no claim can be made for statistical significance. However, the respondents are among the world's most knowledgeable and experienced individuals in concrete materials technology. Therefore, the survey can be considered a valid representation of industry experience and perceptions regarding those materials' characteristics that affect concrete performance. And anecdotal evidence indicates these perceptions remain unchanged today.

The survey provides guidance on specific materials' properties and performance attributes that impact concrete properties and performance. This information is valuable in identifying properties that must be controlled to achieve uniformity of performance.

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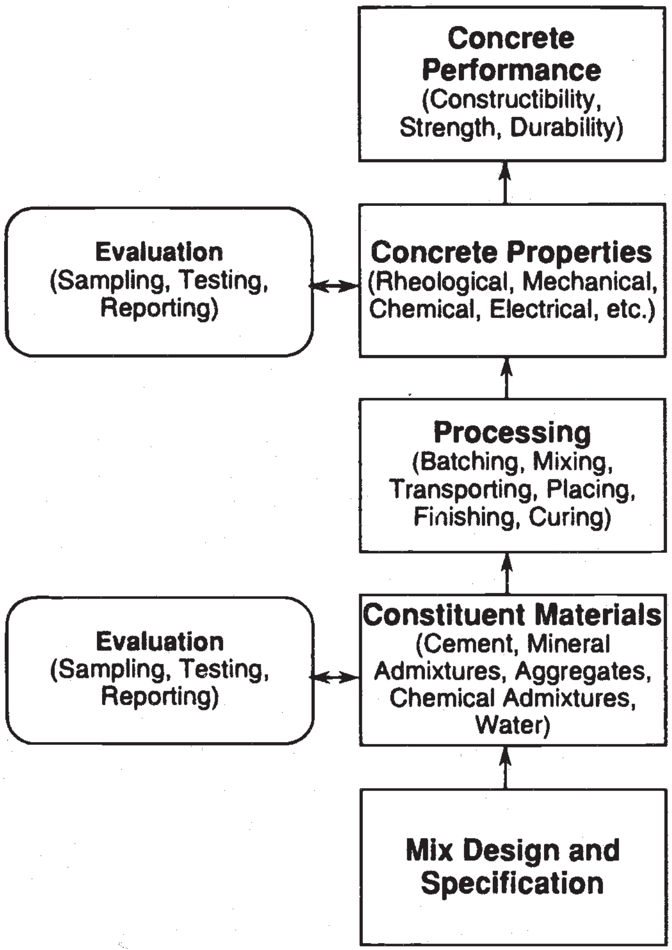

Fig. 1—Uniformity of concrete is a function of the entire design and construction process.

Figure 2 is a summary of responses (27 for this part) to a question that required the respondent to rank ten major constituent materials in order of importance from one being most important to ten being least important. In the context of the question, "important" relates to what impact variations in the constituent material would have on concrete performance. No distinction was made as to what performance aspect—constructibility, strength, or durability–might be affected, but it is likely that strength was the most commonly considered attribute. Variations in cement are identified as the most important by a significant margin. Variations in batch water are identified as least important. The relatively "unimportant" rankings given to slag and silica fume may be related to a belief that these materials have little variability, or to the fact that they are less frequently used than the other constituents.

Figure 3 shows results when constituent materials were rated within different construction types (residential, low-rise commercial, and high tech/high strength). The intent of the question was to determine the overall importance of potential variability in the constituent material for selected types of construction. Answers were to reflect whether the variability of the constituent material can be considered to produce few or numerous field problems. In this part of the survey, the materials were rated (not ranked) on a scale of one (important) to ten (not important). Not surprisingly, the overall importance (lower rating numbers) increased from residential to commercial to high tech. Cement was considered the most important for all construction categories. For residential and low-rise commercial construction, silica fume was considered least important (not likely to be used), while for high-tech/high-strength concrete, batch water was considered least important.

While the results in Figs. 2 and 3 provide a rather general picture of perceptions about the relative importance of constituent materials, another valuable part of the survey is summarized in Table 1. For this part, major constituent materials were evaluated independently of each other to identify those characteristics that are important to performance. Respondents were asked to rate each material property or performance attribute on a scale of one to three with one being most

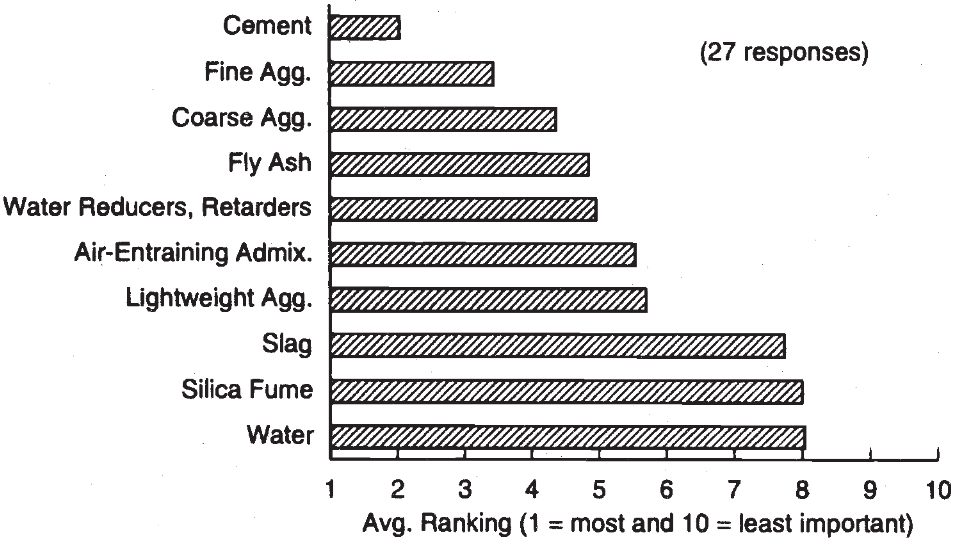

Fig. 2—A 1988 survey of ASTM C1 and C9 committee members revealed their perceptions about the impact of variability of constituent materials on concrete performance.

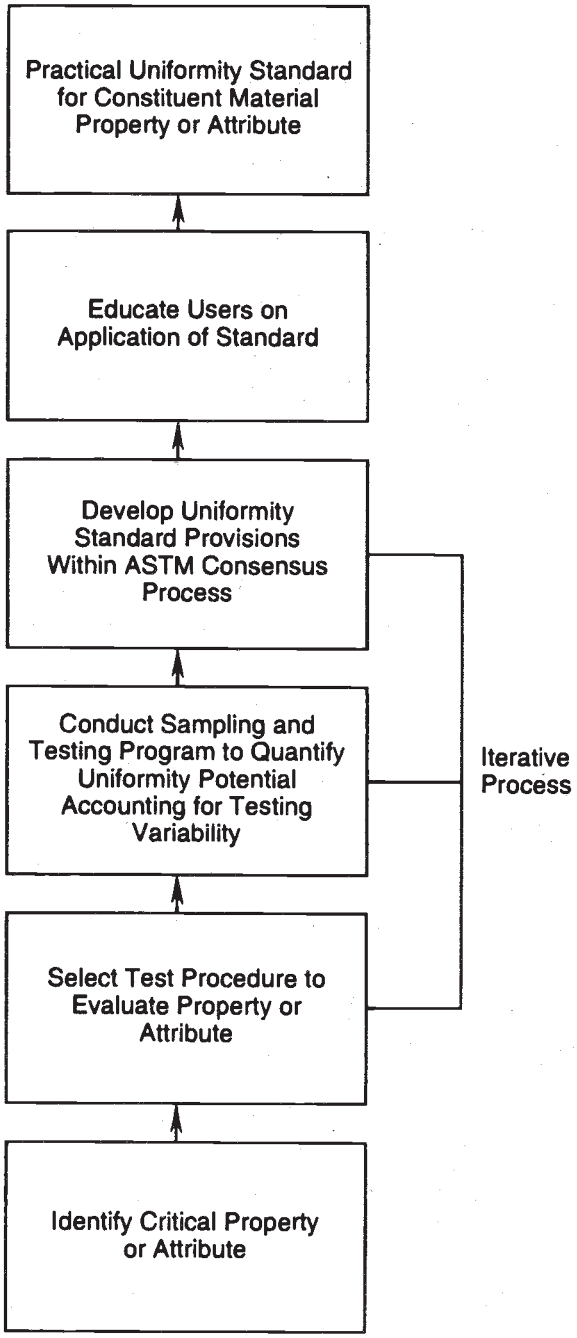

Fig. 3—ASTM C01/C09 1988 survey of perceptions about the impact of variability of constituent materials on concrete performance based on field practice for different construction types.

important and three being least important. Performance attributes reflect the behavior of the constituent material when incorporated in paste, mortar, or concrete.

With further quantification, data such as listed in Table 1 serve as the basis for uniformity standards. Before such standards can be developed, specific characteristics to be controlled must be identified and then quantified with respect to their impact on concrete performance variations. See, for example, Johansen and Taylor's summary of the effect of cement characteristics on concrete performance [1]. Table 1 provides a comprehensive list of properties and attributes for the major constituents in concrete. It also provides an indication of their perceived level of importance relative to defining potential variations in concrete performance. This identifies critical characteristics that should be considered in uniformity standards for constituent materials. Such was the case for the only existing uniformity standard for concrete-making materials, ASTM Test Method for Evaluation of Cement Strength Uniformity from a Single Source (C 917).

Evaluation of Uniformity

An Example: ASTM C 917

The development of ASTM C 917 took place over a number of years, starting in the 1960s and culminating in its first edition in 1979 [2]. It is worth reviewing the development of ASTM C 917 because it is representative of efforts that are needed to implement uniformity standards for properties or attributes of other concrete-making materials. Therefore, the following discussion is presented not to focus on cement strength issues, but to illustrate the process of developing a uniformity standard.

The fact that cement strength was selected as the first attribute to be standardized is not too surprising given the earlier discussion of Figs. 2 and 3 and Table 1 from the 1988 ASTM survey. In fact, the initial impetus for development of ASTM C 917 can be traced to work by Walker [3] and Walker and Bloem [4]. The key point is that a specific attribute of a constituent material for concrete was identified as important to the uniformity of concrete. This led to the establishment of a joint committee of the Portland Cement Association and the National Ready Mixed Concrete Association to address strength uniformity [5]. The joint committee planned a program to develop data on uniformity of cement strengths from individual cement plants [6].

The joint committee selected 7- and 28-day strengths of mortar cubes that conform to ASTM Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens) (C 109) as the reference for cement strength. After a pilot program in 1975, a one-year voluntary sampling and testing program was initiated in 1976. Forty-six cement companies, representing over 100 plants in the United States and Canada, participated. Testing was conducted on grab samples representing 25 ton (23 Mg) lots of cement at the rate of 30 samples per calendar quarter (preferably ten per month and not more than one per day). Mortar cubes were prepared in each plant's laboratory with ten duplicate sets of cubes prepared each quarter to evaluate within-laboratory test error. Data were submitted quarterly for statistical analysis.

Data from the program that includes information on standard deviations, coefficients of variation, 7- and 28-day average strengths, and ratios of 28-day to 7-day strengths were published in the appendix to ASTM C 917.[2] They were arranged in terms of cumulative percentage of plants falling below the value indicated for the statistic of interest, and provide a reference point for comparing strength uniformity results from a particular source.

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[2] Data developed in 1991 were used to update the standard [7].

TABLE 1—Perceived Relative Importance of Materials Characteristics to Concrete Quality (ASTM C01/C09 1988 Survey)

	Number of Responses[a]			
	Average	1	2	3
CEMENT UNIFORMITY				
Material property				
Sulfate form and content	1.36	19	8	1
Fineness	1.39	17	11	0
C3A, C3S, C2S	1.43	18	8	2
Alkalies	1.50	17	8	3
Solubility of alkalies	1.68	12	13	3
Chemical composition	1.78	9	15	3
Microscopically determined composition	1.93	8	14	6
Heat of hydration	2.04	6	15	7
Air content	2.04	6	15	7
Loss on ignition (L.O.I.)	2.11	8	9	11
SiO_2, Al_2O_3, MgO	2.15	3	17	7
Specific gravity	2.46	3	9	16
Performance attribute (in paste, mortar, concrete)				
Strength	1.14	25	2	1
Strength gain from 7–28 days	1.21	22	6	0
Setting time	1.25	22	5	1
Early age strength gain	1.25	21	7	0
Slump loss with admixtures	1.43	17	10	1
Slump loss with temperature	1.71	12	12	4
Drying shrinkage	1.72	10	12	3
Sulfate expansion	1.79	7	20	1
Volume changes	1.80	8	14	3
Bleeding characteristics	1.86	10	12	6
Air entrainment dosage	1.88	8	13	5
Finishing characteristics	1.96	8	13	7
Strength gain beyond 28 days	2.07	8	10	10
Autoclave expansion	2.07	5	15	7
Expansion in moist storage	2.12	5	12	8
FINE AGGREGATE UNIFORMITY				
Material property				
Grading including fineness modulus	1.18	23	5	0
Deleterious particles	1.43	18	8	2
Particles −200 sieve (amount and type)	1.50	15	12	1
Moisture content	1.68	16	5	7
Particle shape	1.82	8	17	3
Absorption	2.07	3	20	5
Specific gravity	2.18	4	15	9
Attrition (grinding during mixing)	2.29	4	12	12
Performance attribute (in paste, mortar, concrete)				
Water requirement	1.21	22	6	0
Air entertainment	1.36	20	6	2
Concrete strength	1.57	14	12	2
Water reducer effectiveness	1.96	6	17	5
COARSE AGGREGATE UNIFORMITY				
Material property				
Grading	1.18	23	5	0
Deleterious particles (amount and type)	1.46	17	9	2
Particle shape	1.68	10	17	1
Particles −200 sieve (amount and type)	1.75	11	13	4
Moisture content	1.82	14	5	9
Absorption	2.04	6	15	7
Attrition (grinding during mixing)	2.07	7	12	9
Specific gravity	2.14	5	14	9
Temperature	2.29	3	14	11
Chemical composition	2.32	3	13	12

(continues)

TABLE 1—Perceived Relative Importance of Materials Characteristics to Concrete Quality (ASTM C01/C09 1988 Survey) *(Continued)*

	Number of Responses[a]			
	Average	1	2	3
Performance attribute (in paste, mortar, concrete)				
Concrete strength	1.39	17	11	0
Water requirement	1.43	17	10	1
Freeze-thaw durability (D-cracking)	1.63	13	11	3
Drying shrinkage	1.82	7	19	2
Thermal vol. changes (cracking, etc.)	2.00	5	18	5
FLY ASH UNIFORMITY				
Material property				
Loss on ignition (L.O.I.)	1.11	25	1	1
Fineness	1.37	17	10	0
Variations in CaO	1.65	13	9	4
Variations in SO_3	1.76	11	9	5
Alkalies	1.78	10	13	4
Variations in SiO_2	2.15	7	8	11
Variations in Al_2O_3	2.27	4	11	11
Variations in Fe_2O_3	2.48	2	9	14
Specific gravity	2.26	3	13	10
Performance attribute (in paste, mortar, concrete)				
Required air-entrainment dosage	1.26	21	5	1
Reactivity with different cements	1.54	14	7	3
Time of set	1.58	13	11	2
Reactivity at different temperatures	1.71	12	7	5
Response to admixtures	1.81	9	13	4
Pozzolanic activity index	1.81	10	12	5
Shrinkage	2.00	3	18	3
WATER REDUCERS, HRWR, RETARDERS UNIFORMITY				
Material property				
Sensitivity to cement composition	1.30	19	8	0
Sensitivity to time of addition	1.48	15	11	1
Compatibility with other admixtures	1.48	16	9	2
Percent solids	1.59	15	8	4
Composition and concentration	1.59	15	8	4
Sensitivity to temperature	1.62	15	6	5
Variations in chlorides	1.78	9	15	3
Temperature stability (freezing, etc.)	1.81	11	10	6
Variation in alkalies (HRWR)	1.85	8	14	4
Stability in storage	1.93	8	13	6
Performance attribute (in paste, mortar, concrete)				
Time of set	1.11	24	3	0
Rapid stiffening	1.15	23	4	0
Early-age strength	1.41	17	9	1
Later-age strength	1.85	8	15	4
Finishing characteristics	1.93	10	9	8
AIR-ENTRAINING ADMIXTURE UNIFORMITY				
Material property				
Percent solids (specific gravity)	1.54	14	7	3
Composition (infrared spectra)	1.91	7	11	5
pH	2.00	7	10	7
pH in deionized water	2.17	3	13	7
Performance attribute (in paste, mortar, concrete)				
Stability of air with fly ash	1.11	24	3	0
Air void system characteristics	1.27	20	5	1
Sensitivity to cement composition	1.37	19	6	2
Sensitivity to temperature	1.44	16	10	1
Generation of air voids	1.50	16	7	3
Compatibility with other admixtures	1.63	14	9	4
Sensitivity to aggregate grading	1.78	10	13	4
Sensitivity to mix water composition	2.15	7	9	11

(continues)

TABLE 1—Perceived Relative Importance of Materials Characteristics to Concrete Quality (ASTM C01/C09 1988 Survey) *(Continued)*

	Number of Responses[a]			
	Average	**1**	**2**	**3**
LIGHTWEIGHT AGGREGATE UNIFORMITY				
Material property				
Unit weight	1.23	20	6	0
Absorption	1.27	19	7	0
Grading	1.38	16	10	0
Moisture content	1.38	19	4	3
Specific gravity	1.60	14	7	4
Particle shape	1.77	8	16	2
Attrition (grinding during mixing)	1.81	8	15	3
Performance attribute (in paste, mortar, concrete)				
Concrete strength	1.31	20	4	2
Shrinkage and volume changes	1.58	12	13	1
Air entrainment	1.85	10	10	6
Absorption of admixtures	1.96	8	10	7
SLAG UNIFORMITY				
Material property				
Fineness	1.24	19	6	0
Glass content	1.48	14	10	1
Variation in chemical composition	1.74	10	9	4
Specific gravity	2.12	5	12	8
Performance attribute (in paste, mortar, concrete)				
Activity index	1.25	19	4	1
Temperature	1.63	13	7	4
Required air-entrainment dosage	1.83	7	14	3
Required water reducer, HRWR dosage	2.00	7	10	7
Shrinkage	2.00	5	14	5
SILICA FUME UNIFORMITY				
Material property				
Composition	1.40	16	8	1
Percent solids	1.68	11	7	4
Stability in storage	1.73	11	6	5
Specific gravity	2.23	6	5	11
Performance attribute (in paste, mortar, concrete)				
Air entertainment and air void system	1.44	16	7	2
MIX WATER UNIFORMITY				
Material property				
Chloride content	1.52	16	5	4
Organics content	1.62	14	8	4
Alkali content	1.83	9	10	5
Sulfate content	1.92	8	11	6
Hardness	2.25	4	10	10
pH	2.29	3	11	10
Solids content	2.38	2	11	11
Performance attribute (in paste, mortar, concrete)				
Air entertainment	1.46	14	9	1
Time of set (Cl, Na_2CO_3)	1.72	10	12	3
Temperature	1.80	10	10	5
Durability (ASR, sulfate resist., etc.)	2.00	7	10	7

[a] 1 = important to 3 = unimportant.

In addition to comprehensive data that quantified potential strength uniformity, the PCA/NRMCA program provided extensive information on sampling and testing procedures, and correction factors for testing variations. Compilation of this information into a draft recommended practice document greatly facilitated the ASTM development process for the new standard. Thus, the first ASTM standard for uniformity of a concrete-making material, ASTM C 917, was approved in 1979, approximately two years after completion of the test program.

Even with approval of a document such as ASTM C 917, there is a continuing development and educational process that must take place to foster appropriate use. A uniformity standard should provide a communication tool between manufacturer and customer that will improve overall concrete quality and performance [2,8,9]. Education of both those providing and those using uniformity data is an important step once a standard is introduced [10,11]. A final test of the standard is whether it is used. Widespread adoption of ASTM C 917 has been slow [12,13]. However, with the increasing trend toward total quality management, use has been growing.

The early work by Walker recognized that concrete strength uniformity was not solely a function of cement strength uniformity and included discussion of such factors as sampling and testing variations, temperature effects, and age effects [3]. Batching, mixing, transporting, placing, and curing also have important implications. Walker also noted that strength was not the only concrete performance attribute of importance; he recognized constructibility and durability as other critical attributes [3].

Given that today's concretes are more sophisticated, it is appropriate to consider standardization of uniformity provisions for other constituent material properties and attributes that affect concrete constructibility, strength, and durability. The following section discusses a standard for determining uniformity of other characteristics of concrete-making materials that was developed by the ASTM C01/C09 Task Group and is now designated as ASTM C 1451.

Standard for Determining Uniformity of Concrete-Making Materials

Figure 4 illustrates the process for developing a uniformity standard. First, the critical property or attribute of the particular constituent material must be identified. Potential properties and attributes are listed in Table 1. It would be prohibitive and unnecessary to develop uniformity requirements for each specific property or attribute in Table 1, so those that are most critical must be selected. This is an appropriate responsibility for ASTM committees that govern standards for materials listed in Table 1.

Once the attribute has been identified for evaluation, the test method must be selected. In most cases, an appropriate ASTM method exists, particularly for material properties. However, for some attributes, standard procedures may need to be developed or existing procedures modified.

After test methods are selected, a comprehensive sampling and testing program to quantify uniformity potential and testing variability for the particular property should be conducted. This will provide data for a specific standard's provisions on sampling frequency and protocol, testing criteria, statistical corrections for testing variations, and reporting requirements. The development of comprehensive test data is considered an essential part of the process because it is necessary to define the effect of inherent variability of testing on the property or attribute being measured.

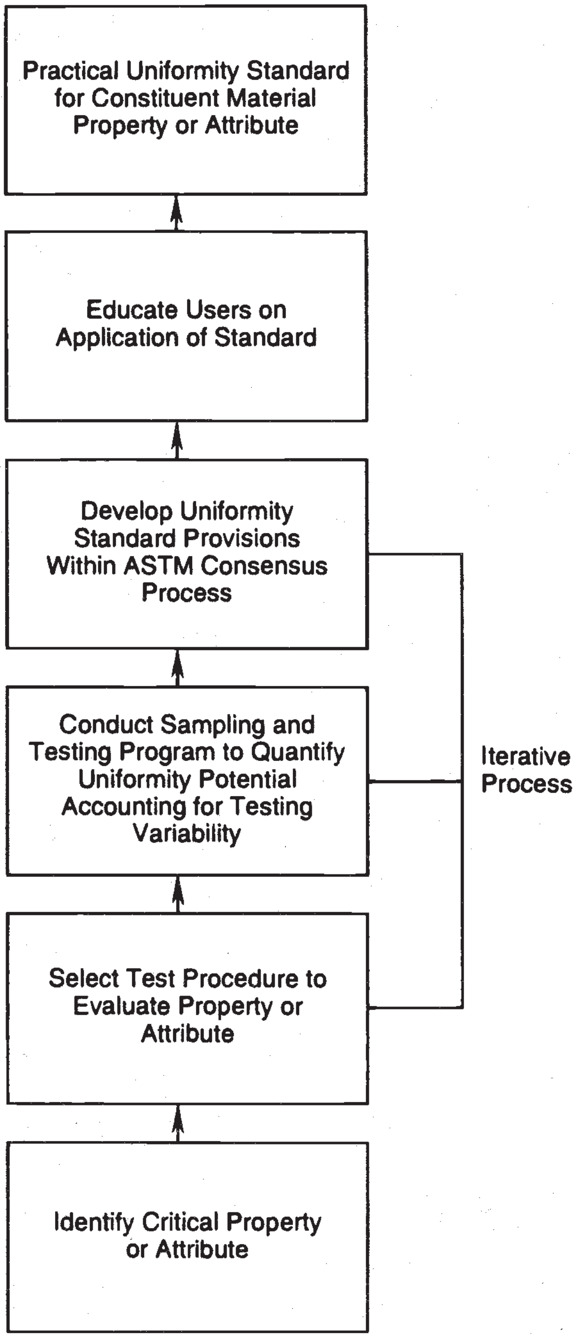

Fig. 4—Process for developing uniformity standards for concrete-making materials.

Testing errors can be significant, particularly for between-laboratory tests, and must be accommodated. Generally, single-laboratory testing is preferred for uniformity work. For example, as pointed out by Gaynor [12], the precision statement in ASTM C 109 implies that duplicate batches of mortar made in a single laboratory should give strengths that do not differ by more than 10.7 % 1 time in 20. However, single batches mixed by two different laboratories should not differ by more than 20.7 %.

ASTM C 1451 should speed the standards development process because the test program data for any property or attribute can be "plugged in" to a standard format. It addresses the following components for determining the uniformity of properties of a material from a single source:

1. Sampling
    - (*a*) Sampling is performed by trained personnel.
    - (*b*) Grab samples are taken at a frequency defined by maximum lot sizes.
    - (*c*) Sampling protocols are defined by existing ASTM standards.
    - (*d*) A minimum rate of sampling of ten per month or two per week is required.
2. Evaluation Procedure
    - (*a*) Samples are tested in accordance with standard ASTM procedures.
    - (*b*) Variations from a single source are corrected for variations inherent in the test procedures.
    - (*c*) Within- and between-laboratory testing variations are considered as required.
    - (*d*) Single-laboratory test variations are established by duplicate testing if no history is established.
    - (*e*) Between-laboratory variations are quantified by sample exchange or standard reference samples.
3. Statistical Calculations
    - (*a*) Equations are defined for average and total standard deviations of the measured values.
    - (*b*) Equations are defined for testing standard deviation and coefficient of variation.
    - (*c*) Equations are defined for standard deviation corrected for testing variations.
4. Report Requirements
    - (*a*) Reports identify materials tested.
    - (*b*) Reports cover a minimum of three and a maximum of twelve months.
    - (*c*) Reports include duplicate test results.
    - (*d*) Reports include specific statistical results for time period covered.

This format follows that of ASTM C 917 and provides a "generic" approach to accommodating any property or attribute.

As discussed here, even after the development of the consensus standard it will be necessary to educate users. This will be a continuing process. In addition, uniformity of concrete-making materials is necessary, but not sufficient, for obtaining uniform concrete performance. Field practices must also be addressed.

Summary

This chapter has addressed uniformity of concrete-making materials. Those properties and attributes of constituent materials that are considered to affect concrete uniformity are identified and a protocol for developing uniformity standards is discussed. With the growing sophistication of concrete mixtures, and the importance of total quality to concrete users, it is essential that the industry be prepared to provide uniform product performance. This will lead to increasing reliance on uniformity standards by suppliers of constituent materials and concrete producers.

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Virtual Testing of Cement and Concrete

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Introduction

CONCRETE IS A UNIQUE MULTI-PHASE, RANDOM, complex, and composite material that provides both strength and impermeability to engineered structures. Unlike most construction materials, the properties of concrete continue to develop over time and in place–an aid in processing, but a complication in testing and quality assurance. This is clearly exemplified by the bread-and-butter test of concrete quality compliance, the 28 day compressive strength test. Having to wait 28 days to assure performance compliance is another unique feature of concrete, and not necessarily an attractive one. This holds true both for the field engineer, who would prefer to directly proceed with further aspects of the construction, and the industrial or academic researcher, who would prefer to avoid waiting 28 days (or more) before truly knowing the effects of a new chemical additive or an alternative processing methodology. Thus, many efforts have been made to predict (and ensure) concrete performance based on waiting periods of less than 28 days.

One area of effort has been the development of computer models to predict cement and concrete properties via virtual testing. In these models, typically, starting materials are characterized in some fashion and this information is input into one or more computer models to predict a variety of properties of the fresh and hardening cement or concrete. If properties can be successfully predicted, the time and cost savings to the cement and concrete industry can be tremendous. Many companies spend millions of dollars per year on the testing of concrete, including material costs, storage, labor, and disposal. An additional benefit of virtual testing would be the capability to rapidly perform a large number of "what-if" type computations to explore new material systems and optimize existing ones. With this goal in mind, in January of 2001 a joint industry/government consortium was initiated to develop a web-based Virtual Cement and Concrete Testing Laboratory (VCCTL). An earlier version of the VCCTL, 1.1, is available for the general public at http://vcctl.cbt.nist.gov. The progress of the consortium can be seen in three annual reports [1–3], and in numerous examples of properties that can be successfully predicted using the VCCTL [4] and other existing models.

While at first glance, it may appear that virtual testing has the potential to eventually eliminate physical testing and its accompanying standards, the truth is that a functional VCCTL places a greater burden on testing and standards than ever before. First, model property predictions can only be as good as the characterization of the starting materials. Thus, standardized methods for preparing and analyzing these materials will be critical. And second, measurements of fundamental properties are needed in order to empower and validate the prediction models. The VCCTL will drive the standard tests to move from empiricism towards a firm materials science basis, not eliminate them. Standards and testing are critical for validating and extending the computer models. While current models can predict several properties that are already covered by existing ASTM C01 (Cement) and C09 (Concrete) standards, they can also quantitatively predict other properties for which no standard test method currently exists. Even though much work remains to be done on refining and extending these models, it is just as critical that the necessary standards work be pursued and completed. The dual objectives of this chapter, then, are (1) to show what current models can do, and (2) to highlight needs relative to existing and future standards for characterizing cement and concrete materials and for evaluating their properties.

What Really is Virtual Testing?

"Virtual testing" is an exciting name, given the ubiquity of "virtual" everything around us these days, but what does it really mean for cement and concrete materials? In the field of condensed matter physics, material properties are measured at a fundamental level. These measurements are then compared to quantitative predictions from condensed matter theory that is based on valid mathematical principles and atomic and molecular arrangements. "Simple" materials are usually studied. In materials science, more complex materials are studied, like random composites and biological materials. For many of these materials, it is not possible to carry out analytical calculations and so the field of computational materials science has been developed. All this entails is the usual condensed matter theory, but with the mathematics solved on a computer, since the problems are analytically intractable. Cement and concrete are complex, multiscale materials. The chemistry of cement is well developed and quite fundamental and analytically based, but analyzing and predicting the materials science properties requires a level of theory involving computational materials science. So the

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definition of virtual testing of cement and concrete is just the computational materials science of cement and concrete.

The ideal model for concrete would start from the known chemical composition of the material. Starting with the correct proportions and arrangement of atoms, it would build up the needed molecules, the nanostructure and microstructure needed, and eventually predict properties at the macroscale. Even with modern-day computers, however, this ideal model is a long way away. It is still not possible to systematically build up multiscale models starting from the atomic scale. The science-based virtual models that do exist need good data as input, data from careful characterization of materials. The models can predict physical properties of interest to actual materials users, but are based on fundamental parameters, not empirical tests. For example, measuring the Blaine fineness (using ASTM Standard Test Method for Fineness of Hydraulic Cement by Air Permeability Apparatus (C 204)) as a characterization of a cement does not give useful data for a model. On the other hand, careful measurement of the cement particle size distribution (PSD) does give information vital to the successful modeling of how the cement hydrates and develops microstructure. So the rise of virtual testing in this instance should drive the testing of Blaine fineness toward the measurement of cement particle size distribution. Analogous examples, in which virtual testing could push existing test methods toward more fundamental measurements, could be given in the areas of rheological properties and durability.

The Importance of Materials Characterization

The heart of concrete, as well as the heart of its virtual testing, lies in the cement. The VCCTL models start at the level of the cement particles. Specific needs are what cement particles look like, how the various chemical phases of the cement are distributed in the cement particles, what their particle size distribution is like, and what is their shape. By saying “cement,” gypsum is also included since it is almost always interground with portland cement. If there are other mineral admixtures, such as fly ash, ground granulated blast furnace slag, metakaolin, silica fume, or something else, the same kind of information is needed about them as well.

To characterize a cement so that one has a hope of predicting its hydration and general performance, one must first measure the particle size distribution using some method. By far the most popular method among cement companies and other laboratories is the laser diffraction method [5,6]. This technique uses optical laser light diffracting from the various cement grains to measure/compute an equivalent spherical diameter for each particle. This is critical for virtual testing, since smaller cement grains hydrate faster and more completely than larger cement grains, having more exposed surface area, a phenomenon that must be captured accurately for any model to have a hope of being accurate. A measurement such as Blaine fineness, while useful in conveying information about the relative fineness of the cement, does not give the distribution of sizes. The hydration performance of a cement couples more to the distribution of sizes, not just some measure of the average size. In modeling the hydration of a cement, a 3-D model of the particles themselves is built up, so an accurate size distribution is needed.

Accurate bulk measures of cement chemical phases using Rietveld analysis of X-ray diffraction data, combined with particle size distribution information, can probably capture most of the information needed for experimental characterization of a cement’s hydration properties. There is work in ASTM subcommittee C01.23 at present on replacing the inaccurate Bogue calculations with more accurate X-ray diffraction Rietveld measurements [7]. But the hydration of a cement takes place at the individual particle level, so to model the hydration, one must go to the particle level. Correct particle level information is needed as an input into the virtual hydration model, CEMHYD3D [8–10].

Particle level information includes the detailed breakdown, per particle, of the various clinker phases. In a cement, some particles are purely C_3S, for example, while others are a complex mixture of clinker phases. To acquire this information, a dry cement powder is mixed into an epoxy and cured. A polished section is prepared, and a combination of back-scattered electron scanning electron microscopy (SEM) and X-ray microprobe analysis are used to identify the chemical phase belonging to each pixel in the image. This procedure is being investigated for standardization within ASTM subcommittee C01.23. Figure 1(a) shows the ordinary gray-scale back-scattered electron SEM image for a portland cement, while Fig. 1(b) shows the image after all of the major chemical phases have been identified. More details on this procedure are available elsewhere [8,11,12]. This procedure can identify calcium sulfate, but cannot distinguish between its different hydrated forms. Quantitative X-ray diffraction or thermogravimetric analysis [13] can give this information in bulk, but not at the particle level. In addition to the measurement of bulk volume fractions, the final segmented SEM images can also be analyzed to determine phase surface fractions and autocorrelation functions. Along with a measured particle size distribution, these three characteristics can be utilized to reconstruct an initial three-dimensional microstructure of cement particles in water that is a very realistic representation of the specific cement in question [8,10]. This is a critical step in the successful simulation of cement paste microstructure and performance properties. Mineral admixtures need to be characterized in the same way. A few forms of fly ash and slag have been so characterized [14,15], though further research on better adapting the above characterization techniques to these materials will prove fruitful [16].

Cement particle shape is important, too. The characterization technique for cement particle shape is very similar to that for aggregates, so aggregate shape is considered first. Aggregates used in concrete, both fine and coarse, are of many different mineralogical types, and are either found naturally in the desired gradation, or are crushed to the desired sizes. Their shape, which is measured in the ASTM Standard Test Method for Flat Particles, Elongated Particles, or Flat and Elongated Particles in Coarse Aggregate (D 4971), can vary greatly. The amount of internal porosity and hence water absorption can vary as well. Their different mineralogical types imply that their elastic moduli will also vary. To make a virtual concrete, one must in general use realistic aggregates. For some properties, like chloride diffusivity, the shape of model aggregates used does not seem to matter much. For other properties, like fresh concrete rheology and mechanical properties, especially at early ages, aggregate shape means a lot. There are few standard tests that address aggregate shape (ASTM D 4971 and ASTM Standard Guide for Petrographic Examination of Aggregates for Concrete (C 295)), and none that attempt to characterize the full 3-D aspects of shape, which must be used to understand and predict the effect of shape on properties.

To be able to build models using real-shape aggregates, one must be able to treat a given aggregate particle as a

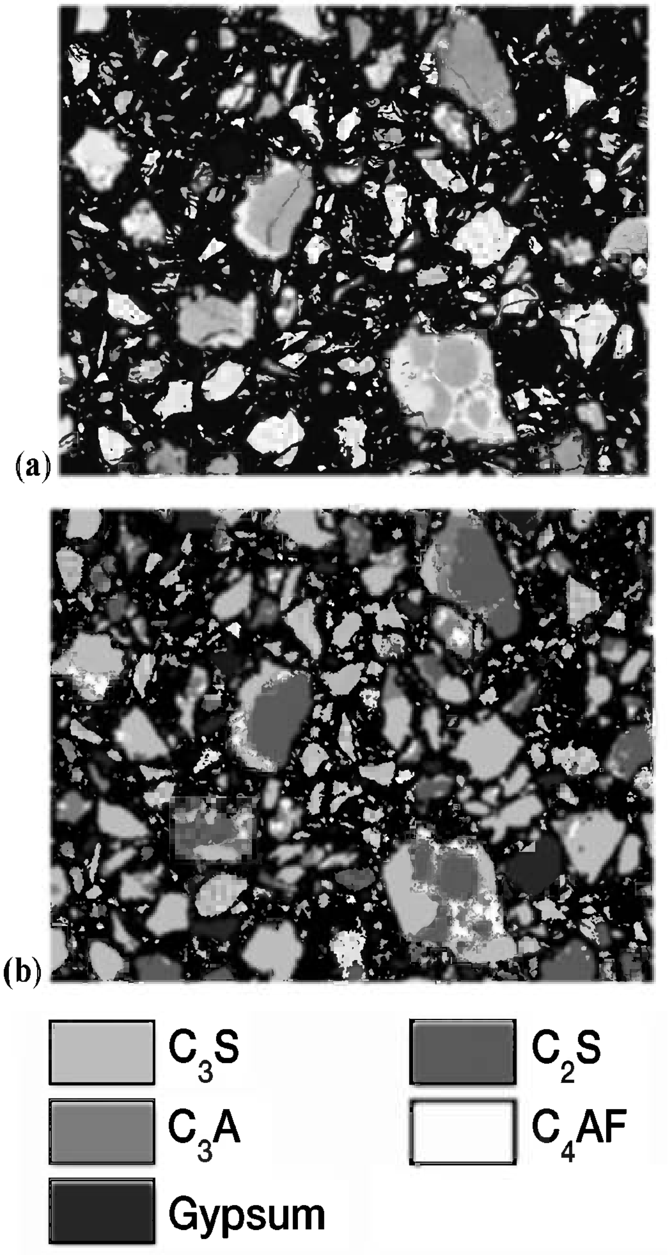

Fig. 1—(a) Backscattered electron SEM image of a cut and polished section of cement 140 from the Cement and Concrete Reference Laboratory proficiency sample program. (b) False gray scale image of the same section, based on X-ray microprobe analysis. The meaning of each gray scale is indicated in the accompanying legend. Images are 256 μm × 200 μm.

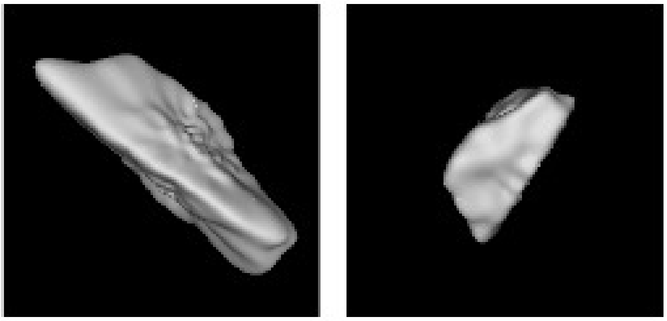

Fig. 2—Reconstructed images of aggregates sampled from AASHTO Materials Reference Laboratory (AMRL) proficiency sample #39. The left image is of a coarse limestone aggregate with an equivalent spherical diameter of 13.80 mm, and the image on the right is of a fine limestone aggregate with an equivalent spherical diameter of 1.46 mm.

mathematical object. Spheres are easy to so use, since there is a simple equation that defines their surfaces: the distance from the center of mass to the surface is a constant. Real-shaped particles can also be characterized in terms of mathematical functions, although this is a little more complicated. A recent paper [17] showed how a combination of X-ray computed tomography (CT) and spherical harmonic functional analysis could produce an analytical function for the surface of an arbitrary aggregate particle. Once this mathematical function is obtained, then real-shaped particles can be handled in a model with the same ease as spherical particles. Figure 2 shows a VRML (virtual reality modeling language) picture of a fine and a coarse limestone aggregate, from an American Association of State Highway and Traffic Officials (AASHTO) Materials Reference Laboratory (AMRL) proficiency sample. Aggregate databases are currently being built up for various aggregates and incorporated into the VCCTL.

To characterize cement particle shape requires X-ray microtomography, which can achieve the length scales of approximately 1 μm/voxel necessary to capture the shape of particles the average size of which are usually about 10 μm to 20 μm [18]. A voxel is a small cubic or rectangular parallelepiped-shaped element of a 3-D digital image. After such an image is obtained, the same methods used to characterize the shape of aggregates can be used for the cement particles [19].

Virtual Testing of the Rheology of Fresh Cement and Concrete

Rheology of concrete is the study of how concrete flows, before the setting point is reached. This is important because the concrete must be placed by some kind of pouring or pumping into the prepared forms. The slump test specified in the ASTM Standard Test Method for Slump of Hydraulic-Cement Concrete (C 143/143M) is an empirical measurement of how concrete flows in a given situation. However, work in the last few decades has shown clearly that concrete rheology is characterized by at least two parameters, yield stress and plastic viscosity. In a similar way, an elastic solid is characterized by two parameters, Young's modulus and Poisson's ratio. Typically, only the Young's modulus is considered, but really both are needed to completely understand and predict the elastic performance of an elastic solid. In the same way, the slump test only measures one parameter, the yield stress, but the plastic viscosity is needed as well to be able to fully understand and predict the rheology of concrete, which in turn determines the workability and flowability of the concrete into a form or through a pumping process.

Because concrete is a multi-scale material, investigating the rheology involves a multi-scale approach. Rheology of the

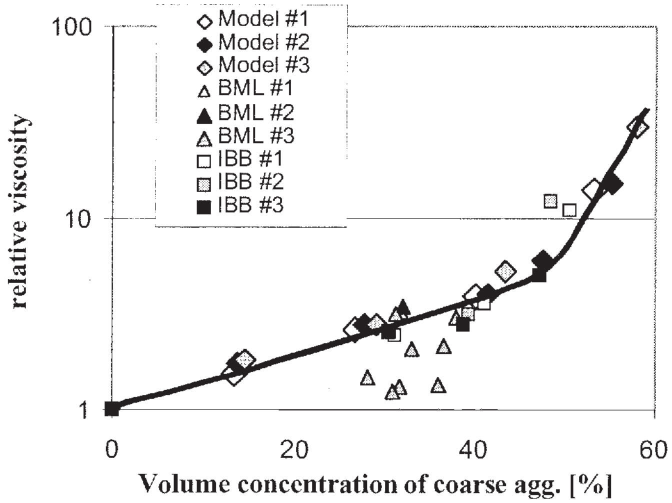

Fig. 3—Comparison of DPD model to experimental data on the dependence of relative viscosity of fresh concrete on the volume fraction of coarse aggregate. The first three sets of data in the legend are the simulations, and the remaining datasets are experimental data obtained using various rheometer types. See Ref [21] for detailed discussions of these concrete rheometer types.

cement paste greatly influences the time-dependent rheology of the concrete, and is itself non-Newtonian and complicated. However, having such a large volume fraction of aggregates, 60 % or more, also has a very large effect on concrete rheology.

Simultaneous modeling of the hydration process and cement paste rheology is beyond current computational capabilities. Therefore, we have taken a combined theoretical-experimental approach. Cement paste and mortar rheology is measured in a custom rheometer [20]. The effect of coarse aggregates on concrete rheology is modeled using a dissipative particle dynamics (DPD) approach. This is similar to a molecular dynamics approach for the movement of atoms and molecules, but adapted for coarse aggregate-size particles. The rheological properties of the matrix of the suspension come from the cement paste and mortar measurements. Figure 3 shows experimental and DPD simulated plots of the plastic viscosity of a concrete plotted versus the volume fraction of aggregate. Adding more aggregate clearly increases the apparent viscosity.

Experimentally, research is being done on measuring concrete rheology using various concrete rheometers. Several different designs are currently available. An intercomparison effort is underway [21,22], and it may be possible to extract fundamental rheological parameters (plastic viscosity and yield stress) from these measurements. This would both enable the rheological models to be validated and, more importantly, begin to allow fundamental rheological parameters to be used in analyzing and predicting concrete flow in field conditions. The DPD simulations can be used to analyze flow in different rheometer designs and extract fundamental parameters from empirical test results. There has also been DPD modeling work of concrete flow in self-compacting concrete (SCC) empirical tests, thus offering the potential for putting these tests on a more fundamental materials science basis. Some of these tests are currently being considered for standardization in ASTM subcommittee C09.47. Figure 4 shows a DPD simulation of coarse aggregate falling through four parallel rebars, similar in some ways to the SCC tests being considered for standardization. The coarse aggregate shapes were taken from the shapes of real aggregates, characterized by the process described in the previous section.

Virtual Testing of the Properties of Hardening Cement Paste and Concrete

Hydration and Degree of Hydration

To model the development of cement and concrete properties over time, a proper understanding (and model) of the hydration process is essential. While a complete understanding of cement hydration is still lacking, a significant knowledge base [23] has been accumulated in over 100 years of experimentation. The two most influential parameters for the properties of cement and concrete are water-to-cement mass ratio (w/c) and degree of hydration. If degree of hydration (of both cement and pozzolans) can be accurately predicted, many properties can be conveniently computed, as will be demonstrated in the subsections that follow. To model the increase in degree of hydration with time, two approaches are possible, depending on whether or not the underlying (blended) cement paste microstructure is explicitly considered.

The first approach consists of utilizing some functional form to describe the relationship between degree of hydration and time. The dispersion models of Knudsen [24,25] or the use of nuclei-growth models [26] are examples of this approach. In this approach, the parameters obtained from fitting the experimental data may or may not have physical significance. In addition, prediction of the performance of other systems (change in cement composition, PSD, w/c, etc.) based on the fitting of one (or more) set(s) of experimental data may or may not be possible. Still, these equations can be useful, as they usually do provide an excellent fit to an individual experimental dataset, and thus could be used to predict later-age degree of hydration from early-age measurements.

The second approach attempts to make greater use of the detailed characterization of the starting materials described above by directly modeling the microstructure development of the cement paste. In this approach, cement PSD, phase composition and distribution, and w/c can all be explicitly considered. In the last 15 years or so, significant developments have been made in this approach, concurrent with the vast increases

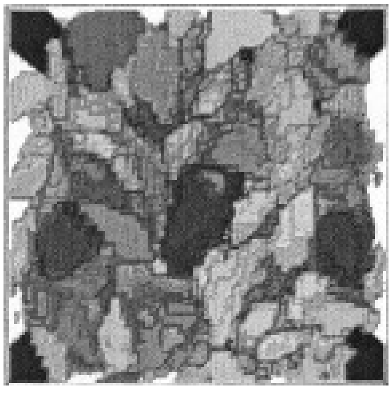

Fig. 4—Static image from simulation of vertical flow of a concrete with real particles through a grid of four steel reinforcing bars which are separated by a distance of 200 mm. The particles are dropping down, due to gravity, through the rebars.

in computer speed and available memory. A pioneering effort in this field was the work of Jennings and Johnson [27], who modeled the cement powder as a collection of spherical particles of tricalcium silicate that resulted in the formation of calcium hydroxide and calcium silicate hydrate reaction products, while explicitly considering the PSD of the powder. Similar models continue to be developed and utilized to this day [28,29]. In the early to mid 1990s, three other research groups, all active at the present date, initiated research on this topic. In Japan, Maekawa et al. developed the DuCOM (durability model of concrete) model, including modules for hydration, microstructure formation, and heat and mass transport [30]. The underlying hydration model, while focused on heat generation and water consumption, also considers the stereological aspects of the overlapping hydrating cement particles. A demonstration of the model is accessible over the Internet at http://concrete.t. u-tokyo.ac.jp/en/demos/ducom (accessed March 2005). In the Netherlands, van Breugel published his Ph.D. thesis [31] on the HYMOSTRUC model for the hydration and microstructure development of cement paste. This model considers the cement particles to be spherical and explicitly analytically accounts for the overlap of hydration products as the individual cement particles expand during the hydration process. The model has been applied by van Breugel and his subsequent Ph.D. students to predict a variety of cement and concrete properties, including heat development, strength development, and autogenous shrinkage [31–33].

During the same time period, Bentz and Garboczi initiated a cement hydration modeling effort [34] at the National Institute of Standards and Technology (NIST) that ultimately culminated in the CEMHYD3D three-dimensional cement hydration model [8–10]. Unlike the other models mentioned previously, CEMHYD3D considers the cement particles at the sub-particle level, applying a digital-image-based approach to the modeling of cement hydration and microstructure development. Each digitized spherical cement particle is composed of one or more digital elements (voxels) that can be assigned to be any of the possible phases of the starting cement powder. Using computational algorithms, the user is able to create an initial three-dimensional cement (in water) microstructure that matches the following measured features of the real cement powder, obtained via the characterization methods outlined earlier in this chapter: PSD, bulk phase composition, and intraparticle chemical phase distribution. An image of a portion of a typical starting 3-D microstructure is provided in Fig. 5.

In CEMHYD3D, a cellular automaton-like computer algorithm is used to simulate the hydration process. Individual cement phase voxels can dissolve, diffuse within the available capillary porosity, and react to form solid hydration products. The model proceeds as a series of dissolution/diffusion/reaction cycles, and a single user-provided parameter is used to approximately convert between hydration cycles and real time. The algorithms are applied so as to maintain the correct volume stoichiometry for the generally accepted reactions of cement hydration [8,10,23], including the chemical shrinkage first highlighted by Le Chatelier over 100 years ago [35]. Degree of hydration, of the cement powder or any combination of the original cement phases, can be determined by a simple voxel counting algorithm, either on a mass or a volume basis. CEMHYD3D has been utilized, worldwide, by cement researchers to predict performance properties and to generate microstructures for the subsequent evaluation of degradation [36–41]. In addition to cement hydration reactions, the most recent version of CEMHYD3D also includes reactions for a variety of mineral admixtures such as silica fume, fly ash, slag, and limestone [1–3]. Figure 6 provides an example two-dimensional image of a portion of a hydrated CEMHYD3D microstructure.

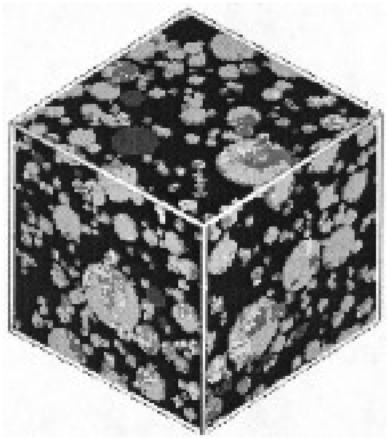

Fig. 5—Typical 3-D reconstructed microstructure used as input for the CEMHYD3D cement hydration model. The phase designated by each gray scale is shown in the legend accompanying Fig. 6.

Experimentally, no ASTM standard method exists for evaluating the degree of hydration of either portland or blended cements. For portland cements, non-evaporable water content [42] is one generally accepted method for estimating the degree of hydration of the hardened paste. In this test, the mass loss of the hydrated cement paste occurring between 105°C and 1000°C (or another similar range of temperatures depending on the preference of the researcher) relative to the mass of the ignited paste (corrected for the loss on ignition of the original cement powder) provides a measure of the non-evaporable water content. Degree of hydration (0.0–1.0) is then obtained by dividing the non-evaporable water content of the specimen by that measured for a fully hydrated cement paste specimen. The value for a fully hydrated specimen can also be estimated based on the potential Bogue composition of the cement using coefficients provided by Molina and given in Table 1 [43] (the effect of the more correct composition as determined by Rietveld X-ray diffraction analysis is not known at

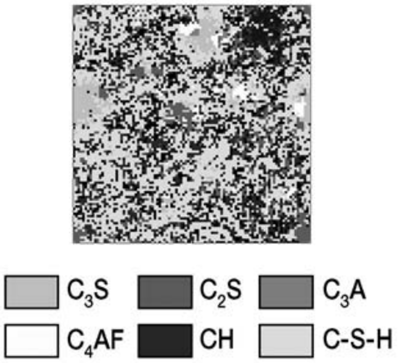

Fig. 6—2-D cross section of a 3-D model cement paste with w/c = 0.4, hydrated for 2000 computational cycles using the CEMHYD3D hydration model. The phase designated by each gray scale is indicated in the accompanying legend. The image is 100 μm × 100 μm.

TABLE 1—Non-evaporable Water Contents for Major Phases of Cement

Cement Phase	Coefficient (g of water per g of phase)
C_3S	0.24
C_2S	0.21
C_3A	0.40
C_4AF	0.37
Free lime (CaO)	0.33

present). For Type I portland cements, a typical value is on the order of 0.23 g of water per gram of cement. For blended cements, non-evaporable water content is no longer an accurate measure of degree of hydration due to the confounding influence of the pozzolanic reactions, which consume calcium hydroxide (and its accompanying non-evaporable water) in producing other cement hydration products [44,45]. The use of SEM/image analysis (e.g., point counting) to estimate the degree of hydration of both portland and blended (with fly ash or slag) cements appears to be a promising new option [46]. Clearly, this is an area where further standardization efforts are needed.

Figure 7 provides a comparison of VCCTL-predicted and experimentally measured (via non-evaporable water content) degrees of hydration for Cement and Concrete Reference Laboratory (CCRL) cement 135 with a w/c = 0.40, hydrated under both saturated and sealed curing conditions [47,48]. Under sealed curing conditions, the paste self-desiccates due to the chemical shrinkage that occurs during hydration, which also decreases the achieved degree of hydration at later ages. It can be observed that the computer model provides an excellent fit to the experimental data for both curing conditions, using a parameter of 0.0003 h/cycle^2 to convert between cycles and time.

Setting Time

Setting time is one of the most important properties of a cement, as it will determine how much time is available to place and finish the concrete. Two ASTM standards exist for the evaluation of setting time: ASTM Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle (C 191) and ASTM Standard Test Method for Time of Setting of Hydraulic-Cement Paste by Gillmore Needles (C 266). The two usually provide slightly different values, with the Gillmore needle times for initial and final set generally being slightly greater than those obtained using the Vicat needle. Both methods basically measure when the hydrating cement paste develops some finite value of resistance to penetration.

To predict setting times using a computer model, one first needs to decide what constitutes the physical process of setting. Fundamental research has indicated that setting is normally controlled by the formation of calcium silicate hydrates (bridges) that link together the original cement particles [49,50]. In systems that undergo a rapid reaction of the aluminate phases and flash set, it is likely the aluminate hydrates that form these linking bridges. Within the VCCTL software, setting is assessed using a specialized percolation algorithm [10]. The algorithm measures the fraction of total solids (mainly cement particles at this point) that are linked together by calcium silicate hydrate gel, ettringite, and calcium aluminate hydrate hydration products. Thus, two touching cement particles are not considered to be connected, for determining setting, unless some hydration product bridges them. When such a structure spans the entire system, one says that the structure is percolated or connected or, in this case, set. In this way, the setting behavior of both well-dispersed and flocculated cement pastes can be consistently evaluated. The VCCTL program returns the percolated (connected) fraction [0,1] for the total solids as a function of either hydration time or degree of hydration.

Figure 8 shows these percolation plots versus time for CCRL cements 135 and 141 [48,51], both hydrated at a w/c determined by the ASTM Test Method for Normal Consis-

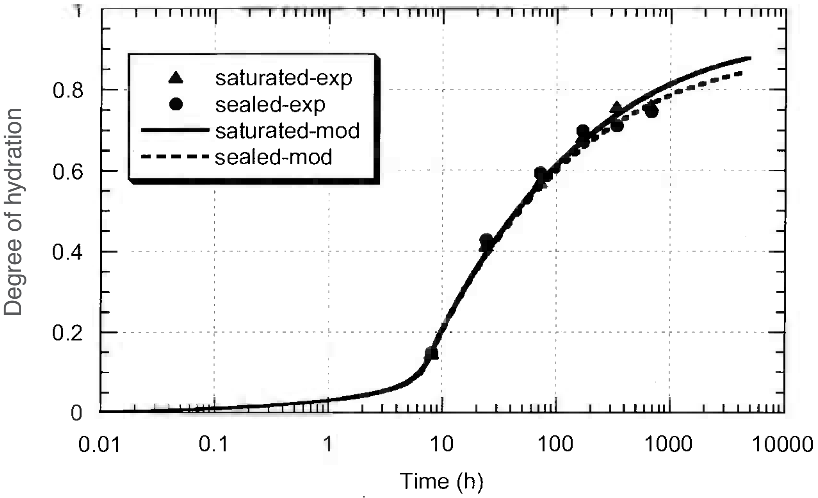

Fig. 7—Degree of hydration as a function of time for CCRL cement 135 with w/c = 0.4, cured isothermally at 25°C. The solid curve is the prediction by CEMHYD3D using saturated curing conditions, and the dashed line is the prediction using sealed curing conditions. The symbols are experimental measurements, based on nonevaporable water content, using saturated and sealed curing conditions, respectively. The error bars indicate ± one standard deviation from the mean value, and are about the same size as the data symbols.

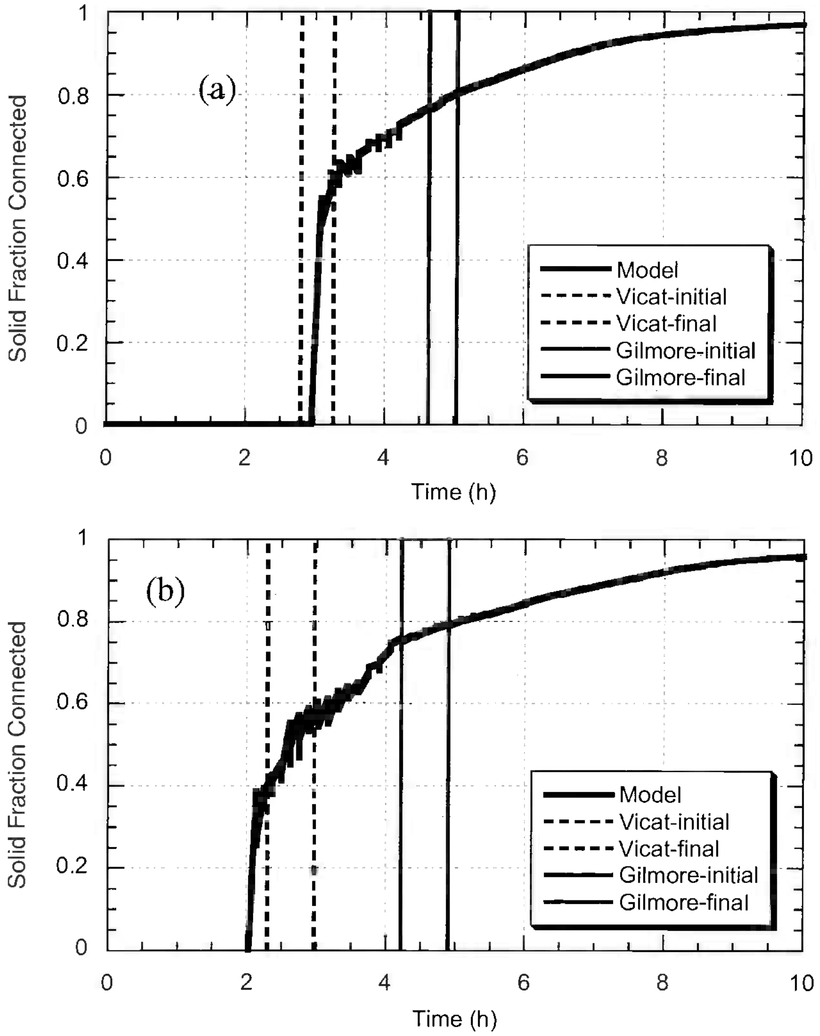

Fig. 8—Fraction of solids connected as a function of time for (a) CCRL cement #135, w/c = 0.25, and (b) CCRL cement #141, w/c = 0.267. The w/c ratio of both cements was determined by the ASTM Normal Consistency test (ASTM C 187). The fluctuations in the model curves is an indication of the randomness of the model due to its relatively small size. The vertical lines in each plot mark the measured initial and final setting times using the Vicat needle test and the Gilmore needle test. The sharp rise in the curves gives an indication of the setting times predicted by CEMHYD3D.

tency of Hydraulic Cement (C 187). The initial and final setting times as determined experimentally by both the Vicat and Gillmore needle tests are shown as vertical lines on the graphs, as noted in the caption. For the Vicat needle, the initial and final setting times are shown to approximately correspond to percolated fractions of 0.4 and 0.75, respectively. The Gillmore setting times are slightly longer, with the initial and final setting times corresponding to percolated fractions of 0.6 and 0.8, respectively. Setting time will be strongly influenced by the w/c of the cement paste and the PSD of the cement powder. As would be expected, coarser cements require more hydration time to achieve set, due to their slower hydration rate. But, these coarser systems actually achieve set at a lower degree of hydration, as fewer interparticle bridges are needed to percolate the microstructure in a coarser particle system [52].

Chemical Shrinkage

In addition to being identified by Le Chatelier over 100 years ago, the measurement of chemical shrinkage was also one of the first subjects investigated by Powers early in his career [53]. Because the cement hydration products occupy less volume than the starting materials (cement and water), a hydrating cement paste will imbibe water in direct proportion to its ongoing hydration [53,54]. This holds true except for low w/c pastes (less than w/c = 0.4) after a few days of curing, in which the depercolation of the capillary porosity may dramatically reduce the permeability of the cement paste and limit its imbibition rate below that required to maintain saturation during the continuing hydration [8,54]. Chemical shrinkage has been shown to be in direct proportion to other measures of degree of hydration such as non-evaporable water content and heat release [8,55]. It appears to provide a rapid method for assessing

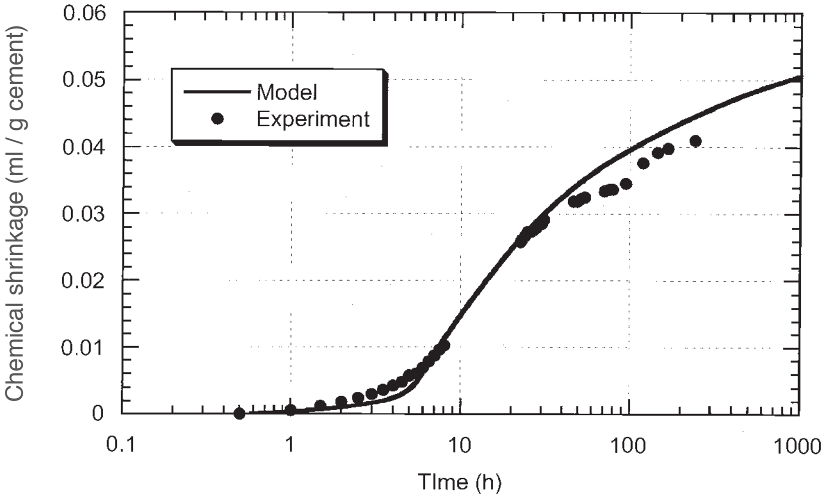

Fig. 9—Chemical shrinkage of CCRL cement 135 with w/c = 0.3, hydrated isothermally at 25°C under saturated curing conditions. The solid curve shows the prediction using CEMHYD3D, and the circles are experimental measurements.

early hydration rates and may provide a means for evaluating cement cracking susceptibility [56]. While no standard method currently exists for the measurement of chemical shrinkage, ASTM C01.31 subcommittee on Volume Change is currently balloting a draft standard for this test.

Knowing the volume stoichiometry (e.g., molar volumes) of all ongoing hydration reactions, it is straightforward to compute chemical shrinkage in the VCCTL CEMHYD3D model. Figure 9 provides a comparison of model and experimental results for CCRL cement 135 with w/c = 0.3. The deviation between model and experimental results at later ages (> 40 h) is likely due to the depercolation of the capillary porosity mentioned above, as excellent agreement is observed between model and experiment up until this time.

Heat Release and Adiabatic Temperature Rise

Another convenient measure of degree of hydration of portland cements is heat of hydration. Heat of hydration is conventionally measured using the ASTM Standard Test Method for Heat of Hydration of Hydraulic Cement (C 186). Within the CEMHYD3D computer model, heat of hydration is computed based on the individual heats of hydration of the various cement clinker (and pozzolan) phases. Knowing the degree of hydration of each individual cement phase, the overall heat of hydration is easily computed. For adiabatic boundary conditions, this heat of hydration can be readily converted to an adiabatic temperature rise, if the heat capacity, specific gravity, and mixture proportions of the concrete are known [36]. Figure 10 provides an example of the

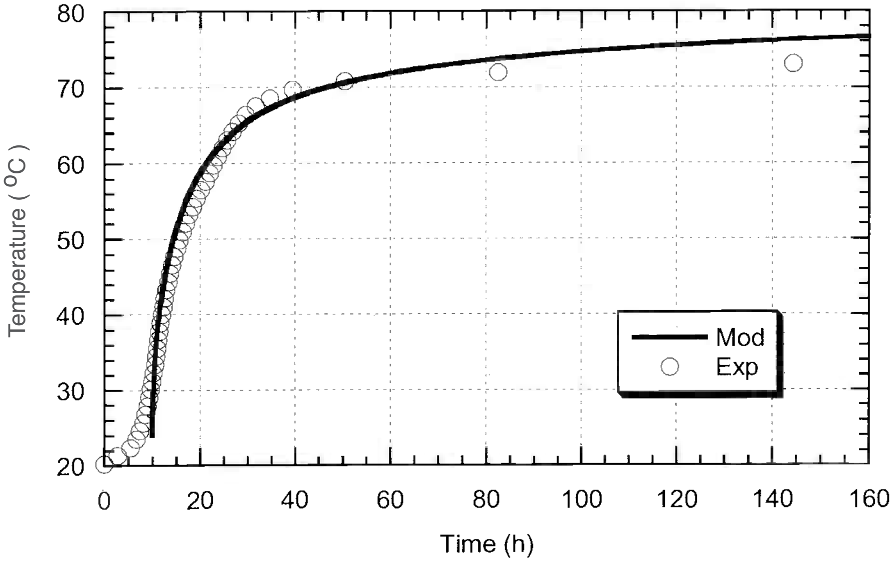

Fig. 10—Comparison of predicted (solid line) and experimental measured (data points) adiabatic heat signature curves for a w/c = 0.65 OPC concrete with 50 % fly ash replacement of cement, by mass.

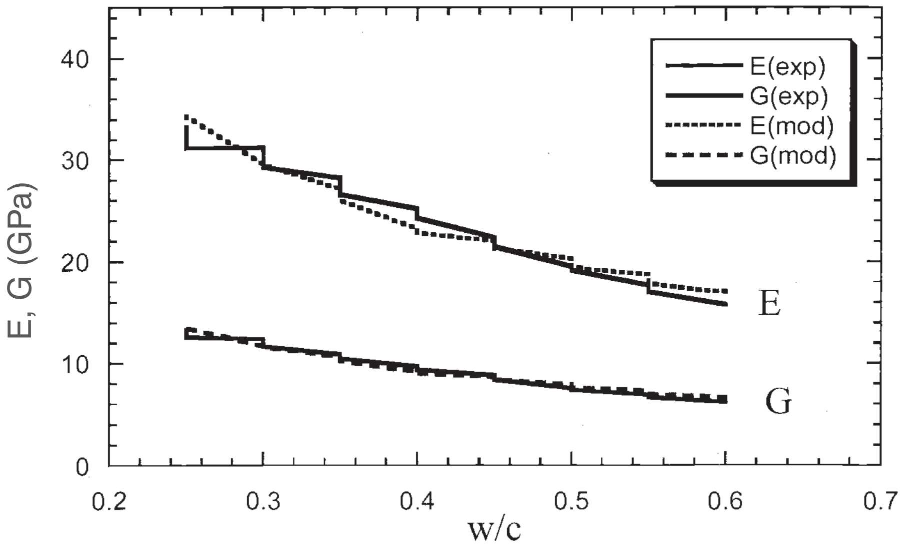

Fig. 11—Comparison of elastic moduli predictions to experimental data, versus w/c ratio, for 28-day and 56-day specimens. At each w/c ratio, the upper point is the 56-day value and the lower point is the 28-day value.

generally observed agreement between model and experimental data for the adiabatic temperature rise of a blended cement concrete.

Virtual Testing of the Properties of Hardened Cement Paste and Concrete

Elastic Moduli

As stated above, the output of the CEMHYD3D model is a digital hydrated 3-D cement paste microstructure, in which each voxel is occupied by a unique cement paste phase. By treating each voxel as a tri-linear finite element, the overall elastic moduli of the cement paste model can be computed directly using finite element techniques. A typical model is 100^3 voxels in size, so that a conjugate gradient relaxation solver must be used [57–59]. This size system can routinely be done on a modern desktop in an hour or less.

Recently, a careful test of this algorithm applied to the microstructures resulting from operation of CEMHYD3D was carried out [60]. Cement paste samples were prepared from a German cement, for w/c ranging from 0.25 to 0.6. After periods of 28 days and 56 days of curing, the degree of hydration was measured using loss on ignition (a method described above), and the elastic moduli were measured using a version of the ASTM Standard Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequencies of Concrete Specimens (C 215). Figure 11 shows the comparison between model and experimental results for 28-day- and 56-day-old samples, graphed as a function of w/c. There is excellent agreement between the virtual and experimental results.

Compressive Strength

Actually, compressive strength is a more highly utilized parameter in the cement and concrete industries than are elastic moduli. Therefore, prediction of the compressive strength of mortar cubes and concrete cylinders is an obvious application for computer modeling. The compressive strength of mortar cubes is generally assessed based on the ASTM Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (using 2-in. [50-mm] cube specimens) (C 109/ C 109M), while that of concrete cylinders is assessed according to ASTM Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens (C 39/C 39M). Computationally, several approaches are possible for the prediction of compressive strength. The most widely used to date has been Powers's gel-space ratio theory [61]. In the CEMHYD3D model, voxel counting is utilized to calculate the volume fractions of "gel" and "space," and compressive strength is estimated as a function of the ratio of the two. Since a pre-factor is present in the strength prediction equation, at least one experimental measurement of compressive strength is required to estimate the pre-factor. Usually, either a 3-day or a 7-day strength measurement is performed and the resulting pre-factor is used to predict the 28-day strength. While originally developed for portland cement systems, it has been recently verified experimentally that the theory can also be applied to blended cement (specifically fly ash) systems [62]. The theory has also been used in commercially available software packages that predict compressive strength development based on the measurement of heat signature curves (as a measure of degree of hydration) [63]. Figure 12 provides an example of the agreement between model predictions and measured compressive strengths for CCRL cement 133 [10]. While it would be preferable to directly predict compressive strength development without requiring an early-age measurement, the above procedure could still result in considerable time and cost savings to the industry, as it reduces the 28-day evaluation window down to either 3 days or 7 days.

The previous subsection has illustrated that elastic moduli development can be accurately predicted using the VCCTL software. Another approach to predicting compressive strength, currently being pursued within the VCCTL consortium, is to first compute elastic moduli, and then estimate compressive strength based on some functional relationship between compressive strength and elastic modulus. A convenient empirical equation relating Young's modulus and compressive strength, based on many experimental results, is found in the ACI standards [64]. It is possible that a multiscale strength of materials theory can be formulated to give accurate, microstructure-based predictions of compressive strength via similar kinds of equations that are microstructurally based.

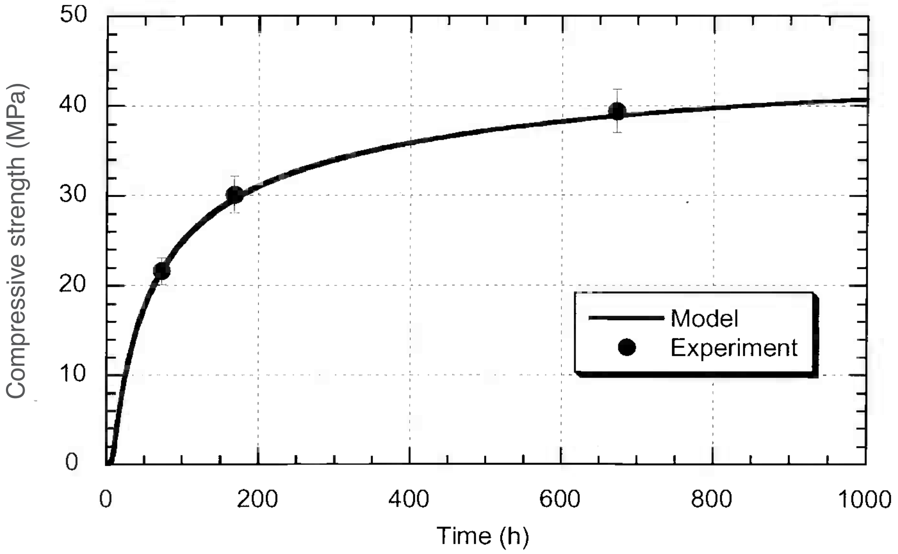

Fig. 12—Experimentally measured (circles) and model-predicted compressive strengths for ASTM C 109 mortar cubes prepared from CCRL cement 133 at 25°C. The error bars indicate ± one standard deviation from the mean, as determined in the CCRL testing program.

Diffusivity

As more and more concrete structures are designed for durability as well as strength, transport properties such as diffusivity are becoming more important. In the past, ASTM Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration (C 1202) has been used extensively to measure the "rapid chloride permeability" of a concrete cylinder. Even though the results of this test are known to be confounded by the conductivity of the pore solution and various temperature effects [65], it is still widely specified within the industry. While for a set of similar materials, the test method may produce the correct performance ranking, using the test as a performance criterion for conformance to specifications is a dangerous but ever-growing practice. The newly approved ASTM Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion (C 1556) is a welcome addition that will provide diffusion coefficients that can be subsequently used to obtain service life estimates for concrete structures. However, one has to be careful in evaluating chloride diffusion through a strong ionic electrolyte such as cement paste pore solution, and so it is possible that more refinements will have to be made [66,67].

For the case of diffusivity, successful predictions at the cement paste level have been also successfully extended to mortar and concrete [68,69]. For this application, a hard core/soft shell (HCSS) microstructural model of concrete has been developed [70]. The concrete is modeled as a three-dimensional continuum volume of spherical aggregates, in which each aggregate particle is surrounded by an interfacial transition zone (ITZ). The diffusivity of the diffusing species is different (generally higher) in the ITZ regions than in the bulk cement paste. Random walker techniques are then employed to determine the diffusivity of the overall 3-D model concrete microstructure [71]. The HCSS model is currently being extended to use real-shape aggregates instead of model spheres.

Using this approach, it has been determined that the major variables influencing concrete diffusivity are w/c, degree of hydration, aggregate volume fraction, and silica fume addition rate (other mineral admixtures have not been considered) [68,69]. Thus, equations have been developed to predict concrete diffusivity for limited ranges of these four input parameters. It must be recognized that these equations ignore a number of field concrete realities such as (micro)cracking, partial saturation (drying), differential curing, and leaching (of calcium hydroxide) from the surface layer. While they may provide adequate predictions of the diffusivities of laboratory concretes produced under carefully controlled conditions, they should be used with caution in field applications.

Modeling the Degradation and Service Life of Hardened Cement and Concrete

An important area for virtual testing is durability, as durability-type tests are usually time consuming. Degradation mechanisms at the microstructural level have been considered, such as leaching of calcium [72], how leaching affects diffusion rates via changes in the amount and connectedness of the capillary pore space [73–76], and how leaching affects elastic moduli via the dissolution of solid phases [41,77]. Virtual durability testing is still in its infancy, however, although it will become a major area of focus as time goes on.

Prospectus and Future Directions

The previous sections have illustrated the wide variety of cement and concrete properties that can be predicted and studied using computer modeling. As computer modeling slowly moves into the industrial mainstream, new and diverse applications surely will be discovered. In addition, it has been clearly shown that standards and computer modeling can and should function in a synergistic relationship. The computer modeling efforts of recent years have clearly pointed out the need for new materials characterization standards and test methods. The models' performance depends critically on high quality input concerning the materials in question. Many of the needed new standards and test methods are already being worked on by the appropriate ASTM subcommittees.

High quality quantitative standards are also needed for the experimental validation of the computer models. Experimental validation is critical both during model development and during their extension to new systems/environments. The models can only be proved, disproved, and improved based on comparison to carefully measured quality experimental results. As the above sections indicate, computer modeling has exhibited the potential to predict a wide variety of cement and concrete properties, several of which are lacking a standard test method for obtaining the necessary experimental data. While degree of hydration, chemical shrinkage, and heat of hydration are all fundamental measures of the reactivity of a (blended) cement system, only heat of hydration is covered by an existing standard (ASTM C 186). Even this standard only provides a measure of heat of hydration at limited fixed times such as 7 days and 28 days. Both chemical shrinkage and isothermal calorimetry can provide nearly continuous monitoring of the early hydration rate of cement-based systems. For both of these measures, ASTM standard test methods would be a welcome addition to the research, production, and end-user communities. While degree of hydration by non-evaporable water content (for portland cement systems) or SEM point counting (for portland cements and those blended with slag and fly ash) typically provide measures of reactivity at limited points in time, standardization efforts for these two measures are also critically needed.

Standard test methods are lacking in a number of other areas. The results of virtual testing clearly point this out, by computing values of important parameters for which no standard measurement is available. Examples include measurement of internal relative humidity in hardened concretes, measurement of elastic moduli and creep of concrete at early ages, and mechanistic-based tests for common degradation problems such as sulfate attack and freeze-thaw degradation. In addition to its usage to predict performance properties, computer modeling should also prove invaluable in providing guidance and direction for these new standards development efforts.

Conclusion

Advances in ceramics, metals, and polymers have been driven by the development of sufficient theory to help explain puzzling experimental results and point the way to new experiments that had not been thought of previously. Theoretical (or virtual) understanding of the material gave material designers a whole new level of control over their material, leading to improvements that opened up new opportunities. Virtual testing for cement and concrete can do the same thing for these materials. Virtual testing will drive the development of empirical physical testing towards materials science-based measurements that will in turn empower the material models to have even greater predictive power and usefulness. Rightly viewed, physical testing and virtual testing are complementary, not competitive. But the development of virtual testing will drive changes in physical testing–there is no doubt about that.

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7

Quality Cement, Concrete, and Aggregates—The Role of Testing Laboratories

James H. Pielert[1]

Preface

THE CONTRIBUTIONS OF J. J. WADDELL AND J. R. DISE, who prepared the chapters on Laboratory testing in *ASTM STP 169A* and *ASTM STP 169B*, respectively, are acknowledged.

Introduction

Concrete construction, which is vital to the nation's economic health, makes up a significant portion of the more than $900B spent annually on construction. Field testing and inspection (including sampling) and laboratory testing of concrete and concrete-making materials are key activities in the construction process. The results of these activities are important to building officials, contractors, concrete producers, owners, and architectural and engineering firms in determining whether the qualities of the materials in the construction comply with contract documents. The role of field and laboratory testing in promoting the quality of construction and protecting the public safety is receiving added consideration, both nationally and internationally. The evaluation of testing laboratories by outside organizations plays a vital role in providing the construction community with confidence in the quality of testing.

This chapter discusses the role of laboratory testing in assuring the quality of in-place construction through testing and inspection activities. Specific areas discussed are the role of standards-developing organizations in the United States in preparing standards which promote quality testing; the implementation of quality system concepts in testing laboratories; the role of evaluating authorities, including accreditation bodies, in recognizing the competency of testing laboratories; and the existing programs used to recognize the competency of laboratory technicians.

Concerns in Testing

The testing of concrete and its component materials consists of sampling, performing specified tests, and reporting test results. The procurement of representative specimens through standard sampling procedures is a critical step in the testing process–poor specimens and faulty sampling techniques will defeat the purposes for which the tests are made. A laboratory cannot produce satisfactory information if the samples it receives do not represent the material under consideration either because they have not been taken with appropriate care, or have been altered by mistreatment in initial storage, curing, or shipment. Suitable detailed sampling procedures are described in standards for concrete and concrete-making materials prepared by the American Society for Testing and Materials (ASTM). Comparable national standards are also prepared by the American Association of State Highway and Transportation Officials (AASHTO) for highways and bridge construction. Among other requirements, adequate training and instruction of sampling personnel is essential. Supervisors at all levels must also be fully acquainted with sampling plans and procedures and recognize their responsibility for ensuring that the plans and procedures are rigorously followed.

Concrete specifications enumerated in construction contracts are often based on the results of test methods developed by ASTM. For example, the Building Code Requirements for Structural Concrete of the American Concrete Institute (ACI 318), which is frequently referenced, specifies that tests of concrete materials and of concrete must be made in accordance with ASTM standards [1]. No matter who the originator of the chosen standards may be, it is imperative that every effort be made to avoid the use of any but the latest testing techniques. Employment of an unsatisfactory procedure is potentially dangerous because it can lead to erroneous conclusions about the characteristics of the concrete and the safety of the structure.

In studying quality assurance for highway construction materials, variance in quality of the product can be divided into material or process variance, sampling variance, and testing variance. In a study conducted by the Bureau of Public Roads in the late 1960s, it was found that 50 % or more of the overall variance could be attributed to two of these factors: sampling and testing [2]. These two processes must be constantly monitored if reliable results are to be obtained.

On completion of testing, a laboratory is usually required to submit a written report to its client. The report should be

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complete and factual. According to ASTM Standard Practice for Laboratories Testing Concrete and Concrete Aggregate for Use in Construction and Criteria for Laboratory Evaluation (ASTM C 1077), the report should cite the name and address of the laboratory, report identification and date issued, name of the client and project identification, sample identification, identification of the standard test method(s) and known deviations from the standard, test results, and other information required by the appropriate standard [3]. This may include comments about any unusual aspects of the appearance or behavior of specimens that might in any way be relevant to the interpretation of the results obtained.

When a final report is available, it can be determined by the laboratory or another representative of the client whether a material complies, or fails to comply, with specification requirements [4]. If there is a question about the sampling or testing methods used to generate results recorded in the report, then the reliability of the entire report should be questioned.

Trends in Improving and Promoting Quality of Testing

ASTM Committee E36 on Laboratory and Inspection Agency Evaluation and Accreditation has responsibility for preparing standards related to testing laboratory quality. In some technical areas, the work of the committee stimulated the drafting of similar international standards, while in other areas, cognizance of international work has had a beneficial influence on ASTM standards. Relevant standards prepared by Committee E36 have done much to raise all aspects of the quality of testing. They are as follows [5]:

- ASTM Specification for Agencies Engaged in the Testing and/or Inspection of Materials Used in Construction (E 329)—Provides requirements for laboratories testing and inspecting construction materials; it references other ASTM standards for specific materials.
- ASTM Guide for Calibration and Testing Accreditation Systems—General Requirements for Operation and Recognition (E 994)—Provides requirements for systems that accredit organizations involved in testing, measuring, inspecting, and calibrating activities.
- ASTM Standard Terminology Relating to Conformity Assessment (E 1187)—Provides definitions of terms under the jurisdiction of ASTM Committee E36.
- ASTM Standard Guide for Proficiency Testing by Use of Interlaboratory Comparisons (E 1301)—Provides guidance for the development and operation of proficiency sample programs including management structure, specimen preparation and distribution, and the analysis and reporting of results.
- ASTM Standard Guide for Evaluating Laboratory Measurement Practices and the Statistical Analysis of the Resulting Data (E 1323)—Provides guidance for assessors to evaluate measurement practices of laboratories and ensures that appropriate protocols are provided for statistically analyzing data.
- ASTM Standard Guide for Surveillance of Accredited Laboratories (E 1580)—Presents procedures which a laboratory accreditation body may use to provide assurance that accredited laboratories continue to satisfy criteria and conditions under which they were accredited.
- ASTM Standard Guide for Development of a Directory of Accredited Laboratories by an Accrediting Body (E 1738)—Provides guidance on criteria to be used by laboratory accrediting bodies in the development of directories of accredited laboratories.
- ASTM Standard Guide for Selection, Assignment, and the Monitoring of Persons to be Utilized as Assessors/Auditing Technical Experts (E 2159)—Provides guidance to organizations that use independent auditors or assessors to assure the soundness of their quality systems and practices.

The key to competent testing is the laboratory's use of an effective and comprehensive quality system involving both quality assurance and quality control activities. ASTM E 1187 defines quality assurance as "all the planned and systematic activities implemented with the quality system, and demonstrated as needed, to provide adequate confidence that an entity will fulfill requirements for quality"; and quality control as "operational techniques and activities that are used to fulfill requirements for quality." In recent years, there has been increasing emphasis, both nationally and internationally, on the need for laboratories to establish and maintain quality systems [6]. ASTM E 1187 defines a quality system as "the organizational structure, procedures, processes and resources needed to implement quality management." A quality system must be tailored to the unique characteristics and capabilities of each laboratory. The International Organization for Standardization/International Electrotechnical Commission (ISO/IEC) 17025 General Requirements for the Competence of Testing and Calibration Laboratories [7], and the AASHTO R18 Standard Recommended Practice for Establishing a Quality System for Construction Materials Testing Laboratories [8] provide information similar to that contained in ASTM E 1187.

Each of these standards concerning quality systems require that the policies, procedures, and practices comprising a laboratory's quality system be documented in a quality manual. ASTM E 1187 defines a quality manual as "a document stating the quality policy, quality system, and quality practices of an organization." Such a manual provides the staff with an understanding of the laboratory's quality policies and operating procedures and the extent of their duties and responsibilities. Assessment of a laboratory by an evaluating authority is based on the existence of a comprehensive quality manual and documentation confirming that the laboratory operates as stated in the manual.

A quality manual is essential since it provides the basic reference to a laboratory's quality system. Typical topics covered in the quality manual include: quality policy; description of the laboratory structure; relations between management, technical operations, support services, and the quality system; job descriptions of staff; identification of the laboratory's approved signatories; procedures for achieving traceability of measurements; scope of testing and references to test procedures; arrangements for ensuring that the laboratory reviews new work to ensure that it has appropriate facilities and resources; procedures for handling test items; reference to major equipment and reference standards used; reference to procedures for calibration, verification, and maintenance of equipment; reference to interlaboratory comparisons; procedure for feedback and corrective action; procedures for dealing with complaints; procedures for protecting confidentiality and proprietary rights; and procedures for audit and review. The actual quality system requirements depend on the standard that a laboratory is trying to meet.

Continuing Improvements in the Quality of Concrete Testing

Among other things, ASTM Committee C09 on Concrete and Concrete Aggregates, and ASTM Committee C01 on Cement prepare documents for use by inspection/accreditation agencies or other parties on evaluating concrete and cement testing laboratories. ASTM C 1077 prepared by Committee C09 provides criteria for the evaluation of the capability of testing laboratories to perform designated ASTM tests on concrete and concrete aggregates [3]. The standard establishes minimum requirements for the testing laboratory's personnel, equipment, and quality system. The 2003 version of C 1077 lists seven mandatory ASTM test methods in which competence is required for laboratories testing concrete (C 172, C 143/C 143M, C 138/C 138M, C 173/C 173M, C 1064/C 1064M, C 31/C 31M, and C 39/C 39M), and five mandatory ASTM standards for laboratories testing concrete aggregates (C 136, C 117, C 127, C 128, and C 40). Further, it lists optional ASTM concrete and aggregate test methods for which a laboratory may request evaluation. A laboratory complying with ASTM C 1077 must establish and maintain a quality system that includes procedures for personnel evaluation and training, participation in a proficiency sample program, procedures for record keeping, procedures for equipment calibration and maintenance, an inventory of test equipment, procedures for handling technical complaints, and procedures for assuring the quality of external technical services. ASTM C 1077 also requires that testing services offered by the laboratory be under the full-time technical direction of a professional engineer with at least five years experience in construction materials testing, and that the laboratory be periodically assessed by an independent evaluating authority.

Turning to cement, ASTM Standard Practice for Evaluation of Laboratories Testing Hydraulic Cement (C 1222), which was first published by Committee C01 in 1993, identifies minimum training and experience requirements for personnel, and equipment requirements for cement testing laboratories [9]. ASTM C 1222 does not identify mandatory test methods that a laboratory must be able to perform, but does require that it have the capability of performing all laboratory testing associated with its intended functions. Standard chemical and physical requirements for various types of cements are listed in the ASTM Specification for Masonry Cement (C 91), ASTM Standard Specification for Portland Cement (C 150), ASTM Standard Specification for Blended Hydraulic Cements (C 595), ASTM Standard Specification for Expansive Hydraulic Cement (C 845), and ASTM Standard Performance Specification for Hydraulic Cement (C 1157). The scope of a testing laboratory may be chemical testing, physical testing, or both. A laboratory complying with ASTM C 1222 is required to establish and maintain a quality system for cement analogous to that for concrete, as described in ASTM C 1077. The manager of the laboratory should be a chemist, materials analyst, or an engineer with at least three years of supervisory experience in the testing of hydraulic cement; or a person with equivalent science-oriented education or experience. A periodic assessment by an evaluation authority is also required.

Evaluation Authorities

ASTM C 1077 defines an evaluation authority as "an independent entity, apart from the organization being evaluated, that can provide an unbiased evaluation of that organization." The standard lists the Cement and Concrete Reference Laboratory (CCRL) sponsored by ASTM Committees C01 and C09 as an evaluation authority that provides laboratory inspection and proficiency sample services. Accrediting bodies listed include the "National Voluntary Laboratory Accreditation Program (NVLAP), the American Association for Laboratory Accreditation (A2LA), Construction Materials Engineering Council (CMEC), AASHTO Accreditation Program (AAP), and other recognized agencies as may be established."

CCRL and the AASHTO Materials Reference Laboratory (AMRL) comprise the Construction Materials Reference Laboratories (CMRL) [10,11] which are located at the National Institute of Standards and Technology (NIST). CCRL and AMRL are NIST Research Associate Programs that operate at NIST under Memoranda of Agreement between the sponsoring organizations and NIST. ASTM is the sponsor of CCRL and AASHTO is the sponsor of AMRL. ASTM provides programmatic and technical oversight of CCRL through a Joint C01/C09 Subcommittee on the CCRL, while AASHTO provides oversight to AMRL through the AASHTO Subcommittee on Materials.

CMRL promotes the quality of testing by assessing the performance of construction materials testing laboratories; providing support to the sponsoring standards committees in the preparation of test methods; operating a research program which complements and benefits from interaction with the NIST Building and Fire Research Laboratory (BFRL) program; and through use of its programs by accrediting bodies, governmental agencies, and other organizations involved in quality assessment. The primary functions of the CMRL are the assessment of testing laboratories and the distribution of proficiency test samples. Laboratory Assessors from the CMRL visit laboratories to evaluate equipment, procedures, and quality systems according to the requirements of the test methods, and provide a report of findings to the laboratory. Concrete materials included in the CCRL Laboratory Inspection Program are hydraulic cements, concrete, reinforcing steel, aggregates, masonry materials, and pozzolans. Concrete materials routinely distributed to laboratories by the CCRL Proficiency Sample Program include portland cement, blended cement, masonry cement, portland-cement concrete, and pozzolan materials; while the AMRL Proficiency Sample Program distributes fine and coarse aggregate proficiency samples as well as samples of other highway materials. Data from these programs are provided to standards committees of ASTM and AASHTO for assessing the adequacy of test methods, determining the impact of revisions to standards, and for use in preparing precision statements. Over 1500 different laboratories in the United States and 25 other countries currently participate in the CMRL Laboratory Assessment and Proficiency Sample Programs. Utilization of the CMRL programs is voluntary and laboratories are not rated, certified, or accredited by the CMRL itself, though because of its reputation for integrity, results from its programs are used by three of the four accrediting authorities referenced in ASTM C 1077.

The American Association for Laboratory Accreditation (A2LA) was formed in 1978 as a nonprofit scientific membership organization dedicated to the formal recognition of testing organizations that have been shown to be competent [12].

A2LA grants accreditation in the following fields of testing: acoustics and vibration, biology, calibration, chemistry, construction materials, environmental, geotechnical, electrical, mechanical, nondestructive testing, and thermal. The construction materials field of testing which includes cement, concrete, soils, asphalt, and aggregates currently has about 100 laboratories accredited. A2LA requires laboratories to participate in the applicable proficiency sample programs of CCRL and AMRL, and generally uses contract assessors who are experts in their field for on-site assessment of laboratories.

The AASHTO Accreditation Program (AAP) was started by AASHTO in 1988 for construction materials testing laboratories [11,13]. AAP certifies the competency of testing laboratories in carrying out specific tests on soils, asphalt cements, cutback asphalts, emulsified asphalts, hot-mixed asphalt, aggregates, hydraulic cement, and portland cement concrete. The Laboratory Inspection and Proficiency Sample Programs of CCRL and AMRL are used by AAP to evaluate the performance of laboratories that test these materials. As of June 2004, AASHTO had accredited 939 laboratories in the United States and Canada of which 599 were portland cement concrete laboratories and 68 were hydraulic cement laboratories. AAP accreditation may also be obtained for compliance with ASTM C 1077, ASTM C 1222, and ISO/IEC 17025.

The Construction Materials Engineering Council (CMEC) was founded in 1983 for the purpose of improving the quality of production, inspection, and testing of construction materials through accreditation, education, and certification programs [14]. CMEC operates laboratory accreditation and proficiency testing programs for cement, concrete, aggregates, soils, asphalt, masonry, and chemical testing, and is a sponsoring group for the American Concrete Institute (ACI) Technician Certification Program. CMEC provides inspection and proficiency sample programs to over 400 different laboratories annually, throughout 15 states (from coast to coast) and three foreign countries. Because CMEC provides its own proficiency samples, CMEC does not use CMRL programs in accrediting laboratories.

The National Voluntary Laboratory Accreditation Program (NVLAP), which is administered by NIST, accredits public and private testing laboratories based on the evaluation of their quality systems, equipment, test procedures, and the technical qualifications and competence of their staffs for conducting specific tests [15]. NVLAP accreditation is based on the requirements of ISO/IEC 17025 and the relevant requirements of ISO 9002. NVLAP accreditation in the construction materials testing field is available for selected methods of testing for concrete, aggregates, cement, admixtures, geotextiles, soil and rock, bituminous materials, and steel materials. Participation in the CCRL and AMRL proficiency sample programs may be used by laboratories to meet the NVLAP proficiency testing requirements for aggregates, cement, concrete, soil, and bituminous material. NVLAP generally uses contract assessors who are experts in their field for on-site assessments of the laboratories. There are currently around 20 laboratories accredited by NVLAP in the construction materials testing field.

Other organizations which accredit construction materials testing laboratories include the International Accreditation Service [16], the Concrete Advisory Board of Georgia [17], and the Washington Area Council of Engineering Laboratories [18].

Technician Competency

The competency of laboratory technicians conducting the tests plays a vital role in assuring the quality of testing. Laboratories have a responsibility to have a means for training their personnel, assessing their competency, and documenting their qualifications to perform assigned tasks. As a means of ensuring this competency, technician training and certification programs are offered by independent organizations including the American Concrete Institute (ACI) and the National Institute for Certification in Engineering Technologies (NICET). ASTM C 1077 references compliance with programs of these organizations, or of an equivalent certification program, as a means of demonstrating the competency of a technician in performing tests on concrete materials.

ACI currently operates eight certification programs for portland cement concrete and aggregates: Field Testing Certification Program, Craftsman Certification Program, Strength Technician Certification Program, Laboratory Technician Certification Program, Aggregate Technician Certification Program, Inspector Certification Program, Tilt-Up Certification Program, and Shotcrete Nozzleman Program [19]. NICET offers four levels of certification for concrete technicians [20].

Conclusions

High-quality field inspection and laboratory testing services are important to achieving safe, efficient, and cost-effective concrete structures. The mechanisms discussed in this chapter which are being developed, standardized, and implemented will continue to promote the quality of these services. The importance of concrete sampling and accurate testing will increase as structural design and modeling by computers require that material properties be determined more precisely. Additionally, the development of high-performance concretes with enhanced mechanical, volume stability, durability, and placement properties require more accurate test results.

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PART II
Freshly Mixed Concrete

8

Factors Influencing Concrete Workability

D. Gene Daniel[1]

Preface

I. L. TYLER AND FRED HUBBARD PREPARED CHAPters on “Uniformity, Segregation and Bleeding” and “Workability and Plasticity,” in *ASTM STP 169* [1,2]. C. A. Vollick combined, revised, and updated these two chapters into “Uniformity and Workability” for *ASTM STP 169A* [3]. D. T. Smith revised and updated this chapter in *ASTM STP 169B* [4]. John M. Scanlon revised, updated, and changed the title to “Factors Influencing Concrete Workability” in *ASTM STP 169C* [5]. The current edition reviews, revises, and updates the topics as addressed by the previous authors. Mr. Tyler was with the Portland Cement Association, Mr. Hubbard with the National Slag Association, Mr. Vollick with Sika Chemical Corporation, Mr. Smith with Marquette Cement Manufacturing, and Mr. Scanlon was a senior consultant with Wiss, Janney, Elstner Associates, Inc.

Introduction

The workability of concrete is partially determined in the eyes of the beholder, who is influenced by the space to be filled with the concrete and the equipment available to assist in the placement and finishing process. Each of the parties having a responsibility for the completed concrete structure views workability somewhat differently depending upon his or her particular responsibilities. The involved parties may include the owner, engineer, contractor, concrete materials suppliers, concrete producer, testing agency, inspectors, and others. The concrete construction portion of a project cannot be successful without reasonable guidelines pertaining to the workability of the concrete for each portion of the work. There are a number of factors that influence the workability of concrete. However, there is no widely accepted test method to measure the workability of concrete.

It is important to note that by using today’s technology, concrete mixtures can be proportioned with practically any workability and retain the capabilities to develop a wide range of hardened properties needed by the structure. Such technology may require the use of specialty products such as nonlocal aggregates, supplementary cementitious materials, and admixtures.

Terminology

ASTM C 125 Terminology Relating to Concrete and Concrete Aggregates defines workability of concrete as “that property determining the effort required to manipulate a freshly mixed quantity of concrete with minimum loss of homogeneity.” The American Concrete Institute (ACI) defines workability in ACI 116R [6] as “that property of freshly mixed concrete or mortar that determines the ease with which it can be mixed, placed, consolidated, and finished to a homogeneous condition.” Workability includes such items as compactibility, consistency, finishability, harshness, and pumpability. Project specifications will normally provide guidance on maximum workability as associated with water content of the concrete via maximum water-cement ratio, range of slump as measured in accordance with ASTM Test Method for Slump of Hydraulic-Cement Concrete (C 143/C 143M), and at times a minimum cement content. These specified properties of the fresh concrete may not fully describe the degree of workability desired by the contractor in the fulfillment of the contractor’s responsibilities. Concrete mixture proportioning to provide greater workability then becomes an issue between the contractor and the concrete producer to determine a method to provide a greater workability and maintain concrete that in the eyes of the design engineer meets the specified criteria. Four primary terms have now become important in describing some basic concrete properties. They are workability, which was previously defined, rheology, consistency, and self-consolidating concrete (SCC). Rheology is defined as “the science dealing with the deformation and flow of matter”and “the ability to flow or be deformed.” As related to concrete there is a direct relationship between shear stress and the rate of shear plus a shear stress when the rate of shear stress is zero. Another approach is to characterize the rheological properties of a concrete mixture by the yield stress and plastic viscosity. These properties are currently measured by rather large laboratory rheometers of several designs. The rheology of concrete, like slump, is time dependent as measured from the time of initial mixing to the time of testing. ACI 116R [6] defines rheology as “the science dealing with flow of materials, including studies of deformation of hardened concrete, the handling and placing of freshly mixed concrete, and the behavior of slurries, pastes, and the like.”

The importance of rheology technology and measurements is that it advances the industry past a single descriptive measurement for concrete workability. Information on behavior of fresh concrete during vibration and rheology on consolidation can be found in ACI 309.1R [7]. Unfortunately a reliable field test has not been developed to measure the rheological properties. What has been shown is a reasonably definite relationship between slump and a rheometer-determined yield stress [8,9]. Both a field test and a greater understanding of the

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information provided by rheology test results and the relationship to the concrete composition [8] are current research goals.

Consistency is a term related to the freshly mixed concrete. ASTM and ACI definitions of consistency are virtually identical. ACI 116R [6] defines it as "the relative mobility or ability of freshly mixed concrete or mortar to flow; the usual measurements are slump for concrete, flow for mortar or grout, and penetration for neat cement paste." The word consistency has multiple usages and two of these are pertinent to concrete. The flow of the mixture, as measured by the slump test, and the variability from batch to batch are each important properties for successful projects. Once an acceptable level of workability has been established, the concrete quality relies on control of variability by maintaining uniform consistency.

Uniformity of Concrete

Concrete mixture proportions should always be developed in such a way that the finished hardened concrete will attain the required physical properties and be able to withstand exposure to the anticipated environmental conditions. Equally important is that the freshly mixed concrete should possess the workability and other characteristics that permit it to be mixed, transported, placed, consolidated, and finished in a timely fashion without hardship under prevailing conditions. After mixtures have been developed and the necessary characteristics that affect workability are determined, it is essential that the quantities of ingredients be kept relatively uniform.

Nonuniformity may be evident in freshly mixed and hardened concrete. There are two distinct measurements of nonuniformity: within-batch variations and batch-to-batch variations. Within-batch variations refer to variations relating to concrete sampled from the front, middle, and end of the batch as discharged from the mixer. These variations may be attributed to a buildup of hardened concrete in the drum, excessive wear of the mixer blades, inadequate or excessive mixing time and speed, improper loading sequence, or possibly overloading of the mixer.

Batch-to-batch variations in concrete may be attributed to variations in aggregate moisture conditions, aggregate gradings, inaccurate weighing or volumetric dispensing equipment, and, of course, all of the variations related to within-batch variations. The concrete mixture proportions may result in segregation of the mixture due to a relatively high water-cement ratio or poor mixture proportions. The use of improper material-handling equipment may also cause a marginal mixture to separate. The uniformity of concrete production and delivery should always be evaluated, because the better the uniformity during production, transportation, and placing, the greater the opportunity to obtain desired hardened concrete properties.

Control of Concrete Production

Uniform concrete can be obtained only through proper quality control of all operations from selection and production of materials through batching, mixing, transporting, conveying, placing, consolidation, finishing, and curing. All materials must be kept relatively uniform. There is great reliance on the manufacturers or producers of the raw materials for relatively uniform products. Hydraulic cements and supplementary cementitious materials, such as fly ashes, GGBF slag, silica fume, and natural pozzolans are each specified to be in compliance with an appropriate ASTM specification. To cover the wide range of raw materials being dealt with these specifications are predominately composed of minimum or maximum requirements and rarely have an allowable tolerance range for a prescribed property. The uniformity of product achieved is actually the result of the desire for customer satisfaction rather than any specified tolerances. Chemical admixture specifications contain minimum and maximum limits for properties compared to control mixtures that do not contain the particular admixture being tested. Chemical admixture specifications also contain maximum variances between lots and thus are designed to maintain consistency of the product. The tests involved typically involve residue content, infrared analysis, and specific gravity. Control must be maintained at the raw material sources and at the batching and mixing plants and related components.

Aggregates should be tested for grading, density, absorption, and moisture content, and mixtures should be adjusted to correct for changes in these properties. ASTM Specification for Concrete Aggregates (C 33) covers the requirements for both coarse and fine normal weight aggregates. Tests for aggregate properties are all defined as maximum allowables except for prescribed grading ranges for each size aggregate. Only the fine aggregate with a specified maximum variation of the fineness modulus of 0.20 from the base fineness modulus addresses consistency of the product. Consistency of aggregate products is driven by the need for customer satisfaction balanced against economic realities and not by specification requirements. As the demands for more consistent concrete increase the need for consistency requirements in aggregate properties will also increase. Items that may be subject to future variation restrictions include coarse aggregate gradings, moisture contents, absorption, relative density, and bulk density.

Control of aggregate moisture has steadily progressed with computerized batching and the development of moisture probes for aggregate feed bins, particularly for fine aggregates. Van Alstine [10] attributed the uniformity obtained at Denver Reservoir No. 22 Dam, where sand bins were filled 17–20 times each day with sand of widely varying moisture content, to the use of an electrical resistance meter. Even batch plants that do not use moisture determining probes have increased the monitoring of fine aggregate moisture by the use of rapid moisture content determinations. The available methods include using hot plates, ovens, or microwave ovens and ASTM Test Method for Total Evaporable Moisture Content of Aggregate by Drying (C 566), a Chapman flask with ASTM Test Method for Surface Moisture in Fine Aggregate (C 70), or a Speedy Moisture Tester as described in ASTM Test Method for Field Determination of Water (Moisture) Content of Soil by the Calcium Carbide Pressure Tester (D 4944). The chapter in this volume by Yzenas contains information on absorption and surface moisture.

Mixed concrete should be tested for consistency, air content, temperature, and density. Product tolerances are provided by ASTM Specification for Ready-Mixed Concrete (C 94/C 94M) for slump and air content. Slump tolerances are in two different formats: a "maximum" or "not to exceed" value or a specified target value. The tolerance varies by level of slump. The stated tolerance for air content is ±1.5 % of the specified value. Temperature and concrete density tests are useful production control tests, but do not have specific tolerances. Concrete specimens must be properly fabricated, cured, and tested for evaluating hardened concrete properties. The criteria for these specimens is provided in ASTM Practice for

Making and Curing Concrete Test Specimens in the Field (C 31/C 31M) and ASTM Test Method for Compressive Strength of Cylindrical Concrete Specimens (C 39/C 39M).

Routine instructions for measuring, mixing, and placing concrete are given by the American Concrete Institute (ACI) *Manual of Concrete Inspection* [11]. Additional information on practices that lead to better uniformity are found in the ACI 304R [12]. Some variation must be accepted, but consistent concrete of satisfactory quality can be obtained if proper control is maintained. ASTM C 94/C 94M provides specific instructions for critical segments of batching operations plus maximum acceptable batching tolerances for each material. The tolerances for measuring the materials vary with the material, the size of the batch, and the type of weigh batcher system for aggregates. For batches that use more than 30 % of scale capacity cement and cement plus other cementitious materials have a tolerance of ±1 %. Water added to the batch must be measured to an accuracy of 1 % of the total required mixing water. Total water includes any wash water and free water on aggregates and is measured to an accuracy of ±3 % of the specified total water quantity. Chemical admixtures are measured to a tolerance of ±3 %.

Concrete uniformity generally has been measured in terms of compressive strength, slump, density, air content, and content of coarse aggregate and cementitious materials. Uniformity tests have been used to establish required mixing time, mixing speed, mixer batch capacity, and to verify efficient batching procedures. Tests by a number of investigators have been considered in the preparation of ASTM C 94/C 94M.

ASTM C 94/C 94M also contains tolerances of test results that are requirements of uniformity of a single batch of concrete.

Methods of Measuring Uniformity

Tests for Mixer Uniformity

ASTM C 94/C 94M, Annex Al. Concrete Uniformity Requirements

Measuring the uniformity of concrete mixers has been a part of ASTM Specification for Ready-Mixed Concrete (C 94/C 94M) since the 1940s. The requirements of a mixer uniformity test have expanded and changed but the premise of checking for uniformity of concrete within a batch has remained the same. The test method requires two separate samples from each end of the middle portion of the mixer and involves determining the air content of each sample as well as the quantity of coarse aggregate, concrete density, density of air-free mortar, calculated density of air-free concrete, slump determinations, and seven-day compressive strength. The uniformity requirements involve six comparison values, assigning a maximum variation to each tested or calculated property. The ASTM C 94/C 94M requirement is that an acceptable mixer must meet not less than five of the six measured properties.

U.S. Army Corp of Engineers Test Method for Within-Batch Uniformity of Freshly Mixed Concrete (CRD-C 55-92) [13]

This method of evaluation tests three samples of concrete for water content, density of air-free mortar, coarse aggregate content, air content, slump, and seven-day compressive strength. The difference between this test method and the ASTM C 94/C 94M Annex Al mixer performance test is that three samples replace two samples and water content replaces concrete density as a sixth measurement and potential criterion. Tests are to be performed on samples of concrete representing each of the three thirds of the batch, but not the very first and very last portions of the batch to be discharged. The tests are performed also to determine the feasibility of altering the mixing time. The standard guide specifications that are used to prepare project specifications include limits for these various tests.

U.S. Bureau of Reclamation Test of Mixer Performance

The U.S. Bureau of Reclamation's mixer performance test [14] is used to evaluate the ability of a mixer to mix concrete that will be within prescribed limits of uniformity. The uniformity of freshly mixed concrete is evaluated by comparing variations in quantity of coarse aggregate, air content, and the density of air-free mortar of two samples, one taken from each of the first and last portions of the batch.

Large variations in the density of air-free mortar may indicate that the batching procedure is incorrect or mixer blades are worn. Additional mixing time may be required if the unit weight of air-free mortar varies more than 24 kg/m^3 (1.5 lb/ft^3) [15].

Dunagan Test

Dunagan [16] proposed a method for measuring the proportions of cement, water, sand, and coarse aggregate in fresh concrete by a series of wash separations and weighing in air and water. This method has been used by Slater [17], Hollister [18], Cook [19], and others to study the effects of different rates of rotation of truck mixers, effect of time of haul, and effect of mixing time on uniformity of concrete. The Dunagan test has limited usefulness because of sampling errors and difficulties in distinguishing between cement and very fine sand.

Air-Free Unit Weight Test

A study designed to establish test methods and limits for variations in truck-mixed concrete was reported by Bloem et al. [15]. Variations in slump, air content, percent of coarse aggregate, air-free density of mortar, water content by oven drying, and compressive strength of concrete obtained after approximately 1/6, 1/2, and 5/6 of discharge from a truck mixer were determined. They concluded that the air-free density test was an improvement over the density test because the number of variables was reduced and excessive changes in this property reflected changes in water or in proportions of cement and sand. According to their data, a difference in air-free density of mortar of 17.6 kg/m^3 (1.1 lb/ft^3) corresponds to a change in water of about 9.9 L/m^3 (2 gal/yd^3) when the proportions of sand to cement were kept constant and the water alone was varied. They suggested that a variation of more than 16.0 kg/m^3 (1 lb/ft^3) in this test indicates real differences in the proportions of the mortar ingredients, and differences of more than 32.0 kg/m^3 (2 lb/ft^3) should be considered evidence of unsatisfactory uniformity.

Tests for Quality Control Uniformity

Slump

The slump test, ASTM Test Method for Slump of Hydraulic-Cement Concrete (C 143/C 143M), is essentially an indication of the consistency of an individual batch of concrete. Large within-batch variations in slump indicate incomplete mixing and nonuniform distribution of water or other ingredients throughout the batch. Batch-to-batch variations may result from batching tolerances or errors, uncorrected changes in moisture content, grading of the aggregate, or variations in temperature. In reasonably uniform concrete, the slump measurement should not vary more than about 2.5 cm (1 in.) within a batch.

Air Content

Air content has an important influence on concrete workability. Air entrainment increases slump with each 1 % of additional air being approximately equivalent to 2.5 cm (1 in.) of slump. Less air than desired will detrimentally affect workability. Sudden loss of workability may indicate a major change in air content. A sudden stiffening of the mixture may indicate loss of air or a lower air content than desired while a sticky mixture or reduced bleeding is an indicator of increased air content. It is important that the concrete contain a uniform quantity of air. Within-batch variations should not exceed 1 %.

Several methods have been developed to directly determine the air content of fresh concrete. The principally accepted methods include the pressure method, ASTM Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231), and the volumetric method, ASTM Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173/C 173M).

The pressure meter method, ASTM C 231, consists of a special pressure-tight container and accessories designed to hold a precalibrated volume of concrete. This method is used more than any other and is considered satisfactory for all types of concrete and mortar except those made with highly porous lightweight aggregate. This apparatus must be calibrated periodically to guard against changes caused by rough usage, and, if the elevation of the place at which the apparatus is used changes by more than 183 m (600 ft), it should be recalibrated. An aggregate correction factor should be determined with the materials used and subtracted from the apparent reading to determine the actual air content. The aggregate correction factor varies only slightly for the same type of aggregate and need only be checked when there is a definite change in materials.

Due to the advent of new air-entraining admixtures and their ability to entrain much smaller air bubbles, it is recommended that the results of the pressure meter tests be periodically verified by the density test.

The volumetric method, ASTM C 173/C 173M, consists of removing air from a concrete sample by mixing it with water and isopropyl alcohol in a long-necked, closed-top special container. The volume of air is determined from the difference in volume of the sample containing entrained air and the volume of the sample after it has been agitated to permit the air to escape. This method is recommended particularly for lightweight concrete, but it may be applied to other types of concrete as well.

A non-ASTM standard air indicator is a miniature device called the Chace Indicator, which uses the volumetric principle. This test method is described in detail in AASHTO T 199 Air Content of Freshly Mixed Concrete by the Chace Indicator [20]. A small sample of carefully selected mortar is obtained from the concrete and placed in a brass cup measuring 1.9 cm (3/4 in.) in diameter by 1.3 cm (1/2 in.) high and compacted with a wire or knife blade. The glass tube that comes with the device is filled to the top line with isopropyl alcohol, the brass tube is inserted in the tube, and the liquid level is adjusted to the top line. The finger is placed over the stem to prevent alcohol from escaping, and the indicator is rolled from vertical to horizontal several times until all mortar has been removed from the cup. With the indicator in a vertical position, the finger is carefully removed from the stem, and the number of graduations from the top to the new liquid level gives an indication of the air content in the mortar sample. A correction factor, based on the mixture proportion, must be applied to convert to percent air in concrete. Meticulous care must be used in the selection of the mortar sample, method of inserting the stopper, agitation of the sample, and removal of the finger from the tube. The test can only provide an indication of relative air content and cannot be considered as reliable as the pressure meter or the volumetric method and should not be used to accept or reject concrete. It is most useful as an indicator of the batch-to-batch consistency of the air content of the concrete.

Another non-ASTM standard air indicator is a mini-volumetric air meter developed by K. Nasser in Canada [21,22]. This instrument is approximately 50 % of the height of an ASTM C 173/C 173M volumetric meter. Aggregates larger than 25 mm are removed by sieving before the test procedure commences. The concrete is placed in an inner bowl using two layers of material that are each rodded 15 times with a 9.4-mm diameter rod before tapping the sides to remove large air bubbles and striking off the top to produce a level surface. Water is placed in an outer bowl before the concrete-containing inner bowl is inserted. This water covers the concrete upon insertion of the inner bowl. The long-necked top section is then connected to the outer bowl and more water is added through the open neck of the top section until the apparatus is completely full. The apparatus is inverted and shaken vigorously a minimum of 15 times. After shaking, the apparatus is set upright and jarred to release trapped foam. The top is opened and a measured quantity of antifoaming agent that may be an isopropyl alcohol is added. A calibrated rod is inserted through the top of the neck and the air content is read to the nearest 0.25 %.

This test method can be used to measure air contents of concrete containing any type of aggregate including lightweight. The potential problem with either of these non-ASTM test methods is greater variability of test results than with approved test methods.

The chapter in this publication by Roberts contains additional information on air content, temperature, density and yield.

Density (Unit Weight)

Air content of normal weight concrete may be computed by comparing the actual density of concrete with the theoretical mass based on the density of the materials used, as outlined in ASTM Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete (C 138/C 138M). Results obtained by this method are influenced by variations in mixture proportions, density of ingredients, and changes in moisture content of aggregates. Consequently, variations in concrete density are difficult to evaluate as to cause or significance. The test is more definitive when the mass and solid volume of coarse aggregate and volume of air are eliminated as in the density of air-free mortar test.

The density test is recommended as a job control measure, especially for lightweight aggregate concrete in conjunction with air determinations and slump. If the slump and air content are kept constant, a change in density indicates a change in mass of aggregate. If the mass of aggregate per cubic meter of lightweight concrete changes, it may be the result of a change in moisture content, grading, or density of the aggregate. Additional tests, including density, moisture content, and grading of the aggregate are required in order to determine the cause of the variation.

The chapter in this publication by Roberts contains added information on air content uniformity as related to density.

Temperature

Workability is also effected by the temperature. As the temperature increases the time period for a given workability decreases. The heat increases both the hydration rate and the rate of water evaporation. Measurement of the freshly mixed concrete temperature is accomplished using a calibrated temperature measuring devise accurate to ±1°F (±0.5°C). The measurement and reading are performed in accordance with ASTM Test Method for Temperature of Freshly Mixed Hydraulic Cement Concrete (C 1064/C 1064M). The primary precaution in determining the temperature of fresh concrete is to properly and adequately cover the temperature sensor with concrete.

Flow Test

The measurement of concrete flow by ASTM Test Method for Flow of Freshly Mixed Hydraulic Cement Concrete (C 1362) is a quick indicator of batch-to-batch consistency of the concrete or within-batch consistency. It is applicable for concrete with coarse aggregate up to 37.5 mm (1½ in.). The dimensions of this relatively small and compact apparatus are provided in ASTM C 1362. The flow testing device was originally known as a K-slump tester [23]. ASTM C 1362 contains a precautionary note that this test method may not be appropriate for use with gap-graded aggregate concrete.

Vebe Apparatus

A modified Vebe apparatus is now used to determine the consistency of very stiff concrete. The current test method, primarily used for roller-compacted concrete, is applicable for any low-slump concrete. The ASTM Test Methods for Determining Consistency and Density of Roller-Compacted Concrete Using a Vibrating Table (C 1170) were approved in May 1991 and are under the jurisdiction of Subcommittee C09.45 on Roller-Compacted Concrete. This is a laboratory test method, thus limiting its usefulness as a quality control method. This test method includes a vibrating table with a surcharge mass (Test Method A) or without a surcharge mass (Test Method B). ASTM C 1170 also prescribes a method to determine the density of the consolidated concrete specimen.

Strength Testing

Measured concrete strength is used widely as a criterion of concrete quality. Other factors such as durability, abrasion resistance, thermal properties, dimensional stability, placeability, and compactibility may be more critical, but strength tests are easily made and variations in strength are assumed to be indicative of variations in other properties.

Compressive strength as a control test is particularly useful in determining the degree of uniformity of concrete. ASTM Test Method for Compressive Strength of Cylindrical Concrete Specimens (C 39/C 39M) is the most-used strength test for this purpose. ACI Committee 214 [24] has developed ACI 214R-02, Evaluation of Compressive Test Results of Concrete as a statistical evaluation method for control of compressive strength based on the coefficient of variation. The larger the variation in strengths of production concrete the larger the compressive strength overdesign needed to ensure strength requirements are met. Not to be overlooked in an evaluation process are any improper laboratory procedures that may be a source of strength variations. ASTM C 94/C 94M lists requirements for within-batch uniformity of concrete, and these are expressed as the maximum permissible difference in results of samples taken from two locations in a concrete batch. The permissible difference for the average compressive strength at seven days for each sample (not less than three cylinders), based on the average strength of all comparative specimens, is 7.5 %. In a well-controlled laboratory, compressive strength of cylinders fabricated from the same concrete sample may vary from 3 to 5 %. Variations in excess of this amount must be attributed to mixing variations within the batch of concrete being tested for uniformity. An excessive variation for within batch compressive strengths may indicate a mixer containing fins with excessive wear or excessive hardened buildup.

Another ASTM strength test of concrete available for strength comparisons is ASTM Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens (C 496/C 496M). This test method also uses cylindrical specimens, but the load is applied to the edge of the cylinder producing a tensile failure. The variation in test results is greater than with the ASTM C 39/C 39M compressive strength test, thus limiting the practical usefulness of this test to projects using the splitting tensile strength as a pass/fail criterion for the concrete.

Flexural strength testing is normally limited to paving applications. The available test procedures are ASTM Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78) or ASTM Test Method for Flexural Strength of Concrete (Using Simple Beam With Center-Point Loading) (C 293). Flexural specimens are sensitive to small variations in molding, curing, and handling. The result is wider variations in test results than with testing for compressive strength. When strength is to be used as a measure ASTM C 39/C 39M is the test method of choice unless one of the other methods is required as a pass/fail test for strength. The chapter in this volume by Ozyildirim and Carino on concrete strength testing contains information on quality control uniformity.

Tests for Cement Content Uniformity

Centrifuge Test

This test, also known as the Willis-Hime method, is described in detail elsewhere [25]. It provides a basis for within-batch comparisons of cement content of concrete and employs a liquid with a density greater than sand but less than cement. This liquid is used to separate the components of a carefully prepared mortar sample extracted from the concrete.

The centrifuge test was used in the study by Bloem et al. [15]. They concluded that the test is quite involved and, in most cases, the information gained is not commensurate with the time and labor required. This test for cement content was an alternative to the air-free mortar density in mixer uniformity testing during the 1960s but was then dropped from ASTM C 94/C 94M.

Rapid Analysis for Determining the Cement Content of Freshly Mixed Concrete

This test method was ASTM C 1078 and was discontinued in August 1998. This test method is discussed in detail in Chapter 13, "Cement and Water Content of Fresh Concrete," in *ASTM STP 169C* [5].

Cement Content Determined by Nuclear Methods

An instrument small enough to be used in a field laboratory uses nuclear technology to measure the amount of cement in a sample of freshly mixed concrete [26].

The test apparatus contains radioisotopic materials. Cement, primarily composed of calcium, is measured by the absorption of low-energy photons emitted by the apparatus. The calcium atoms absorb the photons in proportion to the quantity of calcium present in the concrete. Calcareous aggregates and Class C fly ash can create potential measurement errors. Several calibration tests are necessary with each concrete mixture and with each change in source of materials. A calibration curve must be developed for each mixture to be tested. The sensitivity of the apparatus to the chemical make-up of other materials has produced variations in reported cement contents in excess of permitted batching tolerances. The use of nuclear methods may be satisfactory for project averages but the accuracy is not sufficient for measuring batch-to-batch cement quantities. As recycling of mixer wash water and process water increases this water becomes another source of non-cement calcium that varies and must be accounted for in the measurements by this apparatus.

Cement Content of Fresh Concrete

Constant neutralization by 3 N hydrochloric acid (HCl) for a fixed period of time (1 h in this procedure) has been employed by the California Department of Transportation [27]. Accuracy of this procedure was reported to be within about 14 kg of cement/m^3 (24 lb/yd^3). Field tests indicate the procedure is most useful for evaluating the performance of a concrete mixer. Relative cement contents of various portions of a batch can be determined in about 1 h.

This test procedure was apparently used on concrete that did not contain calcareous aggregates; at least, there was no discussion of the reaction of HCl on the aggregates, and this is a phenomenon that should have a profound effect on the end results of the test. The test procedure does require a new calibration curve if there is any change in cement or aggregate source and ordinarily a new curve each day.

The authors of the report state in their conclusions that the test was not proven to be of sufficient accuracy for routine control of cement content during normal concrete production and, at present, the most applicable use of the procedure is in evaluating mixer efficiency.

Tests for Water Content Uniformity

Rapid Analysis for Determining Water Content of Freshly Mixed Concrete

This test method was ASTM C 1079 and was discontinued in August 1998. This test method is discussed in detail in Chapter 13, "Cement and Water Content of Fresh Concrete," in *ASTM STP 169C* [5].

Water Content Determined by Microwave Oven Drying

A sample of the concrete is wrapped in a fiberglass cloth and dried in a microwave oven. The particulars are the use of not less than three drying cycles with crushing of lumps and separating coarse aggregate after the first cycle. After each subsequent drying cycle the test specimen is stirred and its mass determined. Each of the first three drying cycles is approximately 5 min and subsequent cycles are approximately 2 min. The 2-min cycles continue until the change in mass is less than 1 g. The water content is calculated by dividing the total change in mass by the mass of the fresh specimen.

The American Association of State Highway and Transportation Officials (AASHTO) has adopted this principal in Test Method T-318 [28]. ASTM has not adopted this procedure due to its poor precision. Potential modifications being studied to improve the test precision include the use of specimens larger than the current 1500 g minimum plus the use of more powerful ovens.

Water Content Determined by Nuclear Methods

An instrument small enough to be used in a field laboratory uses nuclear technology to measure the amount of water in a sample of freshly mixed concrete. The test apparatus contains radioisotopic materials. Water is actually measured as hydrogen because there is little hydrogen in the other materials making up the concrete. The apparatus contains a fast neutron source that emits these neutrons into a concrete sample by a probe extended into the sample. Upon striking hydrogen atoms the source neutrons lose energy. The apparatus measures the quantity of these neutrons, converting the results into pounds of water [26,29]. Several calibration batches are required for each different concrete mixture. The nuclear apparatus has not been incorporated in an ASTM test method due to precision variations larger than the allowable batching tolerances of ASTM C 94/C 94M.

Workability

Workability is an everyday concern in concrete construction, and it is a factor easily appreciated in practice. Workability means different things to different people and for different placing conditions. Various nonstandard methods have been developed for its measurement. None of these tests evaluate all characteristics that are involved in this property.

Granville [30] defined workability as "that property of the concrete which determines the amount of useful internal work necessary to produce full compaction." Powers [31] defined it as "that property of the plastic concrete mixture which determines the ease with which it can be placed and the degree to which it resists segregation." Both relate to the physical characteristics of the concrete alone, being independent of the methods of placing and compacting.

In actual practice, workability is related directly to the type of construction and methods of placing, mixing, and transporting. Concrete that can be placed readily without segregation or separation in a mass dam could be entirely unworkable in a thin structural member. Workable concrete compacted by means of high-frequency vibrators would be unworkable if vibrators could not be used and hand tamping and spading were required. Concrete having suitable workability for a pavement might be unsuitable for use in a thin, heavily reinforced section.

Properties involved in workability include finishing characteristics, consistency or fluidity, pumpability, mobility, segregation, and bleeding. None of the test methods proposed or in use today simultaneously measures all of these properties. Consequently, measurement of workability is determined to a large extent by judgment, based on experience.

Workability is dependent upon the physical and chemical properties of the individual components and the proportions of each in the concrete. The degree of workability required for proper placement and consolidation of concrete is governed by the type of mixing equipment, size and type of placing equipment, method of consolidation, and type of concrete.

Factors Affecting Workability

Some of the factors that affect the workability [30] of concrete are quantity of cementitious materials, characteristics of these materials, consistency, grading of fine aggregate, shape of sand grains, grading and shape of coarse aggregate, proportion of fine to coarse aggregate, percentage of air entrained, type and quantity of pozzolan or other supplementary cementitious material, quantity of water, mixture and ambient temperatures, amount and characteristics of admixtures used, and time in transit.

Cement

Very lean mixes tend to produce harsh concrete having poor workability. Rich mixes are more workable than lean mixes, but concrete containing a very high proportion of cementitious materials may be sticky and difficult to finish. An increase in the fineness of cement increases the cohesiveness of the concrete mix as well as the rate at which the cement hydrates and the early strength development.

Consistency

Consistency (according to ASTM C 125) and plasticity are terms often used to indicate workability. Consistency generally denotes the wetness of the concrete, which is commonly measured by the slump test. It must not be assumed that the wetter the mix the more workable the concrete. If a mix is too wet, segregation may occur with resulting honeycomb or sand streaking on the exposed surface; finishing properties will be impaired because of the accumulation of laitance on the surface. If a mix is too dry, it may be difficult to place and compact, and separation may occur because of the tendency for larger particles to roll towards the outer edge of the heap formed when it is deposited. It is agreed generally that concrete should have the driest consistency that is practicable for placement with available consolidation equipment. The consistency necessary for full compaction varies with the type of structure, type and size of aggregate, and type of compaction equipment available.

Sand

Concrete containing fine sand requires more water for the same consistency, as measured by the slump test, than an equivalent amount of coarse sand. Very coarse sand can have an undesirable effect on finishing quality. Neither very fine nor very coarse sand is desirable but both have been used satisfactorily. Rounded river sand gives greater workability than crushed sand composed of sharply angular pieces with rough surfaces. Angular sand particles have an interlocking effect and less freedom of movement in the freshly mixed concrete than smooth rounded particles. Natural sand may give satisfactory results with a coarser grading than would be permitted with crushed manufactured sand. In addition, concrete must contain 2–3 % more sand by absolute volume of total aggregate and 6–9 kg more water/m^3 (10–15 lb/yd^3) when crushed sand is used.

Manufactured fine aggregate (MFA) processed from crushed stone is gaining wider use as natural sands become depleted in some geographic areas. The MFA generally contain a greater quantity of fines than natural sands and often mask their good workability with low slump test results. The aggregate particle shape and subsequent finishability characteristics are influenced by the type of crusher used in the MFA production process.

The chapters in this volume by Graves, Yzenas, Forster, and Meininger have additional information on factors affecting workability.

Coarse Aggregate

The particle size distribution of coarse aggregate influences the water requirements and workability of concrete. Coarse aggregates meeting standard grading requirements, such as ASTM Specification for Concrete Aggregates (C 33), should be used. After the grading is established, it should be maintained within rather close tolerances to avoid sudden changes in workability and other concrete properties. Segregation is reduced and uniformity improved by separating the aggregate into several size fractions and recombining these fractions when concrete is manufactured. This recombined grading should then be beneficial in establishing the fine aggregate (sand) content.

Breakage, separation, and contamination of aggregate can occur during handling and stockpiling. Introduction into the mixer of a large quantity of undersize material that may have accumulated will result in a sudden change in workability resulting in a demand for additional water. Plant layouts to minimize aggregate handling are desirable. Training of front-end loader operators on the use of techniques to minimize aggregate segregation will assist in avoidance of sudden changes in workability.

Production of workable concrete with sharp, angular, or crushed aggregates generally requires more sand than similar concrete made with rounded aggregates. The water demand may be increased 9–15 kg/m^3 (15–25 lb/yd^3). If the water-cement ratio is held constant, more cement is required. Flat or elongated particles, defined as particles having a ratio of width to thickness or length to width, respectively, greater than 3:1, are detrimental to concrete workability and finishability. More sand, cement, and water are required when the coarse aggregate contains flat and elongated particles.

The maximum size of aggregate that can be used to produce workable concrete is limited by practical considerations including type and size of structure, amount and spacing of reinforcing bars, method of placing, and availability of materials. Generally, aggregate should not be larger than three-fourths of the maximum clear spacing between reinforcing bars nor larger than one-fifth of the wall thickness or narrowest dimension between sides of forms. ACI Practice for Selecting Proportions for Normal, Heavyweight, and Mass Concrete (ACI 211.1) [32] provides recommendations on the maximum sizes of aggregate for various types of construction. The U.S. Bureau of Reclamation's [33] experience in pumping concrete indicates that concrete containing 6.4 cm (2 ½ in.) maximum size aggregate can readily be pumped through a 20.3 cm (8 in.) pipe, but aggregate larger than 6.4 cm (2 ½ in.) may cause difficulty. ACI 304R [12] recommends limiting the maximum size of angular or crushed coarse aggregate to one-third of the smallest inside diameter of the pump or pumpline.

The chapters in this volume by Graves, Yzenas, Forster, and Meininger have additional information on factors affecting workability.

Air Entrainment

Entrained air increases the paste volume, acts as a lubricant, and improves the workability of concrete. It reduces bleeding and segregation during handling and placing of concrete and increases the cohesiveness or "fattiness" of the concrete.

Improvement in workability resulting from air entrainment is more pronounced in lean mixes that are harsh and unworkable because of poor aggregate grading or type of aggregate used. The chapter in this volume by Roberts has additional information on factors affecting workability.

Finely Divided Material

The addition of finely divided material, including inert or cementitious materials or pozzolans, generally improves the workability of the concrete. Improvement is more noticeable in lean mixes than in rich mixes. These materials have been used to improve the grading of sands deficient in fines. Cementitious and pozzolanic materials are usually substituted volumetrically for 10–70 % of the cement. Workability will be improved if these materials are added as a replacement for part of the sand, instead of substituted for part of the cement.

Chemical Admixtures

Water-reducing admixtures, when added to concrete, permit a reduction in mixing water with no loss in slump, or, if the water content is held constant, produce an increase in slump. Set-retarding admixtures reduce the early rate of hardening and permit concrete to be handled and vibrated for a longer period of time.

It has been reported that there is a decrease in the frequency of plugged pump lines when water-reducing retarders are used in the concrete. This is partially due to the increased slump and typically an increased sand content with the use of a water-reducing chemical admixture. The use of high-range-water-reducing (HRWR) admixtures have greatly increased the placeability capabilities for high-strength, low-water-cement ratio concretes. HRWR admixtures have been used with silica fume providing highly workable concrete mixtures. The self-consolidating concretes require maximum utilization of chemical admixtures. A late generation HRWR is necessary to produce good flowability and a viscosity-modifying admixture (VMA) is usually needed to increase the consistency (viscosity) and prevent segregation.

Mixture Proportions

Concrete workability can be controlled by proper proportioning of the constituent materials. As the proportion of mortar, including sand, cement, water, and air, is increased, the grading and angularity of the coarse aggregate become less important. There should be sufficient mortar to fill the voids in the coarse aggregate plus a sufficient amount to permit the concrete to be placed readily in forms and vibrated around reinforcement as necessary. An excess of mortar increases workability, but excess workability is inefficient. Too much mortar can result in a sticky mixture. It should not be more than is required for consolidation by available equipment. The quantity of mortar required to produce the desired workability with a given coarse aggregate can be determined more effectively by laboratory tests. ACI 211.1 provides a basis for estimating the proportions of coarse aggregate to be used in trial mixes.

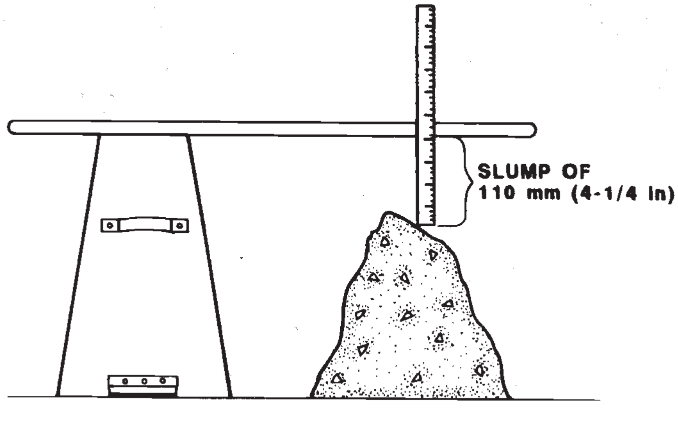


Fig. 1—Slump test.

Methods of Measuring Normal Consistency Concrete

Slump Test

The slump test (Fig. 1) is the most commonly used method of measuring the consistency of concrete. It is not suitable for very wet concrete, very dry concrete, or SCC. It does not measure all factors contributing to workability, nor is it always representative of the placeability of the concrete. However, it is used conveniently as a control test and gives an indication of the uniformity of concrete consistency from batch to batch. Repeated batches of the same mix, brought to the same slump, will have the same water content and water-cement ratio provided weights of aggregate, cement, and admixtures are uniform and aggregate gradings are within acceptable tolerances.

Additional information on the mobility of the concrete can be obtained if, after removing the slump cone and measuring the slump, the concrete is tapped on the side with the tamping rod. Two concretes with the same slump may behave differently, that is, one may fall apart after tapping and be harsh with a minimum of fines, and the other may be very cohesive with surplus workability. The first concrete may have sufficient workability for placement in pavements or mass concrete, but the other concrete may be required for more difficult placement conditions.

The slump test should be performed in strict accordance with the requirements of ASTM C 143/C 143M. Tests are often made at the point of placement and should be made whenever specimens are molded for strength testing. Slump tests may be made at the batch plant in order to check the uniformity of batching operations.

Popovics [34] has presented data indicating that the relationship between consistency values, as measured by the slump test and the water content of concrete, is parabolic, that is, the percentage change in water content required to increase the slump 25 mm (1 in.) may vary from 2.0 % when the initial slump is 127 mm (5 in.) to approximately 4.5 % when the initial slump is 51 mm (2 in.). An average change in water content of 3 % generally is considered necessary for a 25 mm (1 in.) slump change.

As the temperature of the concrete increases, the slump decreases. Concrete placed at a slump of 100 mm (4 in.) at 21°C (70°F) may only have a 76 mm (3 in.) slump when placed at 32°C (90°F), or the same concrete may have a slump of 140 mm (5.5 in.) when placed at 10°C (50°F).

Air-entrainment and water-reducing admixtures will increase the slump of concrete if all other conditions remain the same. Each 1 % increase or decrease in air content will produce approximately the same influence as a change in water content of 3 %.

The slump generally is reported to the nearest 5 mm (1/4 in.). Slump reported by different operators on the same batch of concrete may vary by as much as 13 mm (1/2 in.). The most unsatisfactory form of slump is the shear slump, that is, a falling away or shearing off of a portion of the concrete from the mass. If this condition exists, the concrete probably lacks the necessary plasticity for the slump test.

Slump cones manufactured using a stiff, durable plastic material have been approved by ASTM C 143/C 143M after a series of comparative tests at three different slump ranges indicated no measurable difference in test results. Both the classic metal cones and plastic cones can now be used for the slump test.

Flow Tester

The test apparatus is used to measure a concrete flow value with ASTM Test Method for Flow of Freshly Mixed Hydraulic-Cement Concrete (C 1362). The insertion of the instrument into the concrete is limited to 40 s before the measuring rod is lowered and the flow measurement read.

The flow tester (K-slump tester) (Fig. 2) is reported to measure slump directly 1 min after the tester is inserted in the concrete [23]. It measures an index that is related to workability after the device is removed from the concrete. The first reading is taken after the tester has been in place (the disk resting on the concrete surface) for 60 s. This reading, in centimetres, is referred to as the K-slump. The device is removed from the concrete and the measuring rod is again lowered to rest on the surface of the concrete remaining in the tube; this reading, in centimetres, represents the workability of the mix.

Coarse aggregate up to 37.5 mm (1 ½ in.) is permitted in the concrete. The minimum depth of concrete at testing is 175 mm (7 in.) and the minimum distance from the tube to the nearest edge of the level surface of concrete to be tested shall be 75 mm (3 in.). Concrete in the forms can be tested or concrete may be in a pail, wheelbarrow, or other container. The concrete may be retained in the original test position and retested later if desired. The test was developed to provide a method of testing using a minimal concrete area and volume. Studies have been made and reported on 420 concrete batches by five laboratories. Statistical determinations and equations are reported elsewhere [23].

Remolding Test

The remolding test apparatus was developed by Powers [31] to measure "the relative effort required to change a mass of concrete from one definite shape to another by means of jigging." The equipment consists of a metal cylinder mounted inside a larger cylinder and a suspended plate that fits inside the smaller cylinder. A slump cone is placed inside the smaller cylinder so that the bottom rests on the base. It is filled with concrete, the slump cone is removed, and the plate is placed on top of the concrete. The flow table on which the apparatus is mounted is then operated. The number of 6 mm (1/4 in.) drops required to mold the concrete into a cylindrical form is a measure of the workability of the concrete. This method has not found widespread use and no ASTM standard has been written about it.

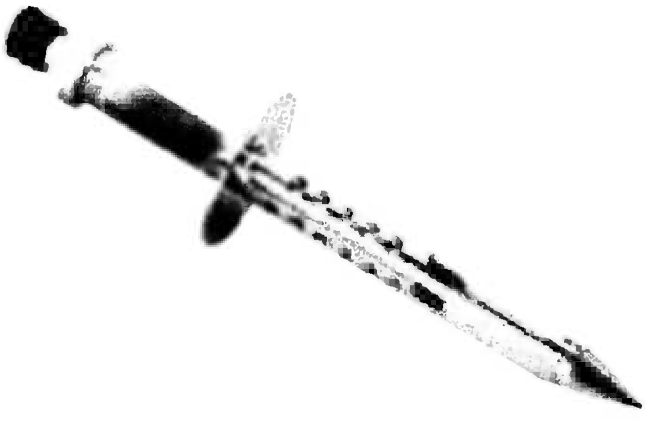


Fig. 2—Flow test apparatus.

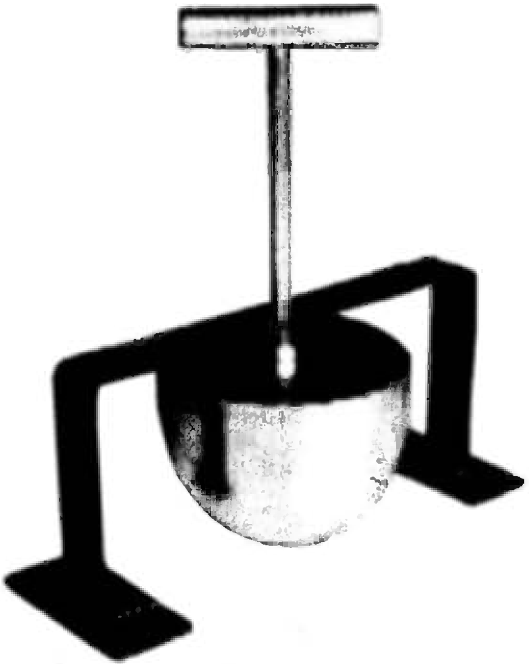


Fig. 3—Ball penetration apparatus.

Ball Penetration Test

The Kelly ball test [35] was developed principally as a convenient method of measuring and controlling consistency in the field. The ball test can be performed on concrete in the forms, space permitting, and it is claimed that tests can be performed faster and precision is greater than with the slump test. One disadvantage of this test is that it requires a large sample of concrete.

The apparatus (Fig. 3) weighs 13.6 kg (30 lb) and consists of a 15.2-cm (6-in.) diameter ball and stem that can slide through the center of a stirrup, the legs of which rest on the concrete to be tested. The depth of concrete must be at least 20 cm (8 in.), and the minimum distance from the center of the ball to the nearest edge of the concrete is 23 cm (9 in.).

The surface of the concrete is struck off level, avoiding excess working. The ball is lowered gradually onto the surface of the concrete, released, and the depth of penetration read immediately on the stem to the nearest 6 mm (1/4 in.). The ratio of slump to the penetration of the ball is between 1.5 and 2 and is fairly constant for a given mix but varies according to the mix. This ASTM Test Method for Ball Penetration in Fresh Portland Cement Concrete (C 360) was adopted in 1955 and discontinued by ASTM in February 1999. Information on this test method was reported in Ref 35 and *ASTM STP 169C* [5].

Truck Slump Meter

Truck mixers now use the hydraulic pressure needed to turn mixing drums to measure the consistency (slump) of the concrete contained in the drum. Tests indicate that higher pressures for mixing indicate lower slump values. After several calibration comparisons of hydraulic pressure readings versus measured slump test results, the truck mixer gage can be used to closely estimate the slump of the concrete. Load size is a factor, but equipment manufacturers claim it is not a major factor. These pressure readings will vary for each truck and mixer necessitating calibration for each mixing unit not of identical design, age, and condition.

Daczko [36] discusses the use of truck mixer slump meters as a method of measuring rheology properties of production concrete. Experimenting with two high-fluidity concrete mixtures having comparable slump values, slump meter pressure readings were taken at various drum speeds and graphically plotted as drum speed versus hydraulic pressure. The results produced the same pattern as that of a Bingham material and relative results comparable to the tests of these mixtures in an IBB rheometer [37]. The latter apparatus is not a true rheometer, but is the instrumented mixer described in the Two-Point Workability Tests.

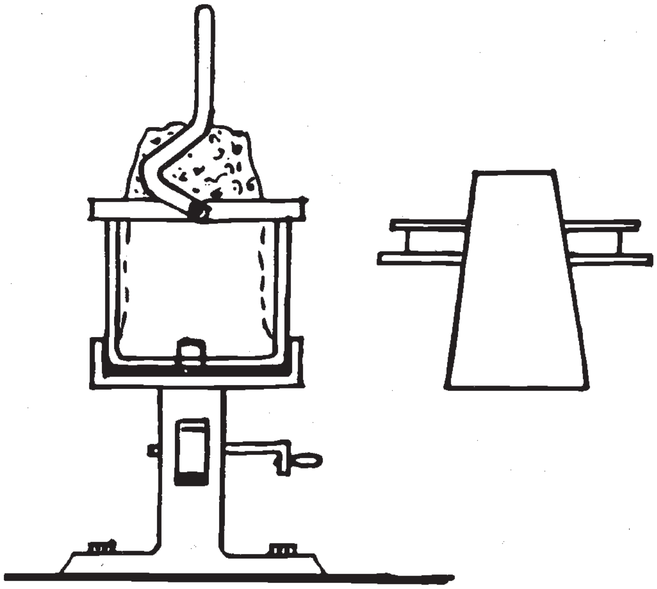


Fig. 5—Thaulow concrete tester.

Wigmore Consistometer

The Wigmore consistometer is described by Orchard [38]. This apparatus consists of a galvanized container and a hand-operated compaction table. A 5.1-cm (2-in.) diameter ball that is fastened to a sliding stem is mounted in the lid of the container. The container is filled with concrete that is compacted on the table by eight drops. The container is again filled with concrete and leveled off; then the lid and the ball are placed in position with the ball resting on the surface of the concrete. The apparatus is placed on the table and the concrete is compacted by turning the handle attached to the cam at the rate of about 1 rps. The table drops 0.6 cm (7/32 in.) four times per revolution of the cam, and the number of drops required to lower the ball and stem 19.7 cm (7¾ in.) into the concrete is considered a measure of the consistency of the concrete.

The number of drops required varies by a factor of ten as the concrete changes from a 150-mm (6 in.) slump to very stiff no-slump concrete. It is claimed that the Wigmore consistometer is an improvement over the slump test because work is actually done on the concrete in a way that resembles field conditions. Variations in results may be expected if the ball comes in contact with large aggregate.

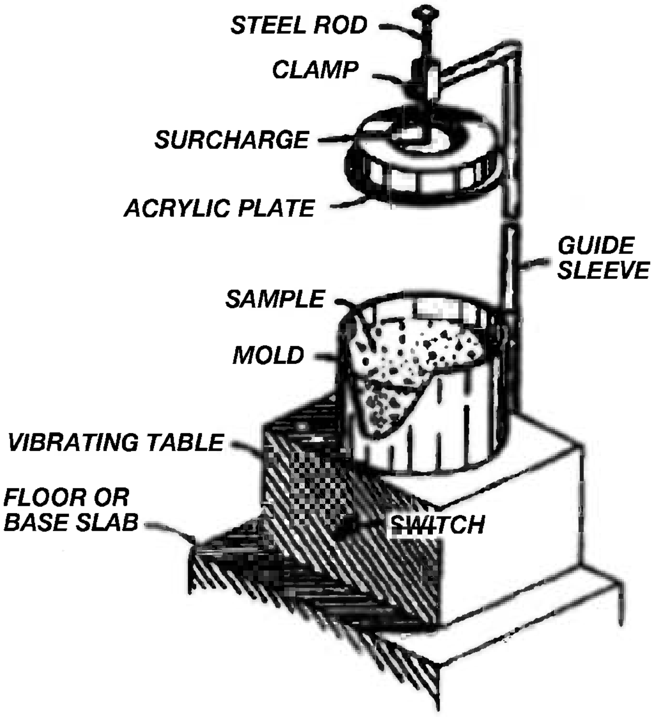


Fig. 4—Modified vebe apparatus.

Methods of Measuring Consistency of No-Slump Concrete

Vebe Apparatus

The modified Vebe apparatus (Fig. 4) is used to measure the consistency of Roller-Compacted Concrete. ASTM Test Methods for Determining Consistency and Density of Roller-Compacted Concrete Using a Vibrating Table (C 1170) is discussed in the chapter by Adaska in this volume.

The Vebe consistometer [39] includes a vibrating table, a sheet metal pan, slump cone, and plastic plate attached to a graduated, free-moving rod that serves as a reference end point. The cone is placed in the pan, filled with concrete, and removed. The plastic disk is brought into position on top of the concrete, and the vibrating table is set in motion. The number of seconds required to remold the cone of truncated concrete to the shape of the cylinder is the measure of consistency and is reported as the number of Vebe seconds or degrees. This method is very suitable for very dry concrete, but the vibration is too vigorous for concrete with a slump greater than about 5.1 cm (2 in.). For example, 0–3 s are required for concrete with a slump of 7.6–10.2 cm (3–4 in.), and 10–32 s [40] may be required for concrete with less than zero slump. The Vebe consistometer differs from the modified Vebe Apparatus of ASTM C 1170 and the results should not be interchanged. In the ACI Guide for Selecting Proportions for No-Slump Concrete (ACI

211.3R) [40], several tables are included referencing Vebe test results. These results are based upon the Vebe consistometer and not the modified Vebe apparatus of ASTM C 1170.

Other Methods

ACI 211.3R [40] provides a comparison of consistency measurements by three methods. These methods are the conventional Vebe consistometer [39], the Thaulow Concrete Tester developed in Norway [41] (Fig. 5), and the Compacting Factor test developed in Great Britain [30]. Differences in consistency of very dry mixes cannot be measured with the slump cone, but the Thaulow drop table is considered to have merit for this application. Concrete with a slump of 0–2.5 cm (0–1 in.) requires 14–28 revolutions of the drop table, and concrete with a slump of 7.6–10.2 cm (3–4 in.) requires less than seven revolutions. The Compacting Factor test is considered only marginal for very dry concrete.

Methods of Determining Consistency of Grout

Flow Cone

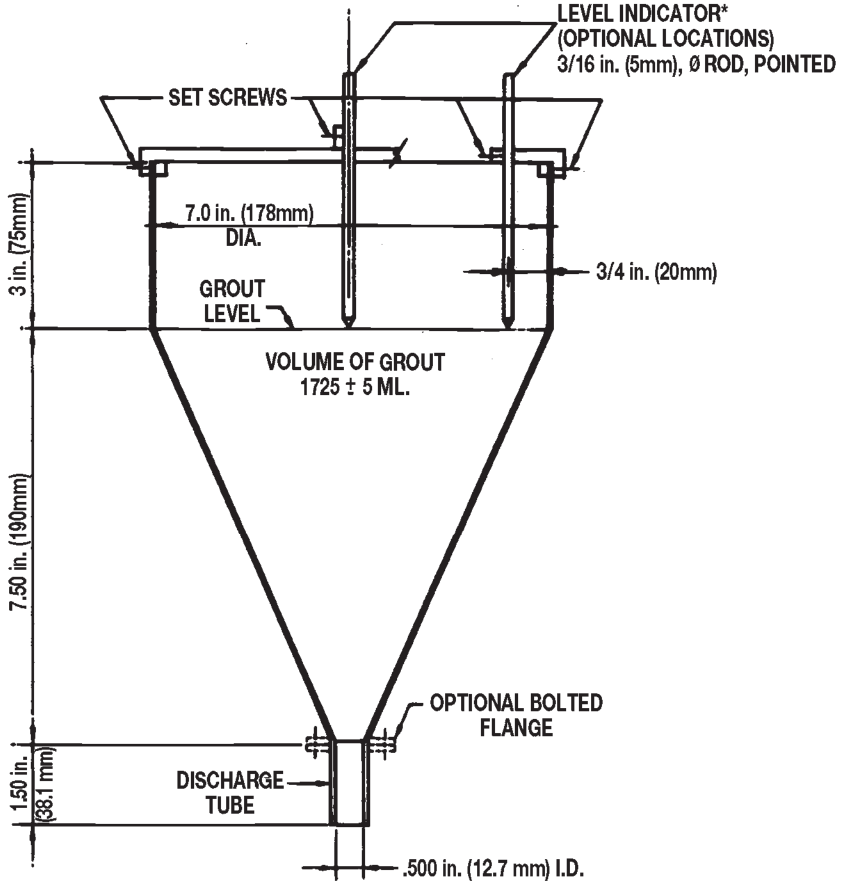
ASTM Test Method for Flow of Grout for Preplaced-Aggregate Concrete (Flow Cone Method) (C 939) is a method used in the laboratory and in the field for determining the consistency of grout mixtures by measuring the time of efflux of a specified volume of grout from a standardized flow cone or funnel. The flow cone (Fig. 6) is mounted firmly with the top surface level, the discharge tube is closed by placing the finger over the end, and 1725 mL of mixed grout is poured into the cone. The finger is removed, and the number of seconds until the first break in the continuous flow of grout is the efflux time. When comparing grouts, the speed of mixing and the mixing time have an influence on efflux time and should be kept constant.

Grout Consistency Meter

A meter for measuring the consistency of grout has been developed at the University of California and is described elsewhere [42]. The grout consistency meter is essentially a torque meter. The sample of grout is placed in a metal pan mounted on a platform that can be rotated at a constant speed of 60 rpm. Suspended from a music wire is a 7.3 kg (16 lb) paddle assembly to which a torque is applied as the sample of grout is rotated. The angle of twist or consistency factor is read by an index pointer attached to a cross strut.

Measuring Workability of Self-Consolidating Concrete (SCC)

The general characteristics of SCC make the usual workability tests ineffective. The high flowability and variable viscosities of SCC have caused the development of a series of new test methods. Some of these tests will be developed into ASTM test methods while others will not. All of these test methods have a goal of being relatively inexpensive and providing immediate results, with good repeatability (single operator) and repro-



Note – Other means of indicating grout level may be used as long as accurate indication of grout level is obtained.

Fig. 6—Flow cone.

ducibility (multilaboratory). The chapter in this volume by Daczko and Vachon presents detailed information on self-consolidating concrete and the testing methods associated with its workability.

Two-Point Workability Tests

Tattersall [43] discusses the principles of measurements of the workability of fresh concrete and a simple two-point test. In this paper, the author points out that an understanding of workability of fresh concrete is important for the following reasons: (a) to make possible the design of mixtures for particular purposes; (b) to provide a method of control in the manufacture of the mixture; and (c) to contribute to the efficient use of manufacturing processes such as vibration, pumping extrusion, and finishing.

Tattersall contends that in spite of all the efforts (papers that have been written and the many proposed tests) over the past 30 plus years, there is no test that is fully satisfactory and the property of workability cannot even be defined except in the most general terms. Each of the test methods is capable of classifying as identical concretes that can be shown to be dissimilar.

Tattersall [37] suggests the following summary of proposed terminology as an effort toward standardization.

I. Qualitative
    Workability
    Flowability
    Compactibility
    Stability
    Finishability
    Pumpability
II. Quantitative empirical
    Slump
    Compacting factor
    Vebe time
    Flow table spread
III. Quantitative fundamental
    Viscosity Mobility Fluidity
    Yield value

The objection to the several workability tests are that, almost without exception, they are single-point tests, whereby only one measurement is made at one specific rate of shear or set of shearing conditions. Tattersall [44] states that such a procedure is valid only for a simple Newtonian liquid whose flow properties are completely defined by the constant ratio of stress to shear rate, and that ordinary observation shows that fresh concrete is not a Newtonian liquid and that, consequently, any test based explicitly or implicitly on the assumption that it is will be inadequate.

Tattersall also states that there is evidence to indicate that, in practice, it may be sufficient to treat freshly-mixed concrete as conforming with the Bingham model, which describes a material with a yield value and a plastic viscosity, which together describe the shear stress of the material. The yield value and plastic viscosity are constants, and it follows that measurements at two shear rates are required to determine them. The balance of this paper by Tattersall [44] discusses test procedures, results, and conclusions, and further modifications to allow application of vibration.

Tattersall [37,43,45] further explains the rationale of a two-point workability test and the relationships between slump, compacting factor, Vebe time, and the two-point test.

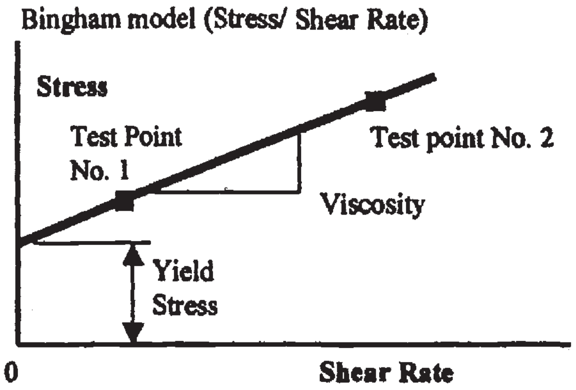


Fig. 7—Rheometer test results.

Rheometers

The basis of the two-point test to measure workability is a variable constant-speed rheometer. In the last ten years five rotational rheometers have been designed to measure the flow properties of concrete [46]. Three different types of configurations are used in these instruments. Two use coaxial cylinders, one uses parallel plates, and the other two use rotating vanes. Unfortunately each rheometer produces numerical test values that are not directly comparable to values from a different rheometer design. Brower and Ferraris [46] found when testing several different concrete mixtures in each of the five styles of rheometers that comparisons of test results were reasonably good. It is nevertheless advisable, if the properties of different concrete mixture proportions are to be compared, that all tests be performed in one style of rheometer.

IBB Rheometer

The IBB Rheometer, the principals of which were developed by Tattersall [37,47], is not like other rheometers. The original design of the IBB rheometer was a converted food-mixer with a planetary motion and ultimately a pressure gage and an H-shaped impeller. The test procedure consists of measuring the power required at three separate speeds to operate an 18.9 L (20 qt) food mixer when empty, then repeating the power measurement at all three speeds when the bowl contains a prescribed quantity of concrete. Values for yield and plastic viscosity are obtained by plotting $(P - P_E) / w$ against w, where w is speed, P is power under load, and P_E is power when the bowl is empty. Tattersall [37] claims thousands of tests have proven beyond doubt that the flow properties of fresh concrete closely approximate the Bingham model and that there is a simple straight line relationship between torque and speed with an intercept on the torque axis. The two-point test yields more information concerning the performance of a concrete mixture (Fig. 7) and appears to have good potential for determining and consequently controlling concrete uniformity.

Conclusions

Concrete knowledge and technology have advanced slowly during the years, but additional knowledge will be required if concrete is to maintain the position it has established as the universal building material. New products and new technologies are being developed in all phases of the concrete industry at a rapid rate as evidenced by SCC, truck mixer slump meters, and an increasing use of rheology in concrete research. Improvements in concrete production, control, delivery, and placing techniques are also being developed. At the same time, more widespread use of available knowledge for controlling uniformity, quality, and workability will improve the competitive position of concrete.

New methods of mixing, placing, consolidating, and finishing concrete may permit the use of less water and improve concrete quality. New tests and methods of quality control and measurement of workability must be developed concurrently with methods of mixing and placing concrete. The slump test and other tests used to measure uniformity may be replaced by more efficient test methods in the future.

Meters have been developed to measure moisture content of sand and coarse aggregate in the bins at concrete plants and promote better control of mixing water. The widespread use of aggregate moisture meters, particularly in sand, has greatly improved the quality control of concrete throughout the industry.

The development of slump meters for equipment, both hydraulic and electrical resistance, has improved the consistency of concrete while being mixed and transported, but this technology is expected to become even more accurate in the future. The slump test and other tests used to indicate consistency can be made only after the concrete is discharged, and corrections can be applied only to subsequent batches. Perhaps future research will reach into such areas as detecting changes in aggregate absorption and gradings during the batching process.

The slump test is an old friend and has served its purpose well. Some say that the slump test has already outlasted its time, but several of the new tests for SCC are being developed around the slump mold. As antiquated as it sometimes seems the slump test continues to be in demand because of its simplicity, economy, and friendliness in the field. The development of improved testing procedures for freshly mixed concrete in both the laboratory and the field remains a requirement of the concrete industry.

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9

Air Content, Temperature, Density (Unit Weight), and Yield

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Preface

THIS CHAPTER CONTINUES THE TRADITION OF previous editions of *ASTM STP 169*. Due to the fundamental nature of these topics, much of what is here has appeared before in *ASTM STP 169B*, authored by F. F. Bartel, and *ASTM STP 169C*, authored by the present author. This chapter updates the language and expands on the utility of density measurement in conjunction with air content measurement to improve test reliability. The author would also like to acknowledge the support and encouragement of his company and the many colleagues who have provided useful insight.

Introduction

Measurements of air content, temperature, and density (unit weight)[2] of freshly mixed concrete are the backbone of field quality control of concrete construction. Without these, proportioning of mixtures, control of yield, and assurance of placeability, strength development, and durability of concrete exposed to freezing conditions are not possible. The tests involved are relatively straightforward, use simple equipment, and are thoroughly described in the applicable ASTM test methods. Despite this, job problems, ranging from simple disputes over yield to complete performance failures, frequently can be attributed directly to faulty application of these methods. The purpose of this chapter is to help the interested concrete technician understand some of the key issues controlling the correct application and interpretation of the methods, and hopefully to encourage more careful and frequent application.

Overview of Significance and Use

Temperature measurement of fresh concrete is vital to ensure adherence to maximum temperature specifications, usually imposed to control thermal gradients and possible resultant thermal cracking as the cement hydrates and then cools to ambient conditions, and to minimum temperature specifications, imposed in cold weather to ensure adequate setting and strength performance.

Tests for air content and density are performed on fresh concrete to provide a control of these properties in the hardened concrete, and to determine the volume of concrete being produced from a given batch. The yield data so obtained are then available for the calculation of unit cement and aggregate contents, which are essential in mixture development and may be required in some specifications. Tests for unit weight are also performed to control concrete weight per se of both lightweight and heavyweight concretes.

While air content is most commonly determined to ensure the presence of air entrainment for freeze-thaw durability, knowledge of air content of non-air-entrained concrete is also important, due to the strong negative impact that unexpected increases in air content can have on compressive and flexural strengths. The author is aware of a number of job problems in non-air-entrained concrete that could have been prevented by air measurement or even simple density measurements, thereby significantly reducing the economic impact caused by excess air, brought about by contaminations, changes in materials, etc. Thus, checking air content and density is also a necessary safety precaution for these concretes.

Air contents of air-entrained concretes can vary for a large number of reasons. Among these are changes in air-entraining admixture type or dosage, changes in cement alkali content [1,2], fine aggregate grading, slump, concrete temperature, mixing intensity and duration, and many others. While a discussion of the impact of each of these is beyond the scope of this work and is well-covered elsewhere [3–5], suffice it to say that the possibility of a change due to any one factor is great enough to warrant close control of the air content. Further, nominally non-air-entrained concretes can also develop significant air contents, due to errors in batching or contamination of materials. The interrelationship between air content and density should be obvious: an increase in volume of air results in a lower density, all other material contents being unchanged. The calculation of material quantities per unit volume from the density depends not only on the correct determination of the concrete density, but also on the accurate knowledge of the weights actually batched. If the density test is to be used to estimate air content directly from proportions, then the specific gravities of the aggregates and cement (usually taken as 3.15 when portland cement is used) must be accurately known. Slight errors can result in significant error in estimating the air content. For this reason, the gravimetric determination of air is much more useful to

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[2] The older term unit weight is now deprecated, but is included here as reference. Density will be used in the remainder of this document.

monitor variation in air content of mixtures, the reference air content of which is determined by other methods. Also, when air contents vary in concretes being controlled to a fixed slump, the density changes may be less than predicted by air content changes alone, due to the reduction of required water for equal slump in many concrete mixtures as air content increases, and the gravimetric method can be used only if all the water weights are accurately known.

It must be emphasized that all methods to be discussed here measure the total air in the concrete, subject to the limitations of each method. While the adjectives "entrapped" and "entrained" are sometimes applied in technical discussion, and unfortunately sometimes in contract documents, to distinguish between large and small air voids, respectively, these methods cannot in any way make such a distinction. Furthermore, the fresh concrete air contents specified by the American Concrete Institute (ACI) and others for concrete durability under freeze-thaw are uniformly the total measured air contents, yet are referred to as entrained air. While some conclusions may be made for research purposes about the expected amount of small air voids by comparing the air contents of similar air-entrained and non-air-entrained concretes, there is no clear dividing line even by microscopic examination, and such distinctions are purely arbitrary. In some cases, contract language for "entrained air content" has been interpreted to mean that the specified level should be in addition to the base air content of non-air-entrained concrete. This is an inappropriate application of these fresh concrete methods, since no such distinction is possible.

Sampling

These methods are regularly applied under a variety of conditions—in the laboratory during mixture proportion development, at the plant for control of production, at the job site discharge point—to ensure compliance with specifications, and, at the point of placement, to best estimate the resulting hardened concrete properties. Due to the limitations of these various locations, a clear understanding of proper sampling procedures and acceptable deviations from test specifications is needed.

For example, all the methods to be discussed nominally require that concrete be sampled according to ASTM Practice for Sampling Freshly Mixed Concrete (C 172), which requires that the concrete come from two or more portions of the load taken at regularly spaced intervals during discharge of the middle of the batch. This is clearly not possible if a truck is being sampled prior to discharge for compliance with specifications, which is perhaps the most common application of these methods. This represents an inconsistency that needs resolution; therefore it is best if the specification documents clearly outline the sampling requirements. In practice, samples must be taken from the initial discharge, but enough concrete must be allowed to discharge, usually about 10 %, to obtain concrete representative of the load, and all the other directions of care described in ASTM C 172 must be adhered to. If, after testing, obvious changes in the concrete being discharged are noticed, this must be noted and good practice indicates re-testing. Resolving this sampling time issue takes a clear eye for concrete, good judgment, and is sometimes a source of contention on job sites.

One source of error in sampling during application of these methods deserves special mention. Frequently, one or more of the air content methods is applied to a load of concrete arriving at a job site at the same time the slump test, ASTM Test Method for Slump of Hydraulic Cement Concrete (C 143), is run. The air is found to be within specifications, but the slump is too low, and the specifications allow addition of retempering water. This water is added, the slump re-run and found to be within specifications, but the air content of the retempered concrete is not determined. The addition of water and further mixing alter the air content, and subsequent determinations of the hardened air contents may not agree with the results recorded for the fresh concrete properties. Questions about the accuracy of the air content measurements then arise, solely because the concrete tested in the fresh state was altered after testing but before being allowed to harden. The job specifications should make clear what steps are to be taken in this situation.

Test Methods

Temperature

Concrete temperature measurement is determined in accordance with ASTM Test Methods for Temperature of Freshly-Mixed Portland-Cement Concrete (C 1064). This test method describes the types and precision (0.5°C) required of the temperature measuring devices to be used. Although liquid in glass thermometers may be used, the conditions of field concrete testing in many cases make metal dial thermometers more practical, and these are most frequently employed. Yearly calibration against 0.2°C precision reference temperature measuring devices is required, using two temperatures at least 15°C apart; but due to the ease with which some metal dial thermometers can lose calibration, it is recommended that a single temperature comparison against a reference thermometer be performed daily as an equipment check, with a full recalibration being run if deviation is noted. Formerly, precision liquid in glass reference thermometers was required by the test method, but now direct-reading resistance thermometers are acceptable, so long as their calibration is done at least yearly, and is traceable to NIST standards.

Another key provision of ASTM C 1064 relates to sample size. Clearly, if the sample is small enough to gain or lose significant heat to its surroundings during the time of testing, the result will not be representative of the mass of the concrete. Accordingly, ASTM C 1064 calls for the sample to be large enough for a minimum of 75 mm of concrete to surround the temperature-measuring device.

While the test method does not now explicitly indicate this, it is obvious that the measuring device should remain embedded in the concrete while the reading is being taken, as rapid evaporative cooling could reduce the temperature read if the device were withdrawn prior to reading. Language to clarify this is currently in the balloting process.

Finally, aggregate of 75 mm or greater in size may cause the concrete to take as much as 20 min to temperature equilibrate, especially if there are large differences between the aggregate and other material temperatures. Thus in large aggregate concretes sampled early, replicate readings over time are warranted to ensure that the equilibrium is reached. No guidance is given in the method as to how far apart these should be, but, for practical purposes, if after a 5 min delay the temperature remains within 1°C, effective equilibrium is reached.

Air Content

The three tests for air content of fresh concrete, using pressure, volumetric, and gravimetric methods, each have their own advantages and limitations. We will discuss each in order, with focus on the proper selection of method.

Pressure Air Measurement

The pressure method for determining air content of fresh concrete is based on Boyle's law, which states that the volume of a gas is inversely related to the pressure. By applying pressure to a known volume of concrete containing air voids, the voids are compressed, the concrete is reduced in volume, and the volume change can be measured and related to the initial volume. Knowledge of the pressure difference allows calculation of the total volume of air. This principle was first applied by Klein and Walker in 1946 [6], while Menzel [7] refined the apparatus and proposed a standard test procedure. This method has the advantage that no knowledge of specific gravities or batch weights is required to obtain the required answer.

Two types of meters are defined in ASTM Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231). The Type A meter relies on direct measurement of the volume change, by means of a column of water above the known volume of concrete. A calibrated sight tube allows measurement of the volume reduction directly as pressure is applied. This method is very straightforward, but has the drawback that recalibration is necessary if barometric pressure or elevation changes exceeding 183 m occur. A change of 183 m is approximately equal to a 2 % change in barometric pressure at sea level.

In the Type B air meter, a known volume of air at an established higher pressure is allowed to equilibrate with the known volume of concrete in a sealed container. The drop in pressure measured in the high-pressure air chamber can be related to the amount of air within the concrete. This method does not have the ambient air pressure recalibration requirement of the Type A meter, but the complexity of valves and seals make the apparatus prone to leakage; operators should be prepared with tools and replacement parts for repair, and be sensitive to instability in dial readings that may signify leakage and therefore incorrect results. A common mistake is to close the petcocks prior to pressurizing the high-pressure chamber. If this is done, it is possible to not observe a leak in the needle valve that would normally be noticed due to air bubbles escaping the open petcocks. This would result in incorrectly low air content readings.

This method requires complete consolidation of the concrete in the bowl; any large air voids due to lack of consolidation will be measured as air content of the concrete. To ensure proper consolidation, rodding is required above a slump of 75 mm, internal vibration below 25 mm, and either in between. Vibration should cease when all the coarse aggregate is submerged and the surface takes on a smooth, glistening appearance. Extreme care must be taken to avoid removal of the intentionally entrained air by over-vibration, and for this reason vibration is rarely used in slumps above 25 mm. Concretes containing aggregates larger than 50 mm must be screened using the 37.5 mm sieve prior to testing, since representative sampling becomes difficult with larger aggregate.

Strike-off of the concrete is possible with either a bar or a strike-off plate. In the case where only a pressure air test is being run, the precise volume of concrete is less critical, and the bar is acceptable. When the air meter base is being used for determination of density, a strike-off plate must be used, as described later.

The pressure method is limited to use with concretes containing relatively dense aggregates. Air in the interconnected porosity within the aggregate particles will be compressed just as air within the cement paste, thus indicating a higher than true air content. This is corrected for by application of the required aggregate correction factor, the lack of which application is a common flaw in observed field testing procedures. While this correction factor will compensate for the air in the aggregate, it is not appropriate to apply the pressure test to concrete containing lightweight aggregates in which the aggregate correction factor exceeds about 0.5 %, because with aggregate of this high porosity, relatively minor changes in aggregate porosity will lead to significant variation in measured air content.

Due to the use of water, the concrete must be discarded at the end of the test.

A degree of controversy has arisen regarding the ability of the pressure meter to measure air content when the air voids are very small. This is usually perceived to occur when the air contents are high and may be to some extent dependent on the type of air-entraining agent used. Several authors have reported significantly higher air contents in hardened concrete measured according to ASTM Practice for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete (C 457) than was obtained using the pressure method [8–12], as shown in Fig. 1. Meilenz et al. discussed the theoretical possibility of air in the smaller, higher pressure air voids dissolving, then coming out of solution in lower pressure, larger voids, leading to a net increase in air volume [13]. Hover [14] calculated the effect of the incompressibility of very small air voids. Other authors have failed to find such an increase [15].

Closer examination of the data indicates the problem may largely be a manifestation of the sampling problem mentioned earlier. While Hover's calculations [14] show that significant underestimation could occur if the very small air voids comprised a significant portion of the air volume, this is not the case, even in concretes of high air content [15]. Thus, this underestimation is unlikely to exceed around 1 % air content, not the 3–6 % reported by some.

Ozyldirim [16] followed up on field reports of higher air in the hardened concrete with a thorough test program in which all types of fresh concrete air content tests were compared with hardened air results from ASTM C 457. In no case did the differences display the extreme variation previously reported. Only when the concrete was retempered and the hardened air contents compared to the fresh concrete results prior to retempering were significant differences seen. Re-examination of the data included in reports of such underestimation problems [10]

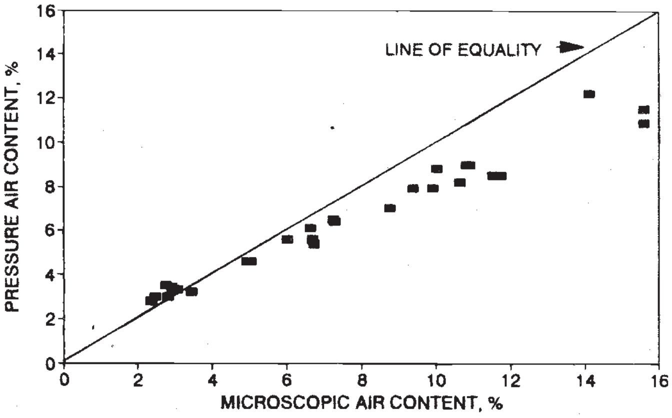

Fig. 1—Pressure versus microscopic air content (data from Ref 10).

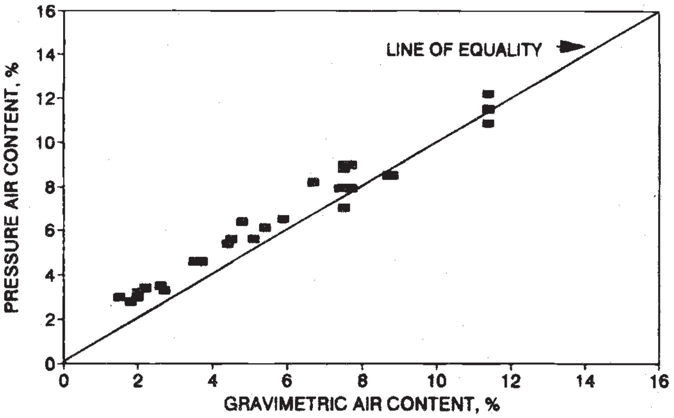

Fig. 2—Pressure versus gravimetric air content (data from Ref 10).

shows that the pressure test corresponds well with the gravimetric method results (see Fig. 2). This would seem to preclude a specific problem with the pressure test, although some change in the actual concrete from fresh to hardened state could be invoked.

Further consideration makes this unlikely. The air differences reported are up to 6 %. Accepting that the fresh concrete air content measurements are accurate, a net growth of the concrete would be required to cause this measured difference. This would result in up to an 18 mm increase in height of a typical 300 mm cylinder. Such growth is not reported. The answer may well lie in the difficulties associated with the ASTM C 457 test, which are covered elsewhere [3]. Some preliminary data showing this effect have been presented [17], as seen in Table 1. Poor sample surface preparation tends to erode the void edges, effectively making them seem larger, and thus contributing higher measured air contents.

Volumetric Method

The ASTM Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173/C 173M) measures air content by washing the air out of the concrete through agitation of the concrete with an excess of water containing some level of isopropyl alcohol, primarily by a rolling action. A known volume of concrete is covered by water, an amount of isopropyl alcohol appropriate to the type of concrete added, and the water level adjusted to a zero mark in a calibrated clear neck on the described apparatus. The apparatus is sealed; then the concrete, alcohol, and water are agitated by rocking and rolling until the air in the original concrete is displaced and rises to the top of the neck of the apparatus. The drop in water level from its original mark provides a measure of the air content. During agitation, it should be possible to hear the aggregate rolling around within the chamber. To ensure that all the concrete is dislodged from the base, it is frequently necessary to tip the apparatus upside down, but this should be done briefly, as aggregate particles can lodge in the neck of the meter. If this occurs, they usually may be dislodged by sloshing the apparatus from side to side. The agitation must be repeated until there is no further drop in the water level. As defined in ASTM C 173/C 173M, this is considered to be when there is less than 0.25 % change in the measured air content.

TABLE 1—Effect of Surface Preparation on Measured Air Content (Data from Ref 17)

Air Content, %		
	ASTM C 457	
ASTM C 231	Good Polish	Bad Polish
8.0	7.7	11.0
8.8	8.4	11.2
7.5	7.23	8.69

This method has the advantage that air trapped in the aggregates has no impact on the test results, and thus is the method of choice for lightweight aggregate concretes. However, it has two significant disadvantages. First, the effort involved in agitating the filled apparatus is significant and can lead to severe operator fatigue over a work day. The fatigue factor can be reduced by using smaller apparatus with containers of 2.0 L capacity as are now allowed. Second, it is absolutely vital for accurate measurements that the agitation indeed be repeated until no significant change in air content occurs. These two factors work in opposition to each other in practice, especially in high cement factor, sticky concretes having high air contents.

The operator should be alert to any free water on or around the apparatus, indicative of a failure to properly seal the unit. Such a leak will result in a higher air content being measured than actually is present in the concrete. Fine particles adhering to gaskets and the cap frequently cause such leaks.

A further constraint is that with high-air-content concretes it may be very difficult to dispel residual bubbles of air in the neck of the apparatus. It is for this reason that the addition of isopropyl alcohol, previously an option, is now required in the test method. Generally the higher the air content and the stickier the concrete, the more isopropyl alcohol will be found necessary to dispel the bubbles during the first rocking and rolling, as described in notes 2 and 3. When needed, further increments of isopropyl alcohol may be added using the small cup provided, the volume of which is equal to 1 % air content as measured in the calibrated neck. Care must then be exercised to note the volumes added and add to the air content finally read as described in the calculation section of ASTM C 173/C 173M. For a consistent class of concrete, generally an ideal level of isopropyl alcohol will be found and it should be added to each test. When the test operator is monitoring more than one class of concrete on a job site, very different levels of isopropyl alcohol may be needed, and it will require extreme care to remember to add the different amounts to different concrete classes.

Recent reports of problems with volumetric measurement of high-air-content concretes have paralleled the problems attributed to the pressure method as described earlier, and may be related to the wider application of air-entrained high cement factor concrete in recent years. In most cases, the air contents measured by pressure and volumetric methods agree with each other reasonably well, and with the gravimetric method, leading to the conclusion that the problem is not in these test methods. Figure 3, based on data from one paper in which a lack of correspondence with hardened air contents is shown [10], confirms the good cross-correlation of fresh concrete methods. This is in line with previous experience [18,19].

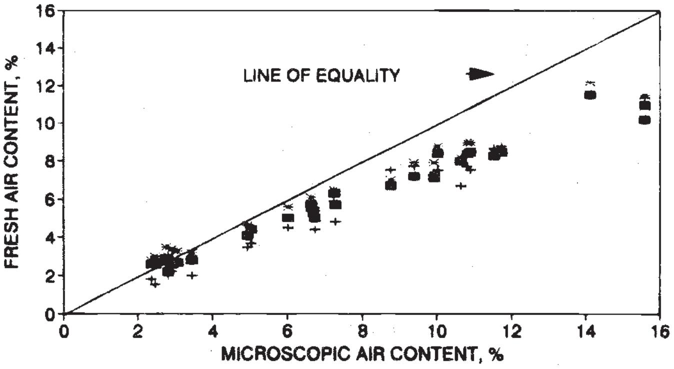

Fig. 3—Fresh versus microscopic air content (data from Ref 10).

However, in some instances [20], it does appear that with extremely sticky concretes it can be so difficult to wash out all the air that at the very least the volumetric method takes an impractically long time to complete (this information was previous to the inclusion of isopropyl alcohol). Therefore, care should be exercised in the use of this method when such concretes are encountered, with special attention paid to using the proper amount of isopropyl alcohol, to continuation of agitation until no air increases are registered, and to the checking of the bowl after completion to ensure that all the concrete was loosened from the base. If such attention results in unacceptably long test times, alternate methods should be employed. Such situations may require two test technicians to ensure that fatigue does not unduly influence the willingness to ensure that thorough separation of the concrete has taken place.

Gravimetric Method

In the gravimetric procedure, the density of concrete is determined in accordance with ASTM Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete (C 138/C 138M). The concrete is placed in a container of known volumetric capacity and weighed. Using the known volume, the density of the concrete is calculated and, from this, the air content and yield can be determined provided the requisite mixture proportion information is available. This dependence on extraneous information is a major limitation of this method when used for other than density.

Aggregate size determines the minimum capacity of the measures used, since as the aggregate increases in size, edge effects tend to bias the sampling toward lesser aggregate contents. The minima given in ASTM C 138/C 138M are reproduced in Table 2, from which it is important to note the very high volumes required when aggregate size exceeds 25 mm. Safety considerations require that means be provided for movement of the buckets on and off the scales without undue strain on the individuals conducting the test.

As with the other air content methods discussed here, the results of this test are completely dependent on proper filling and consolidation of the measure. Rodding may be used above 25-mm slump; vibration is required for lower slumps. Vibration may not be used if the slump exceeds 75 mm. ASTM C 138 requires that measures smaller than 11 L be consolidated by rodding, due to the possibility of excessive loss of entrained air, while ASTM C 231 permits vibration at this size. Thus, the smallest measures cannot be used in accordance with ASTM C 138/C 138M for concretes of less than 25-mm slump.

The top surface of the concrete must be obtained with a strike-off plate. This is best accomplished by covering about two-thirds of the surface with the plate, pressing down, and withdrawing with a sawing motion. Then replace the plate on the smoothed two-thirds, press down, and advance it over the unsmoothed portion with a sawing motion. Striking off with a bar is specifically not permitted, as it leads to a less precise filling. Do not overfill the measure then push the coarse aggregate down into the measure with the strike-off plate, as this results in mortar being squeezed out, giving a nonrepresentative sample.

Subsequent calculation of volume of concrete produced per batch, the relative yield, actual cement content per unit volume, and air content are outlined clearly in ASTM C 138/C 138M, and will not be repeated here. Each calculation requires precise knowledge of batch weights and material properties not readily available in the field. When the gravimetric procedure for determining air content of fresh concrete is used, the density of concrete as determined is compared with the theoretical density of air-free concrete. This is calculated from the weight and specific gravity of each ingredient of the concrete mixture, and requires knowledge of the saturated, surface dry specific gravities and their moisture contents as batched. This can lead to serious errors. For example, when there is a 2 % error in the moisture content of the fine aggregate or an error of 0.04 in the aggregate specific gravities, an approximate 1 % error in the air content computed according to ASTM C 138/C 138M will result. Thus, ASTM C 138/C 138M is not appropriate for determining air contents of lightweight aggregate concrete, since aggregate-specific gravities and moisture contents are both subject to wide variation.

Further, when air contents increase, required water contents will be reduced in most concretes. The amount of reduction is greatest in the lower cement content mixtures [21], due to the air substituting for the water needed to fill in the voids between the fine aggregate particles. Accordingly, air content increases measured by ASTM C 138/C 138M would be reduced by approximately one-third unless completely accurate water content information were available.

Due to these uncertainties associated with direct calculation of air by the gravimetric method, it is the author's experience that the greatest value is realized when density is used as a cross-check to other methods of air measurement, and that in that regard it is extremely valuable. Testing of concrete air in the field is subject to a number of errors, many of which are enumerated above. The ability to have a simple cross-check by

TABLE 2—Minimum Capacity of Measures for Use in ASTM C 138

Nominal Maximum Size of Coarse Aggregate, mm (in.)	Minimum Measure Capacity, L (ft^3)
25.0 (1)	6 (0.2)
37.5 (1 1/2)	11 (0.4)
50 (2)	14 (0.5)
75 (3)	28 (1.0)
112 (4)	71 (2.5)
150 (6)	99 (3.5)

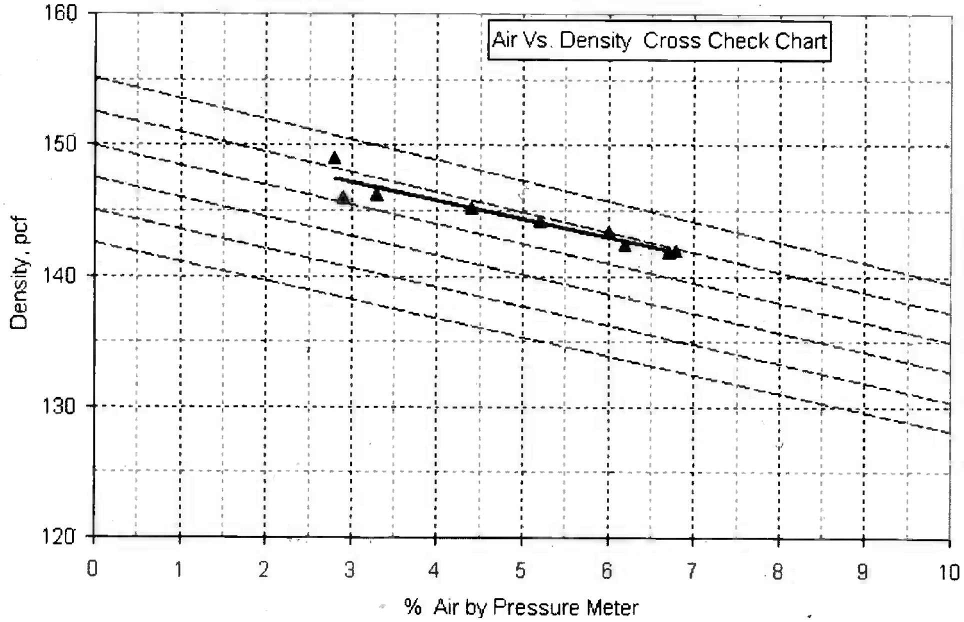

Fig. 4—Air versus density data from field testing of different trucks during one day of field testing.

which problems with the apparatus or method can be found quickly can prevent incorrect rejection of concrete, and also incorrect adjustment of air-entraining agent doses. Figure 2 is an illustration of such a use.

The author is aware of a number of cases in which concrete ultimately had to be removed because faulty pressure method air measurement at the job site indicated low air and plant dosage adjustment was made. Later, when strength problems led to determination of high air in the hardened concrete, the faulty testing became manifest. This could have been easily precluded in the case of pressure meter testing by the simple expedient of using the air pot base to determine the density, and keeping a running simple air cross-check graph as shown in Fig. 3. Figure 4 shows data from a typical well-run field test of a number of different admixtures yielding different air contents, with the same base concrete mixture. The dotted lines represent the expected relationships, while the fitted regression line shows by its slope that the test is well controlled. On close inspection, one mix, at 2.9 % air, is somewhat questionable and would bear repeating; Fig. 5 shows the improvement when that mix is eliminated from the dataset. Having prepared blank graphs at the job site on which the data can be plotted makes this approach easier.

Since no water comes in contact with concrete tested according to ASTM C 138/C 138M, the concrete may be used in other testing.

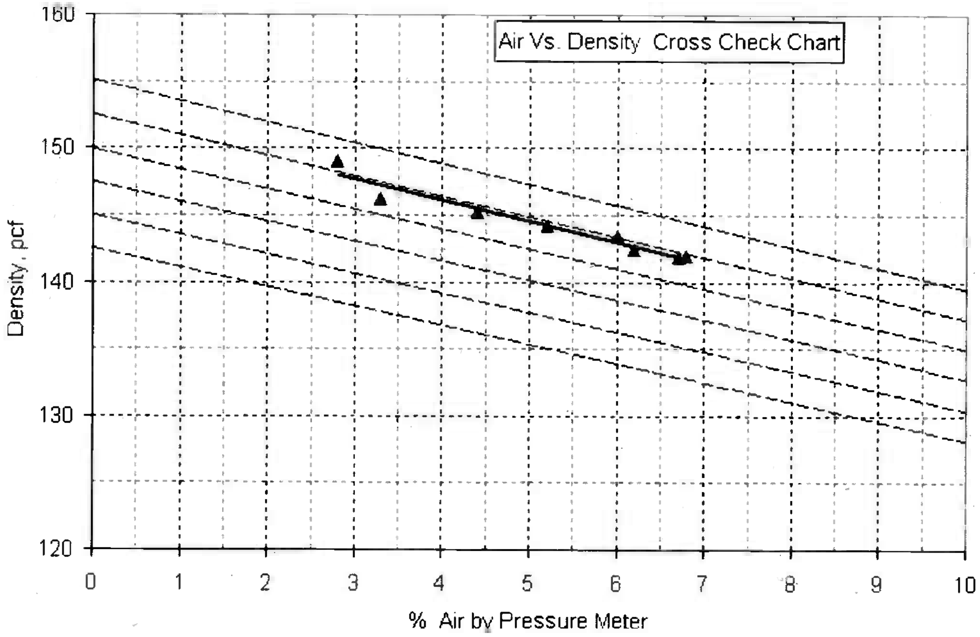

Fig. 5—Air versus density data from field testing of different trucks during one day of field testing, after elimination of one data point.

Summary and Future Trends

Tests run in accordance with these methods will yield accurate and useful results providing the operator follows the methods carefully and care is taken to avoid procedural pitfalls or use of methods inappropriate in certain situations, as described earlier. The specification writer should take cognizance of these limitations and not ask more of the methods or the operators than they can provide.

The growing emphasis on high quality, durable concrete, coupled with advances in digital electronics, are changing this situation. Numerous in situ temperature measurement systems are available, and are employed for maturity methods under ASTM C 1074. A complex, but potentially valuable test known as the Automatic Void Analyzer, or AVA, has been developed [22], based on buoyancy of released air voids. This shows promise in allowing determination not only of air content but also of air-void size in fresh concrete. There are, however, significant issues of sampling reproducibility, time delay if used as an acceptance method, and experimental issues such as sensitivity to vibration, which requires setting up the equipment remote from job sites or using special vibration isolation techniques. Current use is generally for mix qualification, with job site control still by total air content methods [23]. Evaluation of its reproducibility is currently underway, and possible biases between the results of the method and actual hardened air parameters per ASTM C 457 are not well understood. Some specifying agencies are currently using the method. Good practice would indicate that different mixes should have the AVA results calibrated against actual ASTM C 457 prior to setting ranges in specifications, but this has not always been the case. Focus on this method is likely to bring significant advances in the understanding of its use and appropriate application in the near future.

It is expected that the effort to improve the convenience and rapidity of test methods will yield significant advances in the next decade. For now, the methods discussed here provide excellent quality control of fresh concrete when properly applied.

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10

Making and Curing Concrete Specimens

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Preface

THIS CHAPTER COVERS THE IMPORTANCE OF PROperty making and curing concrete test specimens in both the field and the laboratory. This subject was covered in *ASTM STP 169* [1] in the chapter Static and Fatigue Strength authored by C. E. Kelser and C. P. Siess. In *ASTM STP 169A* [2], this became a separate chapter authored by T. B. Kennedy. In *ASTM STP 169B* [3], this chapter was authored by R. F. Adams. In *ASTM STP 169C* [4], this chapter was under the present authorship.

Introduction

The 1914 Committee Report [5] is the basis for the presently accepted procedures for testing concrete cylinders and beams to determine the compressive or flexural strength. Two ASTM standards have been developed for making and curing test specimens. One is ASTM Practice for Making and Curing Concrete Test Specimens in the Field (C 31) and was originally published in 1920 and updated periodically. The other is ASTM Practice for Making and Curing Concrete Test Specimens in the Laboratory (C 192) and was originally published in 1944 and updated periodically. Since publication of *ASTM STP 169C* [4], C 31 has been revised six times and C 192 has been revised four times. These two standards have had considerable use in concrete research and concrete construction. Specimens have to be made and cured properly since departures from the standard procedures to make and cure specimens will affect the test results.

Applications

The strength of concrete is one of its most important and useful properties and one of the most easily measured. The strength indicated by the specimens is affected by many variables encountered in making and curing test specimens. These include size of the aggregate, size and shape of the test specimen, consolidation of the concrete, type of mold, capping procedure, curing, and temperature. The effect that any of these variables has on the apparent strength of the specimen will often vary depending on the particular circumstances. Among the many who have written about factors that influence the strength of concrete are Price [6], Sparkes [7], *ACI Manual of Concrete Inspection* [8], Walker and Bloem [9], and Richardson [10].

The strength of concrete, in compression, tension, and shear, or a combination of these, has in most cases a direct influence on the load-carrying capacity of both plain and reinforced structures. In most structural applications, concrete is designed primarily to resist compressive stresses. In those cases where strength in tension or in shear is of primary importance, empirical relationships to the compressive strength are often used. However, strength may not necessarily be the most critical factor in the overall performance of the concrete. For example, the need for acceptable durability may impose lower water-cement ratios than required to meet the strength requirements. In such cases, the actual compressive strength of the designed mixture may be in excess of structural requirements. Specimens cast in the field are most often used to determine the compressive strength in accordance with ASTM Test Method for Compressive Strength of Cylindrical Concrete Specimens (C 39). The test results may be used as an indication of the verification of other required properties or characteristics of the concrete as delivered or designed. The most common other use is to determine the splitting tensile strength in accordance with ASTM Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens (C 496). Pavements are often designed for tensile stresses in flexure in the concrete slab. Traffic loads have been found to induce critical tensile stresses in a longitudinal direction at the top of the slab near the transverse joint, and in the transverse direction near the longitudinal edges. Accordingly, a flexural strength test, either ASTM Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78) or ASTM Test Method for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293), is used as an acceptance test for most pavement concrete. However, due to inherent variability of the flexural strength tests many agencies rely on compressive strength tests using an established correlation or empirical relationship to estimate flexural strength.

It is important to keep in mind that the test specimens indicate the potential rather than the actual strength of the concrete in the structure. To be meaningful, conclusions on strength must be derived from a pattern of tests from which the characteristics of the concrete can be estimated with reasonable accuracy. An insufficient number of tests will result in unreliable conclusions. Statistical procedures provide tools of considerable value in evaluating results of strength tests. ACI Recommended Practice 214 [13] discusses variations that occur in the strength

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of concrete, and presents statistical procedures and control charts that are useful in the interpretation of these variations.

Testing Personnel

There is increasing emphasis and a requirement in many building codes, political jurisdictions, project specifications, and ASTM Specification for Ready-Mixed Concrete (C 94) that personnel who make concrete test specimens be certified. Over 59 000 technicians have been certified under the American Concrete Institute's certification program for "Concrete Field Testing Technicians, Grade I." Other equivalent certification programs that test technicians' knowledge of the standard and ability to perform the test also exist and are used.

Adams [3] discussed the importance that personnel are properly trained in making specimens and the details of the various procedures are precisely followed. Only in this way can meaningful and reproducible test results, which are not open to question, be obtained. The significant differences between trained and untrained personnel were reported by Wagner [14]. Wagner's study showed that strength tests made from the same concrete by trained personnel were higher and more uniform than those made by untrained personnel.

In addition to proper training, supervising engineers and technicians responsible for technicians making and curing test specimens must be thoroughly familiar with the test procedures. Personnel should have the required tools and equipment for making and curing specimens. The standards must be reviewed annually for any changes. Also, supervisors should review periodically the making and curing of test specimens and the procedure to verify that it is being done correctly. When strength test results fail to meet a specification requirement and rejection of the concrete is considered, the tests are almost always questioned, particularly if it can be shown that the person making the test had not complied with all the details of the test procedure.

Molds

Specifications

The molds for casting concrete specimens must have the following properties: (1) made of a nonabsorbent material, (2) nonreactive with the concrete (aluminum and magnesium are examples of reactive materials), (3) hold their dimensions and shape, and (4) be watertight. Compressive strength and splitting tensile test specimens are cast and hardened in a vertical position, with the length equal to twice the diameter. Flexural strength specimens are rectangular beams cast and hardened with the long axis horizontal. Molds for casting cylindrical specimens are covered in ASTM Specification for Molds for Forming Concrete Test Cylinders Vertically (C 470). Details for field-cast beam molds are covered in ASTM C 31. Details of molds for laboratory-cast flexural strength and freeze-thaw beams are covered in ASTM C 192. Creep specimens are cylindrical specimens cast horizontally. They are covered in ASTM C 192 and ASTM Test Method for Creep of Concrete in Compression (C 512). The requirements for molds may be covered in more than one standard; therefore, all the requirements must be considered for the type of specimen being molded.

Types

There are two types of molds, the reusable type and the single-use type. The reusable molds are designed to be used more than a single time. Reusable molds are made of nonabsorptive materials and constructed in one piece or several pieces. Single piece reusable molds are made most commonly of plastic while assembled molds are made of steel, iron, or brass. The assembled mold must be watertight; therefore, suitable sealants must be used where necessary to prevent leakage through the joints. The single-use mold may be made of any material that passes the test requirements of ASTM C 470. The most prominent single-use mold materials are plastic with treated paper products, metal molds, and other suitable material being used less frequently. At least three single-use molds and reusable plastic molds shall be selected at random from each shipment by the purchaser to ensure that molds are in compliance with C 470.

Richardson [10] reported on a number of studies and observations on the rigidity, water absorption, and expansion of molds made with various kinds of materials. Reusable steel molds not properly sealed had a tendency to leak at the joints. For high-strength concrete, compressive strength test results may be lower with plastic molds than reusable steel molds [11]. When not properly waterproofed, treated paper products (cardboard molds) may suffer from absorption and elongation problems. All molds can suffer from being out of round. This was noted even in some molds made with light-gage metal sidewalls. The shape tends to be oval at the unsupported tops for plastic and cardboard molds. The sidewalls of molds should be of a sufficient stiffness to prevent the mold from becoming out of round. This can be attained with sufficient wall thickness alone or in combination with a stiffened top. The molds that result in out-of-shape cylinders have an effect on the proper capping and the specimens not meeting dimensional tolerances.

Making and Curing Test Specimens in the Field (ASTM C 31)

This practice is a definitive procedure for performing specific operations to produce a test specimen. These specimens are used in various test procedures to determine a property of concrete. Specimens are fabricated using concrete delivered to a project site sampled from the transportation unit prior to placing. Specimens are fabricated in cylindrical or beam molds and consolidated by either rodding or vibration. The concrete slump measured by ASTM Test Method for Slump of Hydraulic Cement Concrete (C 143) is used to select the consolidation procedure. If the slump is greater than 1 in. (25 mm), the concrete is either consolidated by rodding or vibration. If the slump is less than 1 in. (25 mm), specimens must be vibrated. An example of a concrete with a slump less than 1 in. (25 mm) would be roller-compacted concrete. Roller-compacted concrete would be consolidated using vibration procedures in ASTM Practice Making Roller-Compacted Concrete in Cylinder Molds Using a Vibrating Table (C 1176) or ASTM Practice Molding Roller-Compacted Concrete in Cylinder Molds Using a Vibrating Hammer (C 1435). Other concretes that cannot be consolidated by rodding or vibration, or requiring other sizes and shapes of specimens to represent the product or structure are not covered by this practice.

Uses

ASTM C 31 states that specimens may be used to develop information for a number of purposes. There are two separate conditions for using cylinders or beams: (1) they must be standard cured and (2) they must be field cured. There have been instances where standard curing and field curing have been combined. This usually occurs when initial standard curing is misinterpreted as field curing. The two most frequent uses are standard curing for acceptance testing and field curing for removal of forms or shoring. When strength test results are used for acceptance testing, the cylinders or beams must be standard cured. Also, they are standard cured when used to check the adequacy of mixture proportions for strength or quality control. When strength test results are used for form or shore removal requirements, cylinders or beams must be field cured. Also, they are field cured for determination of whether a structure is capable of being put in service, for comparison with test results of standard cured specimens or test results from various in-place test methods, or to determine the adequacy of curing or protection procedures. Various in-place test methods are discussed in ACI 228 [15]. Curing procedures are discussed in the section on Curing Specimens.

Samples

For evaluation of the test results by statistical procedures to be valid, the data must be derived from samples obtained by means of a random sampling plan. Chapter 3 of this volume on sampling contains information on a random sampling plan.

The sampling must be made after all on-site adjustments have been made to the mixture proportions including addition of total mixing water and admixtures. The procedures for obtaining representative samples of concrete at a construction project from different types of delivery equipment are covered in ASTM Practice for Sampling Freshly Mixed Concrete (C 172), unless an alternative procedure has been approved. The size of the sample for fabricating field test specimens is a minimum of 1 ft^3 (28 L). The sample must be representative of the nature and condition of the concrete being sampled. The sample is collected by taking two or more portions at regularly spaced intervals during discharge of a stationary mixer, truck mixer, or agitator. The elapsed time between first and final portions should not exceed 15 min. The portions are combined and remixed to ensure uniformity and transported to where the test specimens are to be made. Molding of the specimens must begin within 15 min after fabricating the composite sample. The time should be kept as short as possible and the sample should be protected from contamination, wind, sun, and other sources of evaporation.

Concrete is a hardened mass of heterogeneous materials and its properties are influenced by a large number of variables related to differences in types and amounts of ingredients, differences in mixing, transporting, placing, and curing. Because of these many variables, methods of checking the quality of the concrete must be employed. Strength test specimens can only measure the potential strength of concrete in the structure because of different size and curing conditions between the specimen and the structure. Therefore, multiple test results based on a random pattern should be used as a basis for judging quality rather than placing reliance on only a few tests to check uniformity and other characteristics of concrete. For this purpose, statistical methods given in ACI Recommended Practice 214 [13] should be used.

Richardson [10] pointed out that various specifications require different numbers of replicate cylinders to be tested at differing time intervals, usually 7, 14, and 28 days. It is important to make sure that the concrete for a set of cylinders comes from a single truck. A set of cylinders that does not come from the same truck will cause a considerable amount of consternation should the 14-day measured strength be lower than the 7-day strength.

Test Data on Specimens

After all adjustments have been made to the mix including total mixing water and admixtures, measure and record the slump, air content, and concrete temperature. Also measure the density, if required. Chapter 8 in this volume contains workability information on the properties of fresh concrete. Chapter 9 in this volume contains information on the effects of various air contents on the properties of fresh concrete. And Chapter 13 in this volume contains information on properties of hardened concrete.

If concrete is air entrained, then the air must be controlled within tolerances. If air-entrained concrete is accidentally air-entrained, it has to be known and corrected because each percent increase in air content reduces the compressive strength. The concrete temperature test results may help explain some unusual strength results. The slump, air content, concrete temperature, and density test results should be used to assure the concrete is controlled within required tolerances.

Specimen Sizes

It is generally accepted that the diameter of the cylinder should be at least three times the nominal size of the coarse aggregate. For compressive strength specimens, a 6 by 12 in. (150 by 300 mm) cylinder is used when the aggregate is smaller than 2 in. (50 mm) or, when specified, 4 by 8 in. (100 by 200 mm) cylinders can be used when the aggregate is smaller than 1 in. (25 mm). For flexural strength specimens, the standard beam is 6 by 6 in. (150 by 150 mm) in cross section with a length of at least 20 in. (500 mm). Other test methods may require test specimens when cast in the field to be a specific size.

If the aggregate is too large for the size of mold available, the oversize aggregate is usually removed by wet screening as described in ASTM C 172. A larger specimen mold is used when specified and wet sieving is not permitted. In these cases, the diameter of the cylinder or the smaller cross-sectional dimension of the beam should be at least three times the nominal size of the coarse aggregate in the concrete. Attention must be called to the fact that the size of the cylinder itself affects the observed compressive strength; for example, the strength of a cylinder 36 by 72 in. (920 by 1830 mm) may be only 82 % of the 6 by 12 in. (150 by 300 mm) cylinder [12].

The advantages of 4 by 8 in. (100 by 300 mm) cylinders are as follows: 1) they are easier to fabricate, handle, and transport; 2) smaller storage space is required; 3) less capping compound is needed; and 4) higher strength concrete uses a smaller capacity compression machine. A cylinder smaller in size than 6 by 12 in. (150 by 300 mm) will yield a somewhat greater compressive strength [6,16]. The difference in strength between 4 by 8 in. (100 by 200 mm) and 6 by 12 in. (150 by 300 mm) cylinders increases with an increase in the strength level of the concrete [17]. Additional information evaluating cylinder sizes has been published [18–20]. The present C 31 permits the use of 4 by 8 in. (100 by 300 mm) cylinders and the specifier determines it is appropriate, recognizing mixture proportioning adjustments and any testing variability.

Making Specimens

Where the specimens are to be molded is important. It should be as near as practicable to where the specimens are to be stored. The molds should be placed on a firm and level surface that is free of vibrations and other disturbances. Select a small tool to fill the molds with concrete that is representative of the sample and the batch. Select the appropriate rod for the size of the specimen using a smaller rod for specimens less than 6 in. (150 mm) in diameter or width. Concrete should be placed in the mold to the required depth and with the required number of layers keeping the coarse aggregate from segregating as it slides from the scoop, trowel, or shovel. If the specimen is rodded, carefully count the strokes for each layer. The rod should not strike the bottom of the mold when rodding the first layer. If the specimen is to be vibrated, determine the best vibrator and the best uniform time of vibration for the particular concrete. Vibration may reduce the air content of air-entrained concrete. If air content reduction is suspected, verification can be performed by determining the density of the specimen and comparing it to the calculated air-entrained concrete density at the measured air content. Carefully follow the procedure in the practice for rodding or vibrating specimens. The procedure is not expected to produce optimum consolidation but is used in order to permit reproducibility of results with different technicians. Close any holes left by rodding or vibration after each layer is consolidated by tapping the outside of the mold.

The exposed concrete surface should be finished to produce a flat even surface that is level with the rim or edge of the mold and that has no depressions or projections larger than 1/8 in. (3 mm). It is the author's experience that finishing the exposed surface of a cylinder is one of the most violated requirements. This causes poor capping and dimensional tolerances in cylinders.

Mark the specimens to positively identify them and the concrete they represent. The specimens should be moved, if necessary, to curing storage with a minimum amount of handling and immediately after finishing. Cover the top of the specimens with a sheet of plastic, seal them in a plastic bag, or seal the top of the specimens with a plastic cap. Caps may leave depressions in the concrete surface greater than 1/8 in. (3 mm) in depth making capping for testing difficult. Wet fabric may be used to cover the specimens to help retard evaporation, but the fabric must not be in contact with the surface of the concrete or cardboard molds.

Curing Specimens

This practice requires either standard curing or field curing methods. They are not interchangeable. If the specimens are for checking adequacy of mixture proportions, for strength, acceptance testing, or quality control, standard initial and final curing method must be used. If specimens are for determining removal time of forms or shoring, when a structure may be put into service, comparison with standard curing, or curing or protection requirements, field curing method must be used.

Standard curing is exposure of the specimens to standard conditions of moisture and temperature from the time of fabrication to the time of testing.

Field curing is maintaining the specimens as nearly as possible in the same moisture and temperature conditions as the concrete they represent.

Standard Initial Curing

Control of standard curing conditions is very important since variations can dramatically affect the concrete properties and test results.

The standard initial curing period takes place in a moist environment with the temperature between 60 to 80°F (16 to 27°C) for up to 48 h. When the specified compressive strength is 6000 psi (40 MPa) or greater, the standard initial curing temperature shall be between 68 and 78°F (20 to 26°C). It may be necessary to create an environment during the initial curing period to provide satisfactory moisture and temperature. Insufficient moisture during the initial curing can lower measured strength. One study [21] showed that even at proper temperatures, air curing could lower the strength by 8 % at one day, 11 % at three days, and 18 % at seven days. Early-age results may be lower when stored near 60°F (16°C) and higher when stored near 80°F (27°C). For high strength concrete, heat generated during early ages may raise the temperature above the required storage temperature. Immersion in water saturated with calcium hydroxide may be the easiest method to maintain the required storage temperature.

During the setting and initial hardening, the concrete can be damaged by harsh treatment. For traffic-induced vibrations, Harsh and Darwin [22] reported that wet mixtures exhibited as much as a 5 % loss in strength through segregation as opposed to a 4 % gain in strength in dry mixtures due to improved consolidation.

Field Curing

Field curing procedures are unique to each situation. These specimens will reflect the influence of ambient conditions on the properties of the concrete. Since each condition is different, the method selected for moisture and temperature condition will be different. Even then, the effect of the moisture and temperature would not be the same as on the actual concrete structure. This would be the case in a relatively massive structure. They give little indication of whether a deficiency is due to the quality of the concrete as delivered or improper handling and curing of the specimens. ACI Standard Practice 308 [23] gives procedures for checking the adequacy of curing.

Transporting

Specimens transported to the laboratory for standard final curing before 48 h should remain in the molds, then be demolded and placed in laboratory standard final curing. Specimens shall not be transported until at least 8 h after final set. If the specimens are not transported within 48 h the molds should be removed within 16 to 32 h and standard final curing used until the specimens are transported. Transportation time should not exceed 4 h. Specimens should be transported in such a manner that prevents moisture loss and exposure to freezing temperatures, and protects them from jarring. Richardson [10] indicated that rolling and bumping around in the back of a pickup truck could result in a 7 % loss of strength and dropping cylinders from waist level can lower strength at least 5 %. Cylinders and beams should be cushioned during transport and handled gently at all times. Care should be taken to ensure that the moisture condition of field-cured specimens is maintained throughout the period of transportation.

Standard Final Laboratory Curing

Standard final laboratory curing is at a temperature of 73.0 ± 3.5°F (23.0 ± 2.0°C) and a moist condition with free water maintained on the surface of the specimens. Moist rooms and water tanks are usually used for creating the moist environment. A moist room is a "walk-in" storage facility with controlled temperature and relative humidity, commonly called a fog room when the prescribed relative humidity is achieved by atomization of water. All fog rooms should be equipped with a recording thermometer. Water storage tanks constructed of noncorroding materials are also permitted. Automatic control of water temperature and recording thermometer with its sensing element is required in the storage water. The water should be clean and saturated with calcium hydroxide. Continuous running water or demineralized water may affect results due to excessive leaching of calcium hydroxide from the concrete specimens and should not be used in storage tanks. ASTM Specification for

Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the Testing of Hydraulic Cements and Concretes (C 511) covers the requirements for fog rooms and water storage tanks used for standard curing. Price [6] stated that water-cured specimens with a water-cement ratio of 0.55 were about 10 % stronger at 28 days than those cured in a fog room at 100 % relative humidity.

Making and Curing Test Specimens in the Laboratory (ASTM C 192)

This practice, like ASTM C 31, is also a definitive procedure for performing specific operations that does not produce a test result. The procedures are for a wide variety of purposes, such as: (1) mixture proportioning for project concrete, (2) evaluation of different mixtures and materials, (3) correlation with nondestructive tests, or (4) providing specimens for research purposes. Committee C9 has over 150 standards and C 192 is referenced in over 40 of these standards. Therefore, when using C 192 it necessary to coordinate with any other applicable standard.

Equipment

The equipment needed in the laboratory includes the following: molds; rods or vibrators; mallet; small tools; sampling pan, mixing pan, or concrete mixer; air-content apparatus; slump cone; thermometer; and scales. Hand mixing is permitted but not for air-entrained or no-slump concrete. Machine mixing is preferred, especially when a laboratory regularly mixes concrete. Scales for weighing batches of materials and concrete should be checked for accuracy prior to use and be within acceptable tolerances. A sampling pan is required to receive the entire batch discharged from the concrete mixer. Conformance of the molds to the applicable requirements should be verified prior to mixing the concrete. Different tests may require different molds. The dimensions of the molds also vary for different tests and usually according to aggregate size. Some test methods require specimens that are other than cylindrical or prismatic in shape. They should be molded following the general procedures in this practice. A program to determine the number of batches of concrete, number of specimens for all the various tests, and various test ages needs to be performed prior to laboratory mixing of the concrete.

Materials Conditioning and Testing

Before mixing the concrete, all the materials must be at room temperature in the range of 68 to 86°F (20 to 30°C) unless the design is being performed at other than room temperature. Store the cement as required and check it for fineness. If required, determine the relative density and absorption of the coarse aggregate using ASTM Test Method for Density, Relative Density (Specific Gravity), and Absorption of Coarse Aggregate (C 127) and fine aggregate using ASTM Test Method for Density, Relative Density (Specific Gravity), and Absorption of Fine Aggregate (C 128). The moisture content of the aggregates must be known before batching the concrete. The weights of the cement, aggregates, admixtures, and water must be known accurately prior to batching. Proportioning concrete mixtures are covered in ACI Standard Practices 211.1 [24] and 211.2 [25].

Mixing and Testing

Mix the concrete in a mixer that will provide a uniform, homogeneous mixture in the mixing times required. The size of the batch has to be about 10 % in excess of the quantity required for molding the specimens. Machine-mixed concrete should be mixed for 3 min after all the ingredients are in the mixer followed by a 3-min rest and 2 min of final mixing. To eliminate segregation, deposit concrete onto a clean damp mixing pan and remix by shovel or trowel. Perform the slump and temperature tests in accordance with ASTM C 143 and C 1064, respectively. When required, perform the air-content test in accordance with ASTM C 231 or C 173 and the yield test in accordance with ASTM Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete (C 138). Discard concrete used for determination of the air-content test. Make the specimens following the procedures in ASTM C 192, taking the precautions as previously discussed in the section on ASTM C 31. Care should be taken that specimens are cast and stored in accordance with the applicable test methods.

Consolidation

The specimens are consolidated by rodding or internal and external vibration. The selection of the method is similar to ASTM C 31 using the slump of the concrete as guidance for the method to be used. However, a particular method of consolidation may be required by the test method or specification for which the specimens are being made. When vibration is permitted or required, either internal or external vibration may be used. When using external vibration, care has to be taken that the mold is rigidly attached to the vibrating unit. Concrete with low water contents such as roller-compacted concrete are covered in C 1176 and C 1435. Specimens consolidated with low water contents may require a surcharge weight on the specimen as they are consolidated with external vibration. Additional information on consolidating low-water-content specimens is in Chapter 51 of this volume.

Finishing and Curing

Finish the specimens as required. Cover and cure the specimens for 16 to 32 h prior to the removal of the molds. Concrete with prolonged setting time may require the molds not be removed until at least 16 to 24 h after final setting time. Immediately subject the specimens to standard curing conditions after removal of the molds in moist room or water tanks to meet the requirements of ASTM C 511. Flexural test specimens must be stored in water saturated with calcium hydroxide at standard curing temperature for a minimum period of 20 h prior to testing.

Evaluation

A precision statement of all the test methods included in the standard is based on data from the concrete proficiency sample program of the Cement and Concrete Reference Laboratory. Each laboratory should, as part of its quality system, analyze their data against the values in this practice.

Conclusions

The making and curing of concrete test specimens are covered by two ASTM standards: ASTM C 31 for field use, and ASTM C 192 for laboratory use. The field standard is used to make specimens to comply with specification requirements for concrete used on construction projects. The failure to meet specification requirements has resulted in many investigations to determine the adequacy of in-place concrete, as-delivered concrete, and the making, curing, and testing of concrete specimens. The laboratory standard is used to develop mixture proportions for field concrete and research studies. The failure of the laboratory-developed mixtures to perform in the field has caused considerable problems. Research studies on test specimens made in the laboratory have been questioned on many occasions when one researcher's results are different from those of another researcher on the same type of study.

Concrete test specimens should be made, cured, and tested to obtain accurate and representative results. ASTM C 31 or C 192 and the standards they reference should be followed.

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11

Time of Setting

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Preface

SCRIPTURE PREPARED THE ORIGINAL VERSION OF this document, *ASTM STP 169*, in 1956 [1]. Kelly extended this work with *ASTM STP 169A* in 1966 [2], which was subsequently updated by Sprouse and Peppler in 1978 with *ASTM 169B* [3]. In 1994, Dodson prepared the most recent update, that being *ASTM STP 169C* [4]. In the preparation of this chapter, the contents of the previous editions were drawn upon, but every attempt was made to minimize duplication of information from the previous edition. The author acknowledges the contributors of the previous editions and their summaries of the literature on this topic. The current edition will review and update the topics as addressed by the previous authors, provide up-to-date references and focus specifically on new technologies that have been developed. The review period has been limited primarily to contributions made during the last decade. Attempts have been made to uncover all relevant research, but undoubtedly important advances were not covered or were unknowingly omitted in this review.

Introduction

Hydraulic cementitious compositions are unique in that in their early stages they are moldable and castable materials that subsequently are transformed into rigid structures with useful engineering properties. This structural transformation occurs as the result of chemical reactions between the cementitious material and water, a process referred to as hydration. The hydration process results in the consumption of free water and formation of an interlocking network of hydrating particles, giving rise to stiffening, and subsequently a hardening behavior, which is defined as "setting." With portland cement-based compositions, this process normally occurs in a matter of hours from the time of contact of cement and water. Because the hydration process is complex and consists of a series of chemical reactions, the process is affected by changes in the content, the chemistry and/or the surface area of the cementitious component, temperature, water content, and the presence of chemical and mineral admixtures in concrete mixtures. These changes ultimately affect the time at which finishing and curing operations can be initiated, steam-curing can be applied, cold joints can be avoided, and load can be applied to the structure. Therefore, it is important to have methods by which the rate of setting of the cementitious composition can be quantified.

History

A number of methods for monitoring the time of setting of concrete have been investigated over the years. Some of the earliest methods were developed in the late 50s and are discussed in detail in the previous edition of this publication [4]. The intent of this document is to review the current ASTM method, noting the advantages and disadvantages of the method, as well as to highlight some of the other methods that have emerged or are in the process of being developed.

Current ASTM Method

Basics of the Method

The most widely used method for determining the time of setting of concrete is ASTM C 403/C 403M–Standard Test Method for Time of Setting of Concrete by Penetration Resistance [5]. As clearly stated in the title of the method, the time of setting is determined from changes in the penetration resistance of a specimen as a function of time. In particular, a sample of mortar is obtained by wet sieving a portion of the concrete on a 4.75 mm sieve to remove the coarse aggregate fraction or it can be a prepared mortar. The mortar fraction is placed into a rigid, non-absorptive container and stored at a specified temperature. Periodically, the resistance to penetration of the mortar fraction is obtained by using a series of pins of successively decreasing diameter injected to a depth of 25 mm (1 in.). Bleed water is removed from the surface of the mortar fraction on a regular basis. Pin penetrations are spaced in such a way as not to be influenced by previous penetration locations and at a minimum distance from the perimeter of the container. Resistance to penetration is determined by dividing the applied load by the surface area of the respective pin. These resistance values are then plotted as a function of hydration time and fitted by regression analysis to determine the times of initial and final setting.

The resistances to penetration corresponding to the times of initial and final setting are arbitrarily defined as 3.5 MPa (500 psi) and 27.6 MPa (4000 psi), respectively. The time of initial setting is defined to be the time at which the concrete is no longer mobile, even by the application of vibration. Initial set is often the time at which steam-curing is applied in precast operations, as well as generally regarded as the time at which finishing operations can begin. There is not complete agreement on these definitions, though. The British Standards Institute BS

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5075 defines the limit for placing and compaction at 0.5 MPa (72 psi) [4]. Abel and Hover observed that the time to begin finishing operations, which is observed in practice as the time at which the boot of an adult male leaves an imprint approximately 6 mm deep in a fresh concrete surface, occurs at a penetration resistance of approximately 0.1 MPa (15 psi) [6]. Additionally, finishing operations on concrete slabs performed using a finishing machine are understood to begin as soon as measurable values of penetration resistance are obtained on companion mortar specimens [7]. Therefore, evidence currently exists to suggest that finishing operations generally begin much sooner than the time corresponding to initial setting as determined by ASTM C 403/C 403M.

The time of final setting is generally regarded as the time at which the concrete is no longer deformable. This signals the end of the finishing window, at which time the surface of the setting concrete can no longer be manipulated and also corresponds to the time at which curing operations can begin, such as the application of a curing compounds, water, wet burlap, or the like. Measurements of the compressive strength of the concrete specimens can be made at this time and have been reported to be on the order of 0.7 MPa (100 psi) [4].

The equipment typically used in the lab to determine the times of setting is a bench-top penetrometer like that shown in Fig. 1. It consists of a base platen with a vertical post, to which is attached a spring mechanism with a meter to display the applied force. The meter contains a floating needle, which acts as a marker of the maximum force delivered during the penetration step. A lever attached to the side of the spring mechanism is used to slowly inject the appropriate-sized pin into the plastic mortar sample until the maximum penetration distance is achieved. These instruments are fairly rugged, easy to use, and, if regularly calibrated, produce very reproducible results.

A smaller version that is often used in the field is commonly referred to as a pocket penetrometer. This penetrometer, shown in Fig. 2, is a small device that consists of a single pin with a spring-loaded head assembly and a sliding collar. Markings corresponding to different levels of penetration resistance (not force, since the pin size is fixed) are inscribed on the side of the device. While holding the device by the spring-loaded head and manually inserting into the plastic mortar specimen, the head travels downward. When the maximum penetration distance is achieved, the device is extracted from the mortar specimen and penetration resistance determined from the location of the sliding collar. As one might expect, the device is much more operator dependent than the bench-top unit, particularly due to the difficulty in maintaining the penetrometer in an orientation perpendicular to the surface of the specimen during penetration, and as such produces results with a wider variability. This variability is further expanded because only one diameter pin is typically used, thereby limiting the number of useful penetration events that can be executed to get a good statistical sampling. Finally, since the application of sufficient force to achieve penetration at the later stages of the hardening process is very difficult, the device only provides information on time of initial setting and not final setting. Nevertheless, this device does have the advantages of being easily transportable and can provide a quantifiable estimate of the rate of hardening under many conditions.

Fig. 1—One style of bench-top penetrometer used to perform the ASTM C 403/C 403M method.

Fig. 2—Pocket penetrometer used to perform the ASTM C 403/C 403M method when a bench-top style is not available.

Manipulation of the Data

An example set of data that was obtained from a bench-top penetrometer is shown in Table 1. At the time of the development of the method, these data were plotted manually on semi-logarithmic graph paper and the times of setting determined by hand-fitting. With the availability of personal computers and spreadsheet software, it is now a relatively easy task to plot and analytically determine the best function to fit the data. Figure 3 shows the penetration resistance versus elapsed time for the specimen in Table 1 (control), as well as two additional samples. Equations are shown for fits made using either an exponential or a power law function. Fitting a set of at least six data with one of these two functions and removing any outliers generally results in a correlation coefficient that is greater than

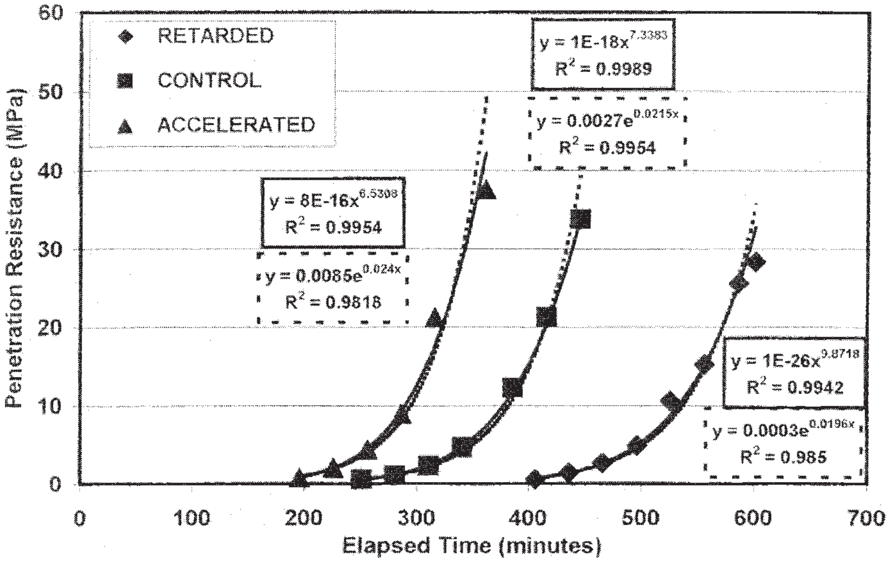

Fig. 3—Penetration resistance versus elapsed time for mortar fractions from three different concrete mixtures. Dotted lines and boxes indicate exponential function fits to the data; solid lines and boxes represent power law fits to the data.

0.98. Once an adequate equation is determined, the times of initial and final setting can be determined by inputting either 3.5 MPa (initial) or 27.6 MPa (final) for the penetration resistance and calculating the corresponding elapsed time. Table 2 shows the results of the calculation on this dataset.

The precision and bias statistics of this method are shown in Table 3. These values were obtained using the inch-pound system and hand-fitting procedures in 1973 and have remained unchanged since that time. The data were collected during a round-robin test program in which five different laboratories participated. Evaluations were performed on prepared mortars for ease of preparation, in place of mortar wet-sieved from concrete as specified in the method.

Advantages of the Method

One of the main advantages of this method for determining the time of setting is that it is based on multiple quantitative measurements on the same specimen. The use of multiple data points improves the precision of the time of setting, as determined by this method. In addition, because the penetrations are of various diameters, which are significantly larger than the largest particle in the mortar specimen, the influence of edge effects is minimized. These characteristics are in contrast to ASTM C 191 or ASTM C 266 methods used to quantify the time of setting of cement pastes. For ASTM C 191, a Vicat needle of one dimension is used throughout the test, while for ASTM C 266, one pin size is used for determination of initial set and another size for final set [8,9]. Subsequently, ASTM C 403/C 403M is used in such specifications as ASTM C 494/C 494M to evaluate the effect of chemical admixtures on the time of setting [10]. Another advantage is the fundamentally similar mechanism of penetration to that used in the field by finishers to determine when to begin finishing operations. The method is very straightforward to perform, utilizes relatively inexpensive equipment ($1–2 K), and is quickly understood by a technician.

Disadvantages of the Method

The first disadvantage of the method is the requirement of wet-sieving the plastic concrete to obtain the mortar fraction. This process can be laborious, especially when the slump of the concrete is low and/or the water-to-cement ratio (w/c) of the mixture is low. In the laboratory, where dozens of concrete mixtures may be evaluated in a day, this can represent a significant investment in time and effort to perform the method. In the field, wet sieving requires transporting a large sieve and capturing vessel to be able to perform the method.

TABLE 1—Penetration Resistance Data for the Control Mixture

Elapsed Time (min)	Penetration Force (N)	Pin diameter (mm)	Surface Area of Pin (mm^2)	Penetration Resistance (MPa)
251	176	20.3	323	0.54
281	176	14.3	161	1.1
311	370	14.3	161	2.3
341	299	9.1	65	4.6
386	396	6.4	32	12.4
416	343	4.5	16	21.4
446	546	4.5	16	34.1

TABLE 2—Calculation of Time of Setting for the Three Specimens shown in Fig. 3

Fitting Function	Time of Setting (Initial or Final)	Accelerated (minutes)	Control (minutes)	Retarded (minutes)
Exponential	Initial	251	333	478
	Final	336	430	583
Power Law	Initial	248	337	489
	Final	340	446	602

A second disadvantage is the requirement to continuously monitor the specimen. Under normal conditions, the specimens can remain unattended for the first few hours after contact of cement and water. Thereafter, though, the specimens must be continuously monitored and tested at successively decreasing time intervals until the time of final setting has been exceeded. Automated systems, such as the robotic system shown in Fig. 4, exist and can perform the method on multiple specimens without human intervention. Unfortunately, this equipment requires a significant capital investment (~$80 K as of 1999) and is impractical for many users.

Another disadvantage that has been already mentioned briefly is the lack of correlation of the results from the method to observations with field concrete. Finishing operations are generally initiated earlier than the time of initial setting as indicated by the method. This issue, though, appears to be a matter of definition, and not a fundamental flaw in the method. Hence, useful information can still be obtained by comparing the relative setting characteristics of different concrete mixtures by penetration resistance.

Modifications to the Method

The basic aspects of the current method appear to have remained essentially unchanged for more than three decades, which is indicative of the usefulness of the method. Nevertheless, there is always room for improvement. One observation that many practitioners have commented upon in recent years is a belief that the operator has the ability to skew the values of initial and final time of setting by varying the location at which the pin is penetrated into the specimen. While there is general agreement among the C09.23.1 committee members that this is indeed true, no data could be found to substantiate the hypothesis. Therefore, the following test was recently performed in the laboratory to investigate this phenomenon.

One large batch of mortar was prepared and transferred into ten identical cylindrical containers (approximately 18 cm in diameter). As the hydration proceeded and the specimens began to stiffen, penetration measurements were made at ten different elapsed times between the time of mixing and the time of final setting. At each time interval, punches were made at five equally-spaced locations along the diameter of the same container, and the sample was then discarded (all punches were made with the same operator on the same penetrometer using the same fitting equation). As a result, the time of initial and final setting could be determined as a function of position in the specimen. Using a fit to all ten data points for each position, a plot was prepared and is shown in Fig. 5. The trend is towards a shorter time of setting at the center of the container as compared to the perimeter, as expected, but the magnitude of the difference depends upon the stage of hydration. The differences in the time of initial setting are small with position, while the maximum difference in the time of final setting is on the order of 40 min. This is significant for specifications such as that of an ASTM C 494/C 494M Type F water reducer, where the maximum allowable delay in time of setting of the admix-

TABLE 3—Precision of ASTM C 403/C 403M

Time of Setting	Coefficient of Variation, % Single Operator, Multiday, Three Individual Results	Coefficient of Variation, % Multilaboratory Average of Three Tests
Initial	7.1	5.2
Final	4.7	4.5

Fig. 4—Automatic penetrometer with ability to monitor eight specimens simultaneously.

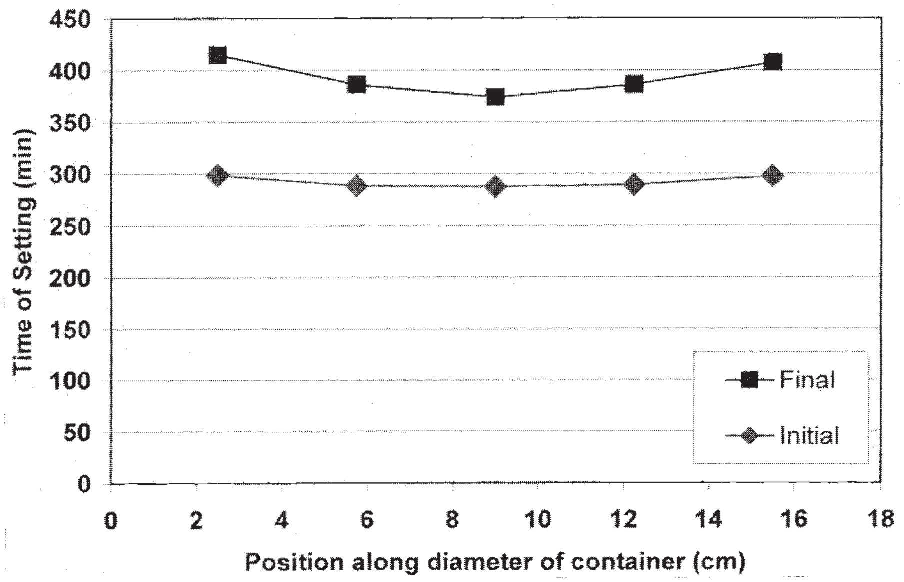

Fig. 5—Time of setting of mortar specimens as a function of the punch location on cylindrical specimens.

ture-treated concrete mixture is 90 min from the control. The thermal loss from the perimeter of the container is likely resulting in less activation of the hydration process than in the center of the specimen. This gradient from inside to the outside of the container is more significant as the hydration process proceeds, further substantiating the observation of wider variation at the time nearer to final setting.

As a result of these observations, additional tests are planned to investigate the extent of this variation in time of setting as a function of different cements and chemical admixture compositions. In parallel, efforts are underway to modify the method. The current proposal is to limit the locations for penetration measurements to a ring of a fixed distance from the outer perimeter of the container.

Additional Methods for Evaluating Time of Setting

Other Penetration Methods

As mentioned previously, one of the limitations of the ASTM C 403/C 403M method is that the mortar must be wet sieved from the concrete. To eliminate this step, Abel and Hover proposed the use of a modified penetrometer with a much larger surface area than the pins typically used in the current method [6]. The surface area is similar to that of the base of an adult work boot and the penetrometer foot can be used directly on the surface of a concrete slab. This apparatus is referred to as the "Finisher's Foot" and is shown in Fig. 6. Two different feet are used, the smallest of which is still several times larger than the diameter of

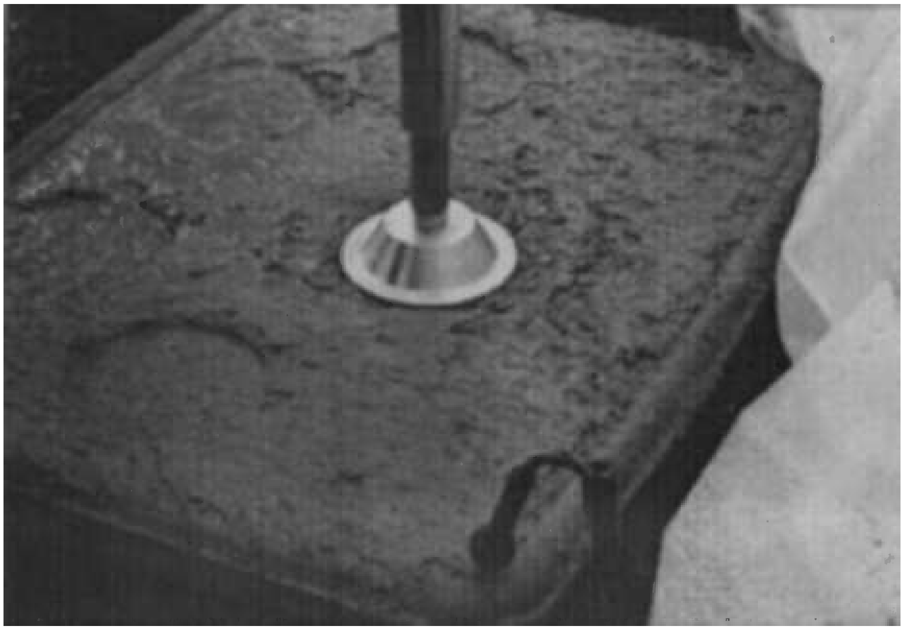

Fig. 6—Finisher's foot apparatus being using on a concrete slab to determine penetration resistance (after Abel and Hover [6]).

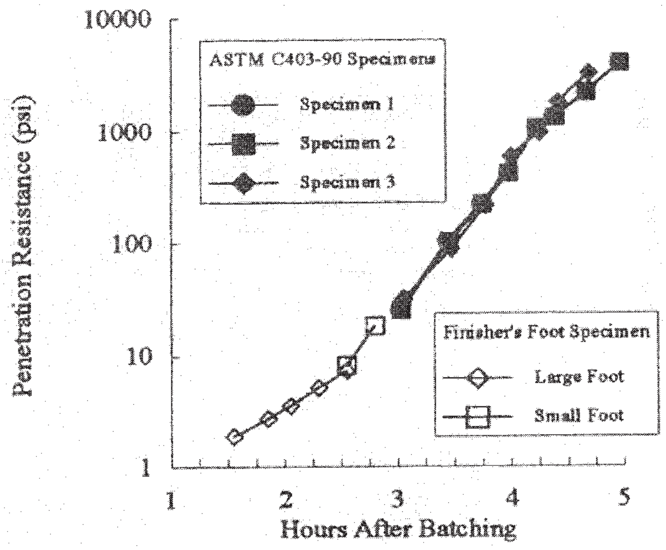

Fig. 7—Penetration resistance versus time after batching for field concrete. Open symbols are generated using the "finisher's foot"; closed symbols are from C 403 measurements on mortar wet sieved from the same concrete (after Abel and Hover [6]).

a typical coarse aggregate. The penetration depth in this method is modified and limited to 6 mm. This depth is chosen to simulate the depth at which the work boot of a finisher will sink into a bull-floated slab when beginning subsequent finishing operations. The abbreviated depths may possibly result in less edge effect from interaction with the coarse aggregate.

In essentially the same manner as the ASTM C 403/C 403M method, the penetrometer foot is pressed into the plastic concrete to the prescribed depth, and the force necessary to achieve the penetration is recorded and converted to penetration resistance. An example plot of penetration resistance versus batch time is shown in Fig. 7. The open symbols on the lower left portion of the plot are obtained on the concrete slab, while the closed symbols in the upper right portion of the plot are obtained on companion mortar specimens sieved from the same concrete mixture. A couple of significant features are apparent from this plot. First is the observation that the penetration measurements can be obtained much sooner on the concrete slab than on the mortar. Second is the smooth transition during the change from the large foot to the small foot, as well as during the transition from measurements on the slab to the mortar specimen. Probably of most significance, which is not apparent from the plot alone, is the authors' observation that the time to begin finishing operations occurred at penetration resistances on the order of 0.1 to 0.2 MPa, which is prior to the first penetrations on the mortar specimens. While some of these observations are specific to the reported projects and some of the effects are likely due to the differences in sizes of the specimens, the time of "initial" setting by this method is much earlier than predicted by ASTM C 403/C 403M. Interest in standardizing this procedure is significant and is currently going through the balloting process as an ASTM Standard Test Practice.

Another limitation of ASTM C 403/C 403M that has been mentioned is the need to continuously monitor the specimen during the hardening process. While this can be addressed with a sophisticated and expensive robotic system, this requirement can also potentially be overcome by using a continuous penetration measurement. Sohn and Johnson discussed an apparatus that could be used to continuously monitor the hardening process of cement-based materials during microwave curing [11]. Figure 8 shows the details of the arrangement. They investigated multiple penetration rates and interpreted the data in terms of hardening rate at various temperatures and hardness values. Results were limited to investigations on cement pastes and mortars.

Further evaluations of this method were carried out in the laboratory on prepared mortars by modifying the orientation of the apparatus and comparing the results to those obtained by ASTM C 403/C 403M [12]. Different penetration rates, as well as specimens with a range of setting times, were investigated. An example comparison of the results of the two methods is shown in Fig. 9. The correlation for the mixtures investigated at this penetration rate was quite good; however, further work was considered necessary to fully investigate the method. Among the limitations of the method, the mortar fraction still needs to be sieved from the concrete mixture. While in principle a similar system can be designed with a large penetrometer for use on concrete, economically impractical loads

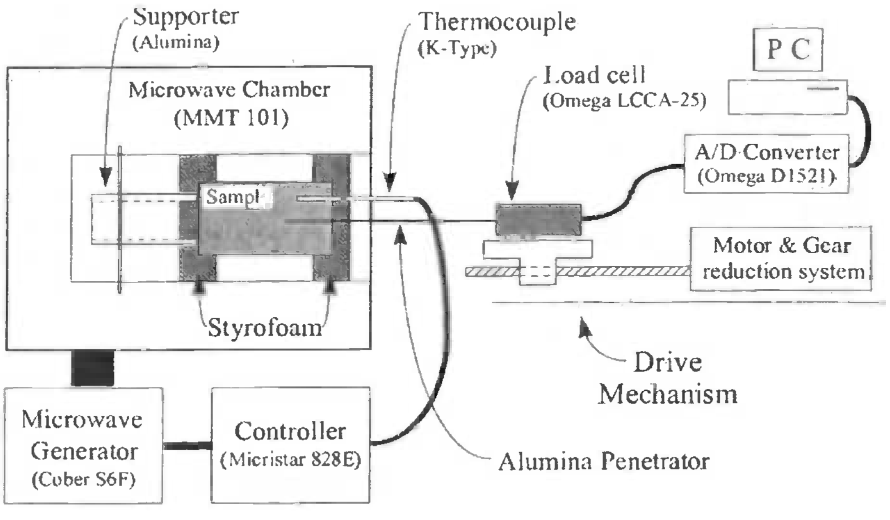

Fig. 8—Continuous penetration apparatus discussed by Sohn and Johnson [11]. Reprinted with permission.

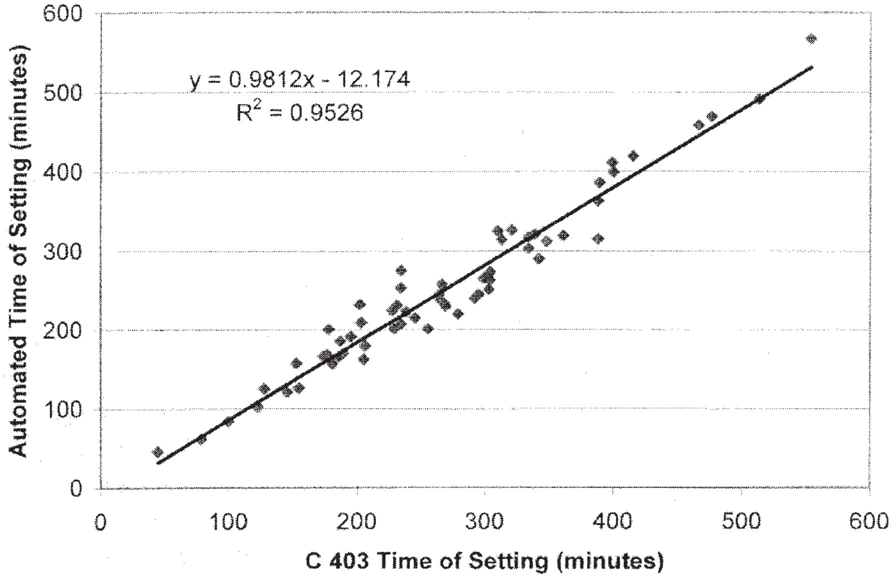

Fig. 9—Comparison of initial and final times of setting determined by the continuous penetration versus that obtained by C 403 [12]. A penetration rate of 6.4 mm/hr was used on prepared mortar specimens.

would be required. Another limitation of the method is that the apparatus must be dedicated to one specimen throughout the hardening process, so multiple pieces of equipment are necessary for use in a development laboratory.

Thermal Methods

Because the hydration reaction of portland cement is exothermic, researchers and practitioners have used this attribute as a means of assessing the state of the hardening process [13–17]. Isothermal calorimetry is one technique that is very useful for quantifying the energy liberated in this process, as well as a very good tool for elucidating the reactions of the calcium sulfate and other mineral phases. An example plot of energy liberated versus hydration time is shown in Fig. 10. In general, the time corresponding to the onset of the rise in the curve after the induction period is considered to correspond closely with the time of initial setting, but many factors in the field can alter that correlation. Equipment is now available commercially that allows testing of multiple specimens at the same time, but the cost is significant (~$20 K). This equipment is an excellent tool for laboratory investigations and for pre-qualification of field mixtures, but is not transportable to the field. Another limitation of this method is that the cell size is small so that only pastes and mortars can be tested.

One of the simplest approaches is merely to embed a thermocouple wire into the center of the concrete specimen and monitor the temperature as a function of time. This condition is often semi-adiabatic and the results are heavily dependent upon the size of the specimen, environmental conditions, and mixture design. Nearly adiabatic conditions can be achieved by placing the specimen in an insulating chamber, which is useful in some applications, but may not translate well to the setting characteristics of a slab or other structure that is not fully insulated. Nevertheless, portable equipment with the ability to

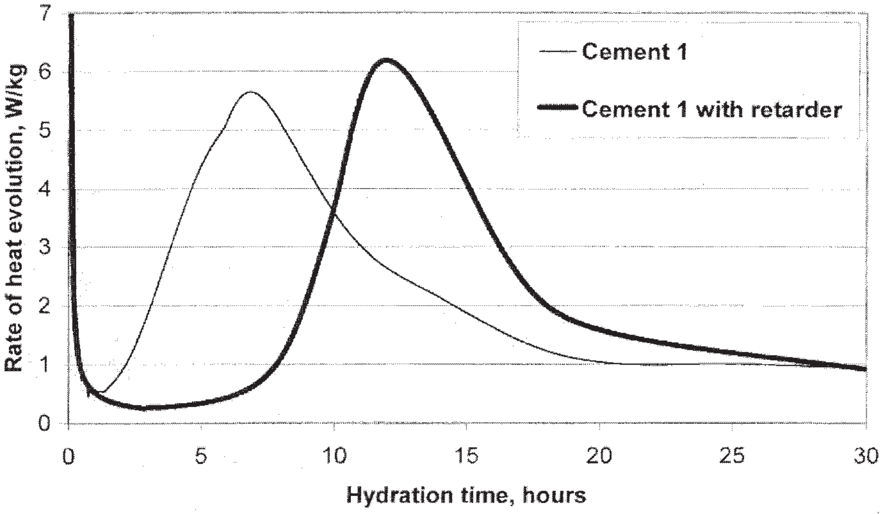

Fig. 10—Typical isothermal calorimetry curve for hydrating cement pastes (20 °C).

Fig. 11—Insulating chamber with cavities for concrete specimens used in proposed thermal method. Thermocouple tips are imbedded in the base of the cavity.

read multiple thermocouples and that are relatively inexpensive and convenient to use are now available. As such, investigations have been performed recently to further develop the appropriate sample storage conditions, with the intent of improving the correlation to ASTM C 403/C 403M [14].

This author has proposed a sample configuration and data manipulation technique for temperature versus time measurements that provides good correlation with ASTM C 403/C 403M [14]. The method is intended for use in the laboratory and allows the use of a concrete sample, instead of mortar sieved from plastic concrete. An example of the sample configuration is shown in Fig. 11. The concrete mixture is placed into 15 cm × 15 cm plastic cylinders, which are subsequently placed into insulating cavities, but no insulation is placed on the top surface of the specimens. Thermocouples are attached permanently to the interior base of the cavities, thereby eliminating the need of embedding and removing the thermocouple wires every time a specimen is tested. An example plot of temperature versus time for a specimen tested in this configuration is shown in Fig. 12. Determination of the first and second derivatives of temperature versus time, and plotting the data versus time, results in the curves shown in Figs. 13 and 14. The time corresponding to the maximum in these plots is then compared to the times of initial and final setting determined on companion mortar specimens (wet sieved from the same concrete) using ASTM C 403/C 403M. These comparisons for three different concrete mixtures are

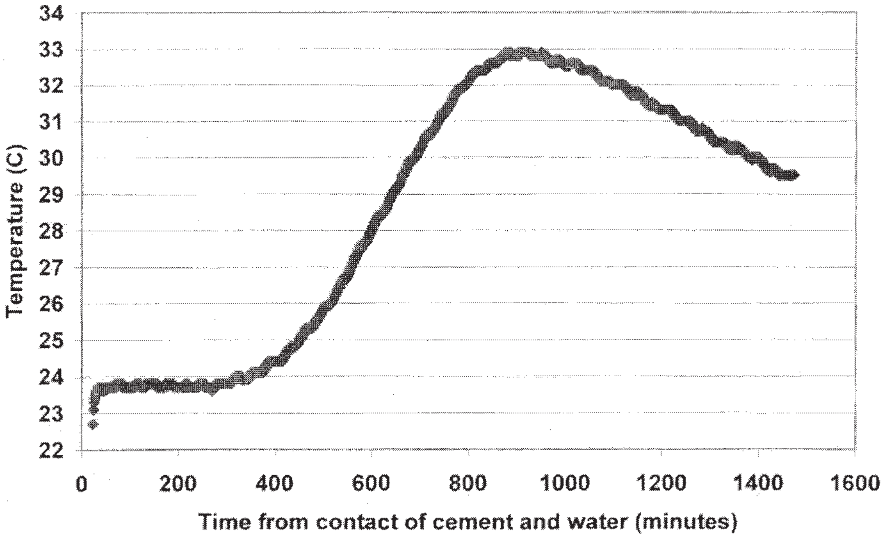

Fig. 12—Example of the temperature versus time plot obtained for a specimen placed in one of the cavities shown in Fig. 11.

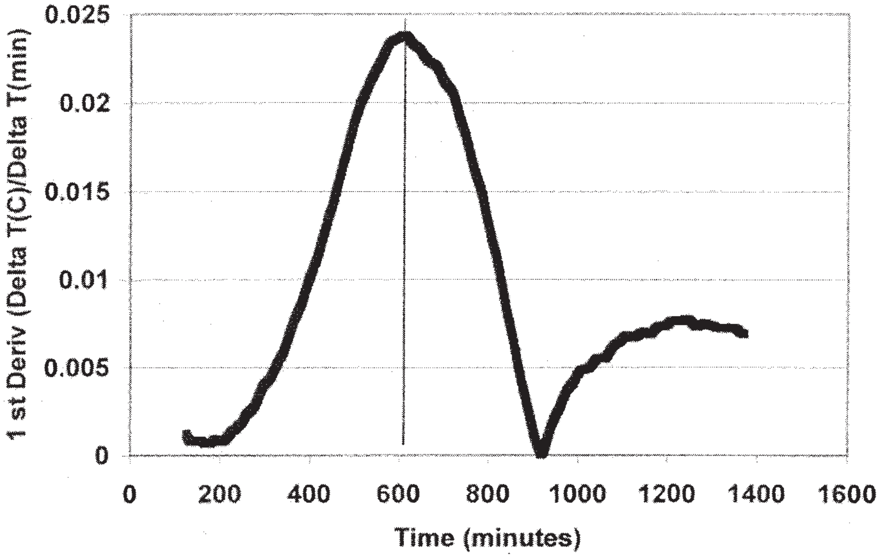

Fig. 13—Plot of the first derivative of temperature with time (from Fig. 11) as a function of time. Time of the maximum appears to correlate with time of final setting determined from C 403.

shown in Fig. 15. Interestingly, the correlation between the time of final setting and the time of the maximum in the first derivative versus time is reasonable. The time of the maximum in the second derivative versus time occurs earlier than the time of initial setting by ASTM C 403/C 403M and seems to correlate better with the time at which the penetration resistance on the mortar specimen is approximately 1.25 MPa (200 psi). Further work on a range of mixture designs is necessary to further validate the method, but initial observations are very promising. Nevertheless, because this method addresses many of the operational deficiencies of ASTM C 403/C 403M (wet sieving and constant human monitoring), the consensus of the C09.23.1 committee has been to move forward with standardization of the method.

Ultrasonic Methods

A number of investigations have been published on the use of ultrasonic waves to follow the setting of concrete [17–27]. Some techniques rely upon the propagation of the waves through the material, hence the specimen must be thin, due to its lossy nature. Others utilize reflection of the wave off of the near surface, where the response is independent of the thickness of the specimen. Typically, either shear waves or compression waves are used, though shear waves are only supported in solids, so assessing the extent of shear wave absorption or reflection can be used as a basis for investigating the transition from a fluid to a solid. Subramaniam et al. have characterized this relationship as a wave reflection factor

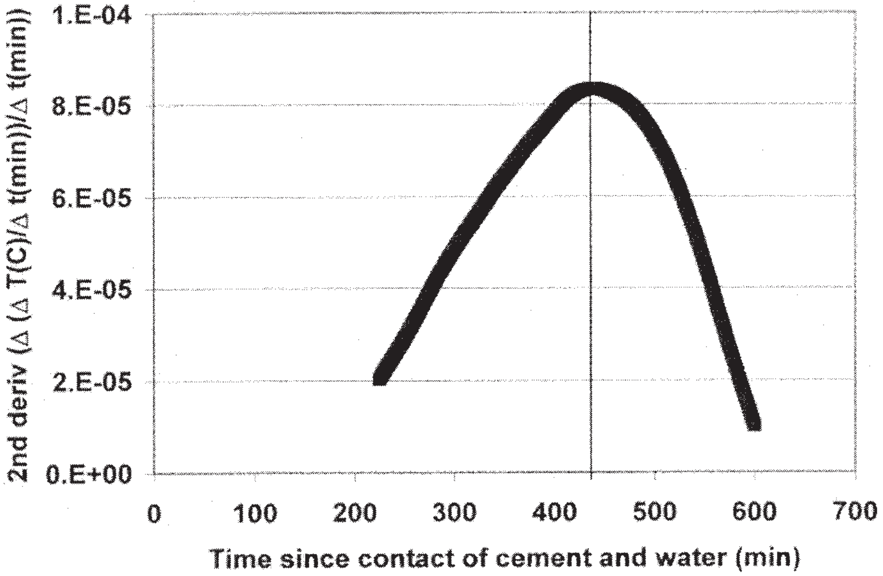

Fig. 14—Plot of the second derivative of temperature with time (from Fig. 11) as a function of time. Time of the maximum appears to occur slightly before the time of initial setting determined from C 403.

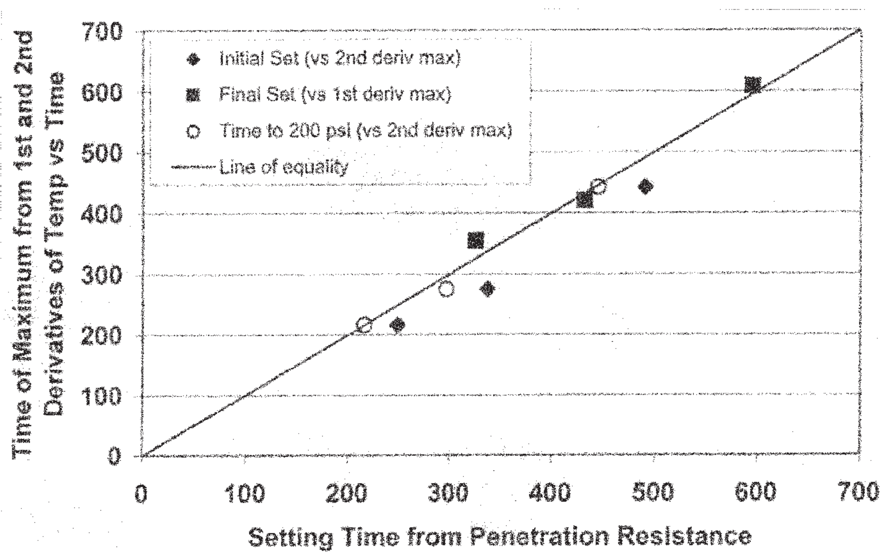

Fig. 15—Comparison of the time at the maximum in the 1st or 2nd derivative of time plot versus time of setting from ASTM C 403/C 403M. Data from three different mixtures with a range of setting times are presented, one of which is based on information in Fig. 12.

(WRF), which changes with hydration time as shown in Fig. 16 [20]. They have observed that the time corresponding to the decrease in the WRF exhibits a linear relationship with the time of initial setting determined by ASTM C 403/C 403M [19].

One of the obvious advantages is that this technique is nondestructive, thus is suitable for real concrete structures. WRF can also be used at times beyond the setting regime to characterize compressive strength development, and hence can be used to characterize the concrete mixture throughout the entire hydration process. Continuous data acquisition also allows for automation, so a large portion of the manual effort can be removed. A disadvantage of the reflection method is that a transducer is required for each sample tested and the setup for multiple specimens is somewhat costly (~$10 K for an 8 channel system). Nevertheless, the method appears to be quite promising and further development in this area could be quite fruitful.

Electrical Methods

Investigations concerning changes of the electrical characteristics of hydrating systems have continued since the last edition of this chapter [28–32]. Many of the recent investigations utilize impedance spectroscopy (IS) as the technique of choice. IS allows easy separation of electrode effects, sweeps a range of frequencies to find the true bulk resistance (or inversely the conductance), and provides a means to determine the dielectric properties of the material with time. An example plot of bulk resistance versus hydration time is shown in Fig. 17. A common feature is the inflection point, where the resistance begins to increase significantly with time, concurrent with the end of the dormant period and renewed hydration reactions. Attempts by this author to correlate the time of the inflection point from mortars with the time of initial or final setting from ASTM C 403/C 403M have not shown a clear relationship. An

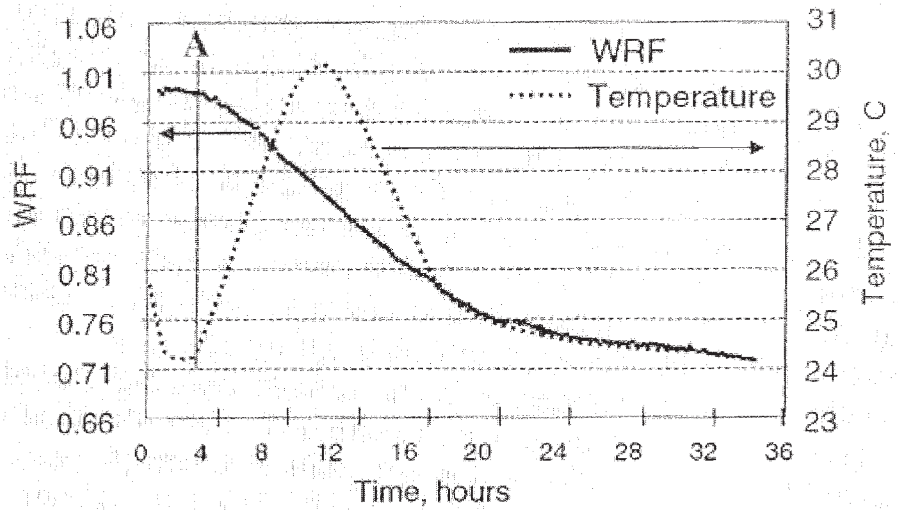

Fig. 16—Wave reflection factor (WRF) versus hydration time for a concrete specimen. Point "A" is where the WRF begins decreasing and corresponds closely with time of initial setting (after Subramaniam et al. [20]). Reprinted with permission.

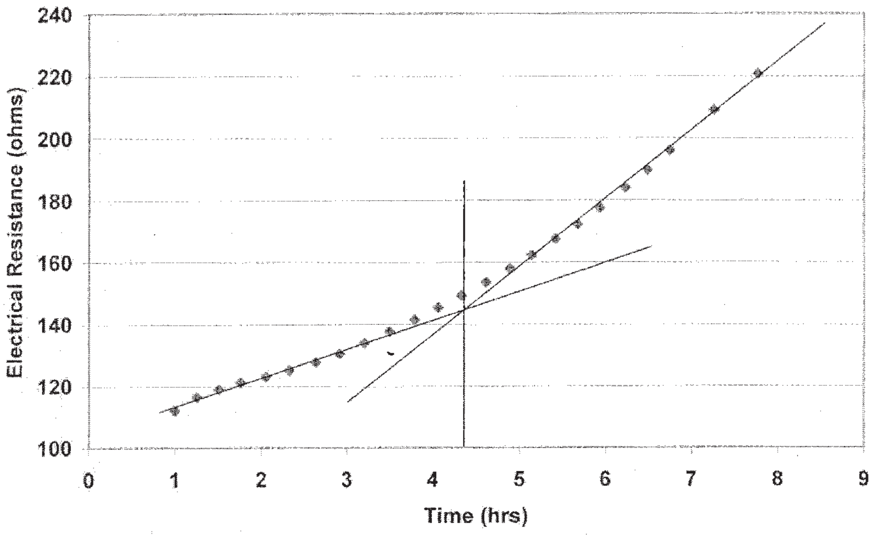

Fig. 17—Example of changes in bulk resistance of a mortar prism as a function of hydration time. Vertical line indicates time corresponding to the inflection point in the curve.

example of the trends is shown in Fig. 18. The specimens, which were retarded, exhibited significant bleed water, which may have adversely affected the test results. Torrents et al. investigated cement pastes with electrical means and the Vicat needle [31]. Reasonable correlation was found between the region defined as "beginning," but poor correlation for the region referred to as the "end."

A disadvantage of the equipment for making IS measurements is equipment cost (~$15 K), which could limit its widespread use. Furthermore, the resistance of the specimen has shown to be dependent upon the resistance of the pore solution, as well as the connectivity of the pore network, both of which can change independently [28,29]. Therefore, this method may not be sufficiently sensitive for some applications.

Rheological Methods

Struble and coworkers appear to be the primary group investigating the relationships between rheological changes and the setting characteristics of portland cement systems during hydration [33–35]. With the use of a constant stress rheometer in a regime below the yield stress, they observed two regions of yield stress in cement pastes. The first corre-

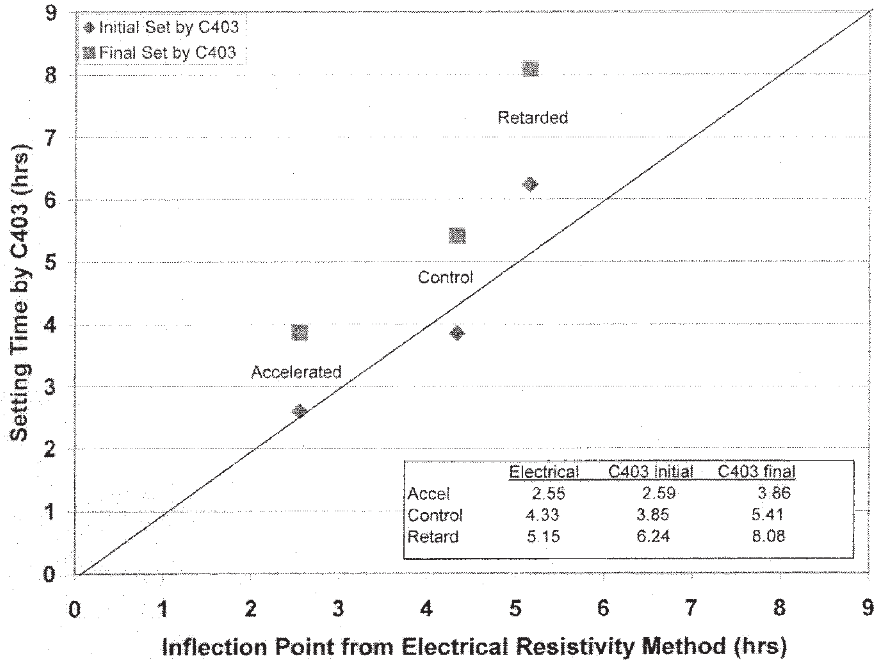

Fig. 18—Comparison of time of setting determined by ASTM C 403/C 403M versus the time of the inflection point in the electrical resistance versus time plot.

lated with the induction period and the other with the acceleratory period, with the time of the transition corresponding to the time of initial setting by the Vicat needle [33]. In another study, they developed a dynamic rheology test and compared the oscillatory shear behavior to the features in ASTM C 403/C 403M and C 191 [34]. Good agreement was observed up to the torque limit of the rheometer, but they were unable to follow the rheological behavior to the time of initial setting as defined by either of the ASTM methods. These results suggest that a higher torque apparatus would be necessary to make comparisons to ASTM C 403/C 403M. In addition, measurements must still be performed on cement paste instead of concrete and delicate equipment is required. Bunt discussed methods for characterizing the rheology of calcium aluminate pastes, in addition to thermal and penetration methods, but comparisons between the methods were not discussed [36].

Other Methods

Methods such as X-ray diffraction, NMR, and computer modeling are also discussed in the literature as a means of assessing the setting characteristics of hydrating systems [37–40]. X-ray diffraction allows characterization of the gypsum/hemihydrate reactions and the formation of portlandite. NMR can be used to determine the physical state of the water, thereby providing some indication of the extent of hydration. Computer modeling allows the possibility to simulate the extent of hydration at which the solid particles are percolated and form a three-dimensional matrix, thereby resulting in initial setting. These approaches are tools of most interest to the researcher, and at present are focused on mechanistic studies rather than routine measurements of time of setting.

Summary

Variables such as temperature, w/c, type of cement, fineness, presence of chemical or mineral admixtures, and cement content all affect the rate of setting of concrete and have been reported on previously [4]. Quantifying the setting characteristics of hydraulic mixtures is therefore an important part of determining when finishing operations can begin, steam can be applied to precast elements, and/or load can be applied to structures. The current ASTM C 403/C 403M method is widely used as a means of assessing the setting characteristics of concrete mixtures, both in the laboratory and in the field. This method has proven to be robust, having remained relatively unchanged for more than three decades. Ease of use, relatively low cost, and suitability for lab and field have been favorable attributes.

Like any method, though, C 403 has some deficiencies. New methods have been investigated to address some of these limitations, as well as to provide additional information to the researcher or the practitioner. Some of these include the "finisher's foot" method for measurements of penetration resistance directly on concrete, as well as continuous penetration measurements on mortars. Others include the use of changes in the thermal wave propagation or rheological characteristics with time. Expectations are that some of these methods will be standardized in the next decade, and be included in ASTM manuals by the time of writing the next edition of this chapter. Other methods, either newly discovered or overlooked in this writing, will undoubtedly become known as well.

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12

Bleed Water

Steven H. Kosmatka[1]

Preface

THE SUBJECT OF BLEEDING WAS BRIEFLY AD-dressed in the first edition of *ASTM STP 169,* published in 1956. Ivan L. Tyler, manager of the Field Research Section of the Portland Cement Association, concisely described the general significance of tests for bleeding in his article on Uniformity, Segregation, and Bleeding in the Freshly Mixed Concrete section of *ASTM STP 169. ASTM STP 169A* and *ASTM STP 169B* did not address bleeding. *ASTM 169C* had a chapter similar to the one presented here. The effects of concrete ingredients on bleeding as well as the significance of bleeding with modern concretes are presented in this chapter. This chapter also reviews the standard ASTM test methods on bleeding and provides data on the bleeding characteristics of a variety of cement pastes, mortars, and concretes.

Introduction

Bleed water is the clear water that can gradually accumulate at the surface of freshly placed concrete, mortar, grout, or paste (Fig. 1). Bleed water is caused by sedimentation or settlement of solid particles (cement and any aggregate) and the simultaneous upward migration of water. This upward migration of water and its accumulation at the surface is called bleeding, also referred to as water gain, weeping, and sweating in some countries. A small amount of bleeding is normal and expected on freshly placed concrete. It does not necessarily have an adverse effect on the quality of the plastic or hardened concrete. However, excessive bleeding can lead to some performance problems with plastic or hardened concrete. With proper mix proportioning, mixture ingredients, placing equipment, and proper construction practices, bleeding can be controlled to a desirable level.

Significance

Bleeding is not necessarily a harmful property nor is excessive bleeding desirable. Because most concrete ingredients today provide concrete with a normal and acceptable level of bleeding, bleeding is usually not a concern and bleeding tests are rarely performed. However, there are situations in which bleeding properties of concrete should be reviewed prior to construction. In some instances lean concretes placed in very deep forms have accumulated large amounts of bleed water at the surface. This not only creates a placing problem but also reduces the strength and durability of the concrete near the surface. Excessive bleeding also delays finishing as finishing should not proceed with observable bleed water present. On the other hand, lack of bleed water on concrete flat work can sometimes lead to plastic shrinkage, cracking, or a dry surface that is difficult to finish.

The first reported case of bleeding in North America was in 1902 during the construction of the stadium at Harvard University [1,2]. During placement, up to 2/3 m of bleed water developed. Up to 150 mm of concrete was removed from the top of each lift prior to the sequential placements in order to remove the less durable and weaker concrete. Even with the high degree of bleeding, this structure survived the elements for over 100 years and will be serviceable for many years to come (Fig. 2). Structures in which severe exposures exist and in which porous concrete was not adequately removed have not performed as well as structures from which the bleeding-damaged concrete was properly removed.

During the construction of massive structures, such as deep foundations, tall walls, or dams, bleeding became of concern in early concrete projects. To study and help control bleeding, a variety of bleeding tests were developed. These tests will be discussed later under the section on Test Methods. By understanding the process of bleeding, Powers [3] and others provided means to control bleeding and today bleeding is rarely a problem.

Bleeding can occur at any time during the transportation, handling, and placing of concrete, as well as shortly after placement. Most of the discussion in this chapter will focus on concrete after it is placed in a form and will no longer be agitated. This chapter will address the effects of concrete ingredients and placing practices on bleeding and the effect of bleeding on various concrete properties. Much of the discussion pertains to cement paste or mortar as bleeding in concrete is a direct function of the paste or mortar bleeding properties.

Fundamentals of Bleeding

A fresh concrete mixture is merely a mass of concrete ingredients that are temporarily suspended due to the agitation and mixing of the material. Once the agitation stops, the excess water rises through the plastic mass to the surface or, more appropriately, the solid ingredients settle. Although the actual volume of the total ingredients does not change, the height of the hardened concrete is less than the original plastic height as the bleed water will come to the surface and evaporate away.

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Fig. 1—Bleed water on the surface of a freshly placed concrete slab.

The accumulation of water at the surface of a concrete mixture can occur slowly by uniform seepage over the entire surface or at localized channels carrying water to the surface. Uniform seepage is referred to as normal bleeding. Localized channels of water coming up through the concrete, sometimes carrying fine particles, is termed channel bleeding and usually occurs only in concrete mixes with very low cement contents, high water contents, or concretes with very high bleeding properties.

As bleeding proceeds, the water layer at the surface maintains the original height of the concrete sample in a vessel, assuming that there is no pronounced temperature change or evaporation. The surface subsides as the solids settle through the liquid (Fig. 3). Fig. 4 illustrates a typical bleeding curve relating subsidence of the surface to time. The initial subsidence occurs at a constant rate, followed by a decreasing bleeding rate (Fig. 4).

Fig. 2—Harvard University stadium after 90 years of service (photo courtesy of Tim Morse).

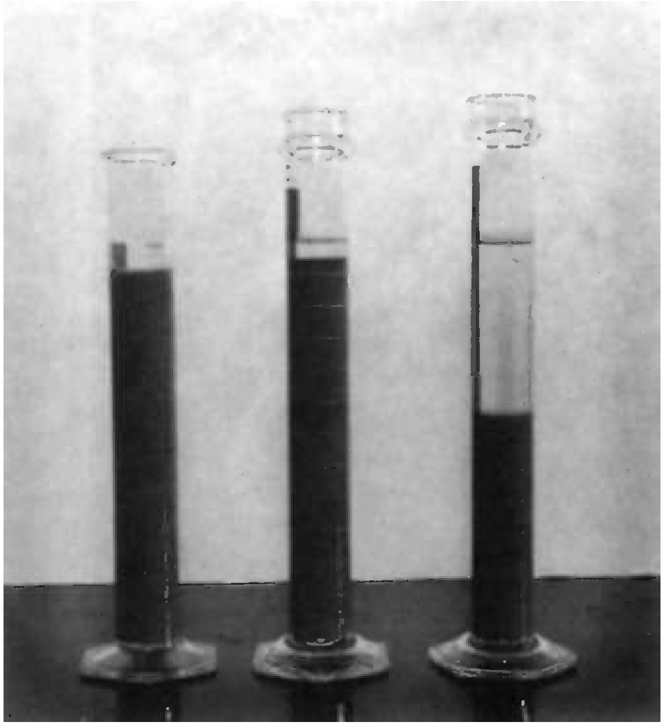


Fig. 3—Demonstration of bleeding or settlement of cement particles in cement paste with water-cement ratios by weight of 0.3, 0.7, and 2.2 (left to right). All cylinders contain 250 mL of paste and were photographed 1 h after the paste was mixed and placed in the cylinders. Observe the accumulation of bleed water for pastes with the higher water-cement ratios.

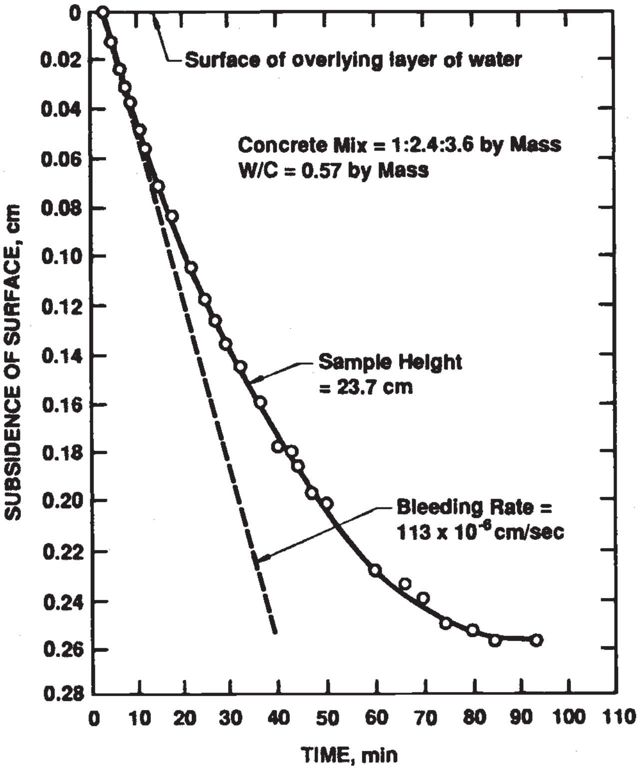


Fig. 4—Typical bleeding curve for concrete illustrating surface subsidence with respect to time [3].

In the interval between the beginning of subsidence and setting, there are three primary zones describing the nature of the bleeding process (Fig. 5). These are the zones of (1) clear water at the sample surface, (2) constant water content or density, and (3) compression. Figure 5 is a simplified version of a five-zone analysis of the bleeding process of paste presented by Powers [3]. In the zone of constant water content, the water-to-cement ratio and density are essentially constant, even though some water is moving through the zone. The compression zone is a transition zone where the paste is being densified. The water-to-cement ratio is also being reduced and the solid particles represent a lesser volume than when originally placed. The paste densifies until it stabilizes and stops settling or bleeding. At equilibrium, the paste achieves a stable volume and the degree of consolidation in this fresh state dictates the hardened properties of the paste, such as strength and durability.

Bleeding is often analyzed in terms of bleeding rate and bleeding capacity. Bleeding rate is the rate at which the bleed water moves through the plastic concrete, mortar, or paste. Bleeding rate can be expressed in terms of cubic centimetres of bleed water per second per square centimetre of sample surface, centimetres per second, millimetres per second, or other applicable units. The bleeding rate of pastes with different cements and water-to-cement ratios is shown in Fig. 6. The rate of bleeding is controlled by the permeability of the plastic paste. As the solid particles settle, the flow of water is controlled by the permeable space or capillaries between particles.

The bleeding rate of concrete is less than that of paste alone with the same water-cement ratio. However, this would be expected as the aggregate in the concrete replaces some of the volume of the paste. The velocity of the water moving through the paste within the concrete is greater than it is for paste alone. This is due to the greater unit weight of the concrete. Consequently, the hydraulic force induced by the aggregate in the concrete disrupts the paste structure more so than in paste alone. Because of this, channeled bleeding develops in concrete sooner than it does in paste alone and it develops at lower water-cement ratios for concrete than for paste. The rate of bleeding in a concrete mixture is controlled by many variables that will be discussed later under the section on Effects of Ingredients on Bleeding.

Bleeding capacity is the quantity of bleed water that a particular concrete, mortar, grout, or paste mixture can release to the surface with respect to a certain depth. It is usually expressed in terms of the settlement or change in height of the paste- or mortar-solid's surface per unit of original sample height (or, in other words, the ratio or percentage of the total decrease in sample height to the initial sample height). Bleeding capacity can also be expressed as a percent of the mix water. Figure 7 illustrates the bleeding capacity for pastes with a range of cements and water-cement ratios. Table 1 shows bleeding capacities for concrete and mortar. Figure 8 demonstrates these data in comparison to paste. Bleeding capacity is directly related to the water and paste content of concrete mixtures. Higher water contents especially increase bleeding capacity. As expected, bleeding capacity is closely related to bleeding rates (Fig. 9).

Duration of Bleeding

The length of time that the concrete bleeds depends upon the depth of the concrete section as well as the setting properties of the cementitious materials. A thin slab of concrete will settle or bleed for a shorter period of time than a deep section of concrete. Likewise, a concrete that sets up quickly will bleed much less than a concrete that takes many hours to set up.

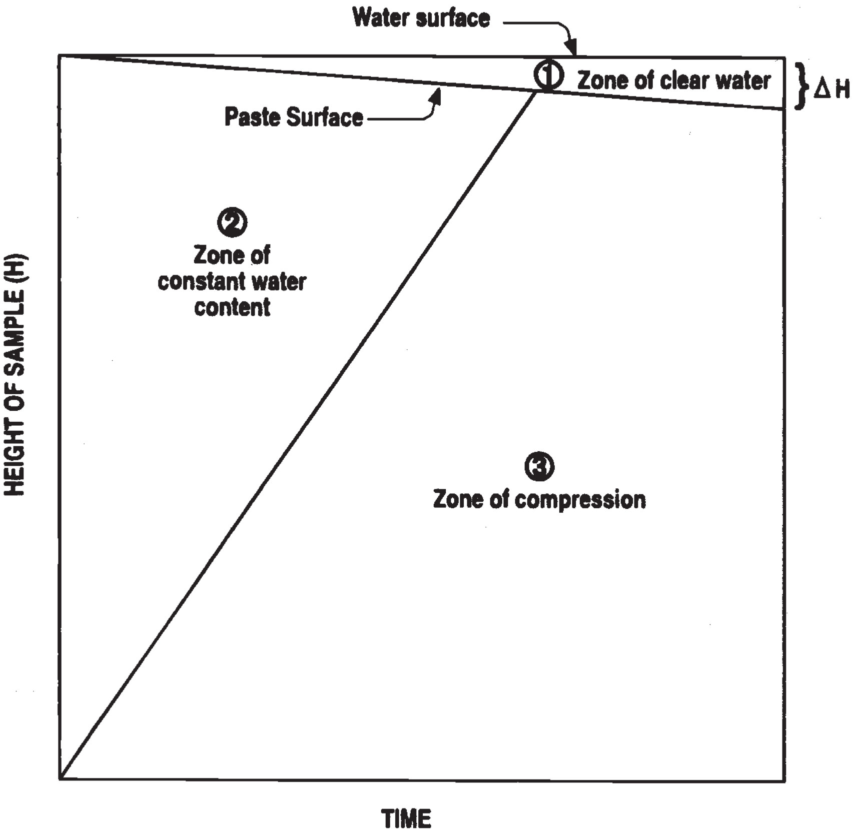


Fig. 5—Illustration of the process of bleeding in cement paste.

Most bleeding occurs during the dormant period, when cementing materials have little to no reaction. The dormant period is commonly around an hour. However, chemical and mineral admixtures as well as different compositions and finenesses of cements can greatly affect the dormant period. Figure 10 illustrates the increase in bleeding with increased paste height and dormant period.

Effects of Bleeding on Plastic Concrete

Volume Change

Combining cement, water, and aggregates in a mixer creates a disbursed and suspended state of particles in plastic concrete. This suspended state is not stable because the heavier particles of cement and aggregate are forced downward through the

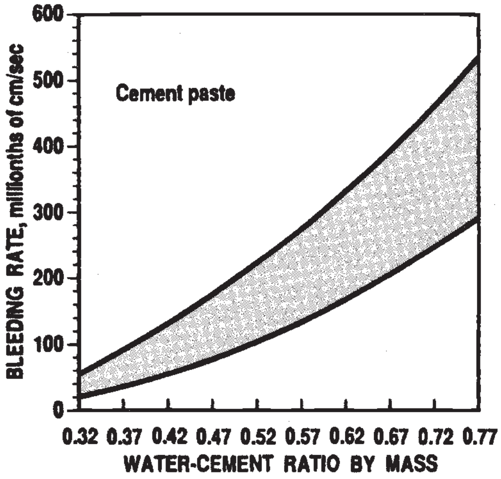


Fig. 6—Range in relationship between bleeding rate and water-cement ratio of pastes made with normal portland cement and water. The range is attributed to different cements having different chemical compositions and finenesses [4].

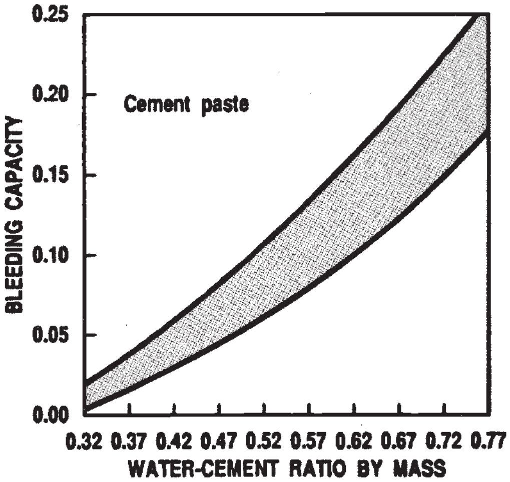


Fig. 7—Range in relationship between bleeding capacity (total settlement per unit of original paste height) and water-cement ratio of pastes made with normal portland cement and water. The range is attributed to different cements having different chemical compositions and finenesses [4].

TABLE 1—Bleeding Capacities of Concretes and Mortars [3]

Mix by Mass, Cement: Sand: Gravel	Water-Cement Ratio by Mass, w/c	Approximate Slump, mm	Bleeding Capacity, ΔH	Paste per Unit Volume, p	(ΔH)/ p
		Concrete (Aggregate: 75 μm TO 19 mm)			
1:0.8:1.2	0.31	102	0.009	0.446	0.020
1:1.2:1.8	0.38	213	0.011	0.374	0.028
1:1.6:2.4	0.43	203	0.013	0.323	0.042
1:1.6:2.4	0.40	119	0.009	0.314	0.028
1:1.9:2.85	0.49	203	0.012	0.304	0.041
1:2.4:3.60	0.53	229	0.013	0.266	0.041
		Mortar (Aggregate: 75 μm TO 4.75 mm)			
1:0.8	0.34	...	0.018	0.688	0.028
1:1.2	0.38	...	0.019	0.614	0.034
1:1.6	0.41	...	0.019	0.570	0.034
1:2.0	0.45	...	0.019	0.506	0.037

lighter water by gravity. The downward movement of the solid particles continues until settlement ceases when the particles are in contact with one another and densify. As the surface of the disbursed solids is replaced by water, the volume of solid matter decreases. Although the total volume of materials is relatively constant, the volume after bleeding will be less than that of the original plastic mixture.

The total amount of settlement is proportional to the depth of the freshly placed concrete. Settlement can occur even though bleed water is not observed at the surface. This is because on many occasions, such as warm windy days, the rate of evaporation is sufficient to remove the bleed water as it comes to the surface. The amount of volume reduction is clearly demonstrated in Fig. 4 in terms of settlement of the surface.

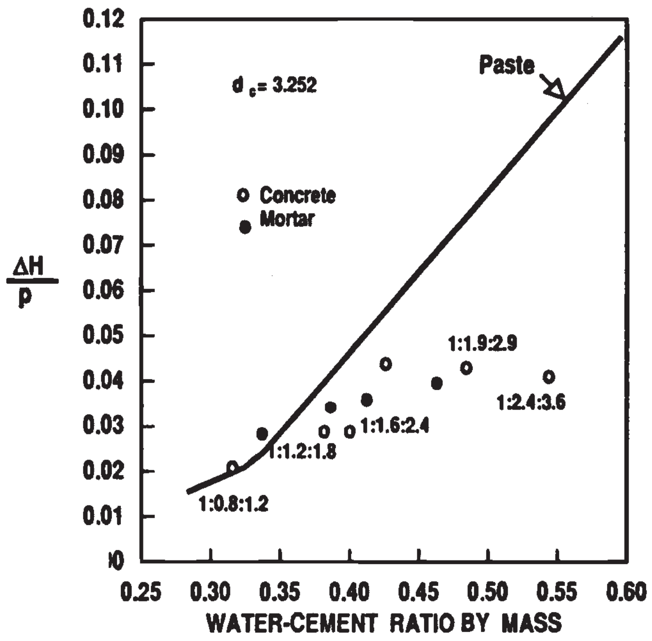


Fig. 8—Bleeding capacities of mortars and concretes per unit of paste in the mix compared with the bleeding capacities of neat pastes of the same water-cement ratio. Concrete mixture proportions are by mass [3].

The small amount of settlement or volume reduction is not of concern for most general construction practices or applications. However, applications in which concrete is being placed under an item that it must support, such as a machine base, should have little to no bleeding to prevent the formation of a void between the concrete surface and the object the concrete is to support. Bleeding also increases the risk of plastic settlement cracks over embedded items such as reinforcing steel.

Postbleeding Expansion

Following the bleeding period, expansion occurs within the paste. This postbleeding expansion is caused by a combination of physical and chemical reactions occurring during the first stages of setting. In effect, the gel coating on cement grains, disrupted during hydration, may exert enough pressure to cause the sample to increase in volume. Most of this expansion occurs within the first day. Typical one-day expansions range from 0.05 % to 1 % for portland cement pastes at a water-cement ratio of 0.38 by weight [5,6]. Expansion beyond the first

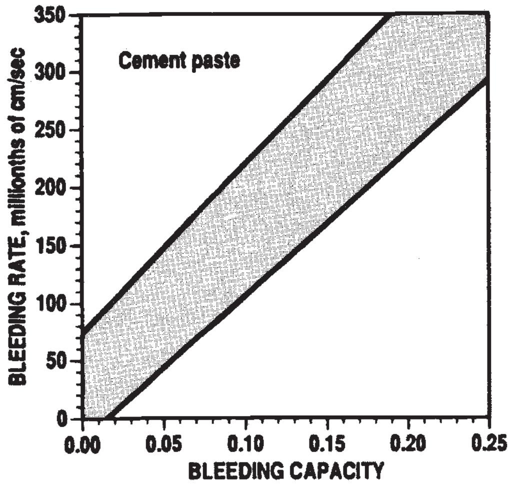


Fig. 9—Relationship between bleeding rate and bleeding capacity for cement paste using a variety of cements. Approximately 100 data points were used to develop the range [4].

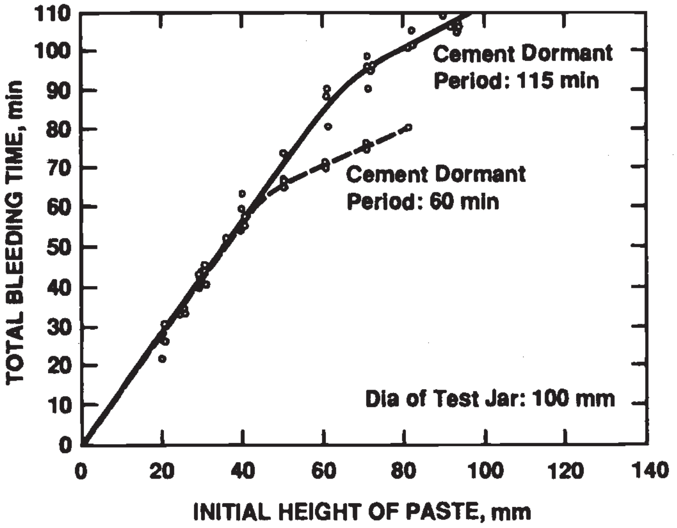


Fig. 10—Bleeding time versus height of paste sample for cements with different dormant periods [4].

day can be expected to be less than half of that which occurs within the first day and is not likely to add more than 0.05 % expansion. Table 2 illustrates the postbleeding expansion of a cement paste. The bleeding period ended at 1 h and 12 min and the expansion began at 1 h and 30 min. It must be realized that the amount of expected expansion for concrete would be much less than that for paste alone or even nondetectable. It must also be realized that the samples in Table 2 were submerged continuously during the test. Otherwise, autogenous shrinkage opposes the expansion as the cement hydrates.

TABLE 2—Post-Bleeding Expansion of Cement Paste[a] [5]

Age Interval, h	Observed Rate of Expansion, %/h	Total Expansion, %
0 to 1 1/2	0	0
1 1/2 to 2 1/2	0.10	0.10
2 1/2 to 3 1/2	0.04	0.14
3 1/2 to 4 1/2	0.04	0.18
4 1/2 to 5 1/2	0.02	0.20
5 1/2 to 6 1/2	0.02	0.22
6 1/2 to 23 1/2	0.01	0.38

[a] Type I cement was used with a water-cement ratio of 0.38 by mass. The age of the paste at the end of the bleeding period was 1 h 12 min. The age at the beginning of the expansion was 1 h 30 min. Expansion is expressed as the percent of the depth of the sample.

Plastic Shrinkage

Plastic shrinkage, sometimes called setting shrinkage in older literature, is shrinkage that occurs before the concrete has hardened. This shrinkage results from a loss of free water in the mixture. The water loss and resulting shrinkage is caused by a combination of loss of free water from the concrete due to bleeding and surface evaporation as well as consumption of the water by the cement during hydration (autogenous and chemical shrinkage). The amount of bleeding and surface evaporation predominantly control the amount of plastic shrinkage. If the rate of evaporation at the sample surface exceeds the bleeding rate, plastic shrinkage occurs. The three factors most significantly contributing to autogenous shrinkage are a concrete's

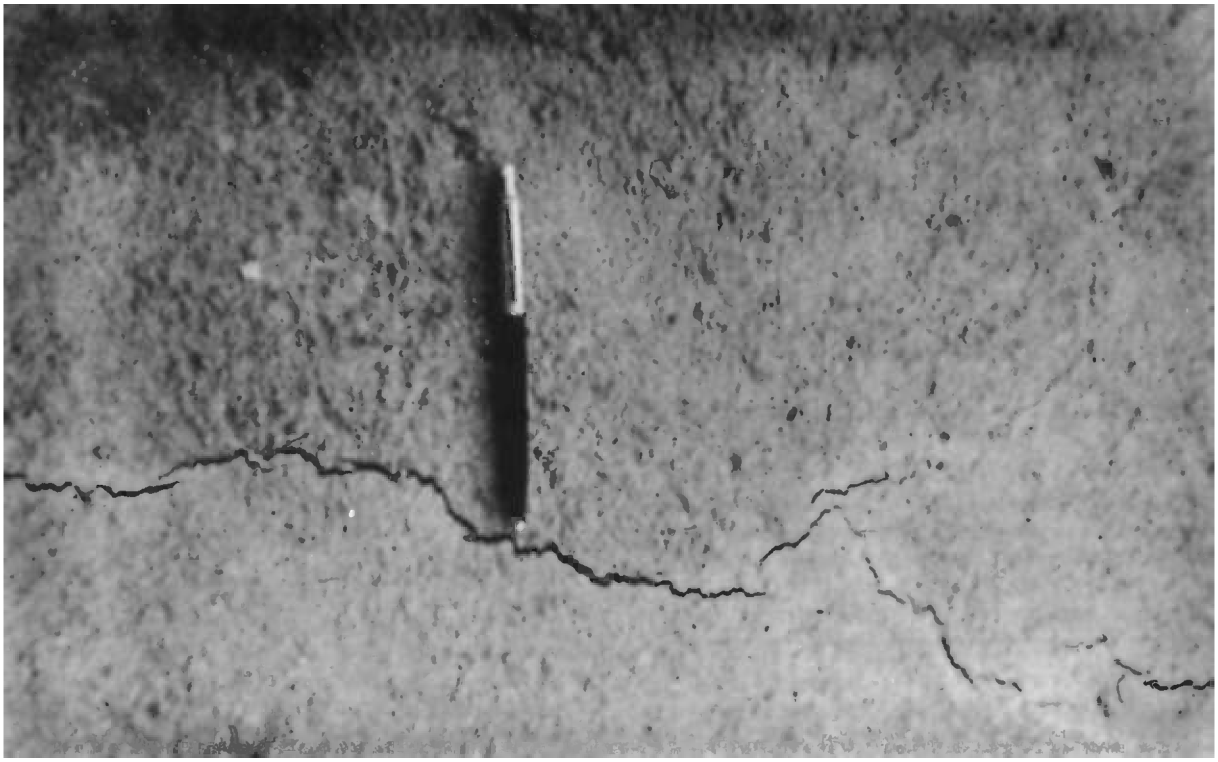
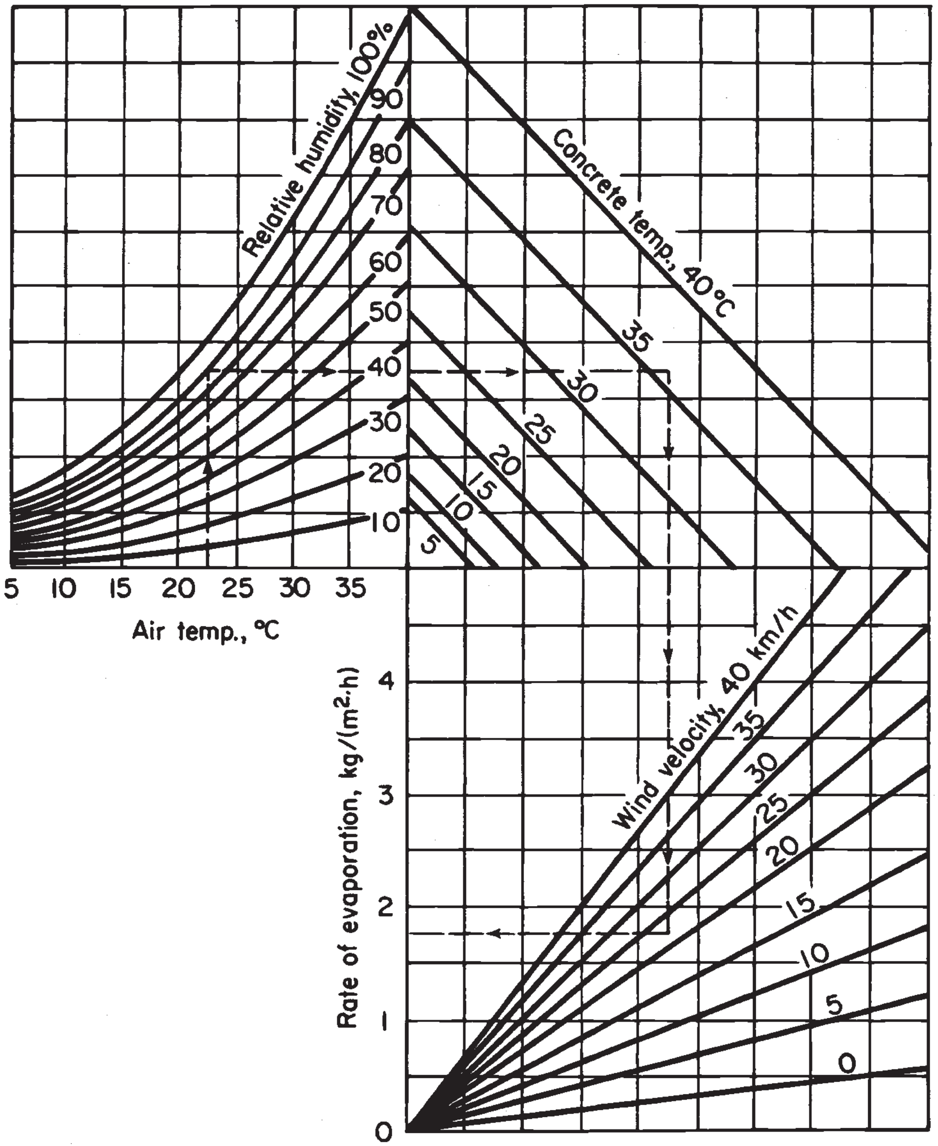


Fig. 11—Plastic shrinkage cracks in concrete (photo courtesy of the Portland Cement Association).

chemical shrinkage, amount of bleeding, and time of hardening. Autogenous shrinkage is very small and usually does not contribute significantly to total plastic shrinkage for concretes with water-cement ratios greater than 0.42 [28]. The evaporation and removal of the bleeding water from the concrete creates tensile stresses near the surface. These tensile stresses can pull the concrete away from the form as well as form plastic shrinkage cracks in the concrete that resemble parallel tears (Fig. 11).

The disappearance of the water from the surface of the concrete indicates when the rate of evaporation has exceeded the bleeding rate. Shortly after this time, plastic shrinkage and cracking occur. The time required to obtain this condition is controlled by the air temperature, relative humidity, wind velocity, concrete temperature, and bleeding characteristics of the concrete (Fig. 12). At this stage, the concrete has obtained a small amount of rigidity yet is unable to accommodate the rapid volume change induced by plastic shrinkage. Consequently, tensile stresses develop and plastic shrinkage cracks form.

The best way to prevent plastic shrinkage cracking is to prevent surface evaporation. Plastic shrinkage cracks can penetrate from one fourth to full depth of a concrete slab. Although plastic shrinkage cracks may be unsightly, they often do not hinder performance of nonreinforced concrete. Plastic shrinkage cracks can also be reduced by the use of concrete with higher bleeding characteristics as well as the use of fibers, evaporation retarders, shades, windbreaks, and plastic sheets or wet burlap covering the slab.



Instructions for using this chart:
1. Enter with air temperature, move *up* to relative humidity.
2. Move *right* to concrete temperature.
3. Move *down* to wind velocity.
4. Move *left*: read approximate rate of evaporation.

Fig. 12—Nomograph demonstrating the effect of concrete and air temperatures, relative humidity, and wind velocity on the rate of evaporation of surface moisture from concrete. Evaporation rates exceeding 1 kg/m^2/h are prone to induce plastic shrinkage cracks in concrete (adapted from Ref [7]).

Senbetta and Bury [33] demonstrated that plastic shrinkage cracking can be controlled in cold weather by lowering the temperature of fresh concrete, thus minimizing the differential between the temperature of the concrete and the ambient temperature. This results in a lower rate of evaporation of bleed water from the concrete surface. The use of a freezing-weather admixture can be used to offset slower setting time associated with a cooler concrete temperature.

Concretes with high cementing materials contents (around 500 kg/m^3) or that use silica fume or low water-to-cementitious material ratios (less than 0.42) aggravate plastic shrinkage crack development.

Water-Cement Ratio

The water-cement ratio of a concrete mixture before bleeding is higher than after bleeding. As illustrated in Figs. 3 and 5, a densification of particles occurs. As the solid material compresses, some of the water leaves the concrete and rises to the surface at which time it evaporates away. This applies to most of the concrete depth. However, where excessive bleeding occurs, the water-cement ratio of the surface concrete may actually be increased and that particular concrete should be removed in extreme situations. In addition, if the concrete is sealed or troweled before the bleed water comes to the surface, the water may be trapped under the surface creating a weakened zone with a higher water-cement ratio than the rest of the concrete. In most applications, normal bleeding is beneficial in reducing the actual water-cement ratio of most of the concrete mixture in place.

Thixotropic Mixtures

Thixotropic mixtures of concrete, mortar, or grout have low bleeding properties. They exhibit a cohesive nature and, because of the ingredients in the mixture, they have little to no bleeding. This is illustrated in the left cylinder in Fig. 3, which contains a paste with thixotropic properties. Thixotropic mixtures are important in applications where the volume of the fresh concrete or mortar must equal the volume of the hardened concrete or mortar. An example would be in the use of supporting grout under machine base plates [8].

Placing and Finishing

Normal bleeding usually does not interfere with the placing and finishing of concrete mixtures. However, excessive bleeding of low-cement-content mixtures may cause undesirable early segregation during transportation and placement. Some minor bleed water is desirable to help keep the surface paste moist and help provide lubrication during the finishing of concrete; however, bleeding should not be so excessive as to interfere with the finishing operation. *Concrete must never be finished with visible bleed water on the surface as such practices promote dusting, scaling, blisters, and other surface defects* [9]. Usually bleed water is allowed to evaporate away before finishing commences.

Concrete placed on a sub-base of low permeability, such as clay, plastic sheeting, metal-deck forms, bituminous concrete, or vapor barriers, bleeds noticeably more than concrete placed on a granular base. Therefore, special care and planning should be used when differences in bleeding rates are caused by isolated vapor barrier locations.

If excessive bleeding occurs between lifts in a wall placement, the water and about 100 mm of surface concrete should be removed prior to placing the next lift. This practice, although not usually needed for concrete with normal bleeding, removes bleeding-induced low-strength concrete of poor durability (see the sections on Durability and Removing Bleed Water).

Effect of Bleeding on Hardened Concrete

Strength and Density

The strength of hardened concrete is directly related to the water-cement ratio. As the solid particles in the paste or concrete settle, they squeeze some of the water out of the paste, especially in the lower part of the placement. This lowers the water-cement ratio and increases the strength. Because the degree of consolidation or settlement is not uniform throughout the height of a sample (more consolidation at the bottom than the top), the strength can be expected to be slightly higher at the bottom than at the top.

The differential consolidation effect is usually identified by an increase in concrete density. For example, a caisson placement in the Chicago area illustrates the consolidation/density effect. The top, middle, and bottom of a 12-m-deep caisson had densities of 2400, 2435, and 2441 kg/m^3, respectively [29]. Hoshino [10] demonstrated the effect of bleeding on strength, illustrating strength increase with depth for normal-strength concretes with water-to-cement ratios of 0.6 and 0.7 by weight. Matsushita and Sue [30] demonstrated that in the occurrence of a cold joint, the strength of an element can be increased by removing bleed water from the lower layer of concrete.

With regular concrete, a differential settlement between the paste and aggregates occurs. The aggregate can settle only until point-to-point contact (bridging) between aggregates occurs. The paste continues to settle in between the stabilized aggregate particles. This phenomenon also contributes to a weakening of the paste due to bleeding, reduction of cement particles, and increase in water content in the upper portion of the concrete. Separation between paste and aggregate due to the accumulation of bleed water around aggregate particles also reduces strength.

Paste-Aggregate Bond

Bleed water can accumulate under and alongside coarse aggregate particles (Fig. 13). This is especially prone to hap-

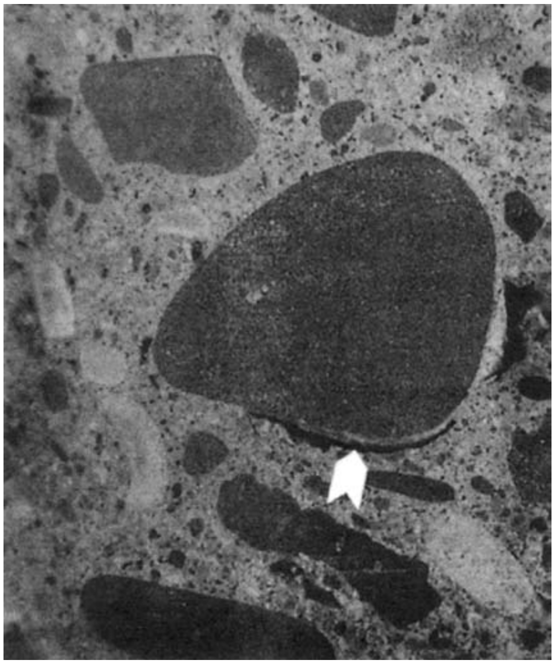


Fig. 13—Cross section view of concrete illustrating bleed water accumulation along a coarse aggregate particle.

pen when differential settlement occurs between the aggregate and paste. Once the aggregate can no longer settle, the paste continues to settle allowing bleed water to rise and collect under the aggregate. Bleed-water channels also tend to migrate along the sides of coarse aggregate. This reduction of paste-aggregate bond reduces concrete strength. This condition can partially be reduced by revibrating the concrete after some bleeding has occurred. A broken fracture face of a horizontally oriented crack from a vertical placement demonstrates the reduced paste-aggregate bond caused by bleeding. The upper portion will have half-embedded aggregate particles, whereas the lower face (opposing face) will expose the socket or imprint of the aggregate in the paste. This reveals the poor bond at the underside of the aggregate particle.

Sometimes, if the concrete's surface mortar sets faster than the rest of the concrete due to hot weather conditions, some of the coarse aggregate particles might settle, leaving a small air void above the aggregate particle. Another phenomenon is the presence of flat particles near the surface that inhibit bleed water from entering mortar that is above the aggregate. Upon rapid evaporation, this mortar dries out quickly and does not have the strength or durability of the surrounding mortar. This can result in a condition called "mortar flaking" over the coarse aggregate particles.

Paste-Steel Bond

Bleed water is prone to collect under reinforcing steel and other embedded items (Fig. 14). This is because not only does bleed water have a tendency to collect under large objects in concrete, but as the concrete settles, the concrete pulls away from the steel leaving an air void and an easily accessible location for water to collect.

A minor collection of bleed water or settlement under reinforcement is not detrimental to strength development in the bar because most of the stress is applied to the bar deformations. However, excessive water collection and void development can reduce bar embedment strength, paste-steel bond, and possibly promote corrosion of the steel at void locations–especially in the presence of moisture, carbonation, or chlorides. Corrosion can occur because the steel is not in contact with the corrosion resistive paste. Welch and Patten [11] demonstrated the effect of bleeding or settlement on the bond stress of reinforcing steel. For plain and deformed bars, bond stress was reduced with settlement. As expected, top bars developed less bond than bottom bars because more bleed water accumulated under top bars than bottom bars (Fig. 15). Some differential bond stress can also be attributed to differential strength development between the top and bottom of the specimen.

Durability

Concrete mixtures are designed to be durable in the environment to which they are exposed. A normal, small amount of bleeding does not reduce durability; however, excessive bleeding can have a serious effect if special precautions are not observed (Fig. 16). Durability and concrete's resistance to aggressive chemicals, chlorides, acids, and sulfates are directly related to the permeability and water-cement ratio of the concrete. An increase in water-cement ratio and permeability caused by excessive bleeding would reduce freeze-thaw, deicer-scaling, and sulfate resistance, and allow aggressive materials to enter the concrete.

The relationship between bleeding and durability is demonstrated in a study of plasticized, flowing concrete experiencing excessive bleeding. Bleed channels extending 12 mm below the surface and areas of high water-cement ratio were observed by petrographic analysis. The weakened surface layer and high water-cement ratio, induced by bleeding, contributed to poor deicer-scaling resistance [12]. Weakened surface layers can sometimes delaminate upon exposure to impact or abrasion. Dusting of a concrete surface can also develop.

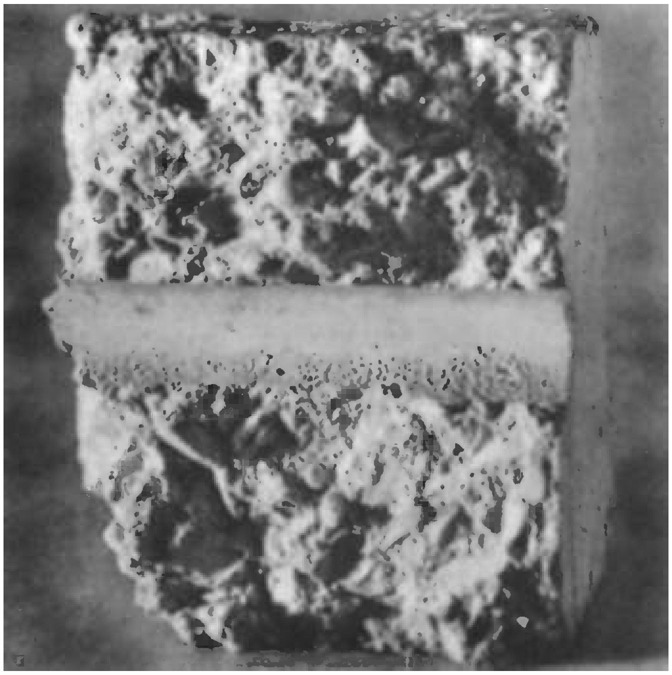


Fig. 14—Imprint in concrete illustrating the collection of bleed water voids under a smooth steel bar held firmly in a horizontal position during and after placement [2].

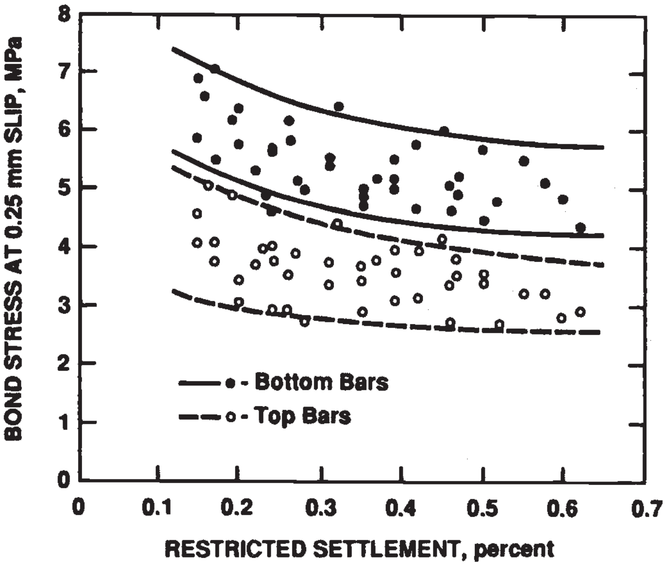


Fig. 15—The effect of concrete settlement on the bond strength of horizontally embedded deformed bars using ASTM Test Method for Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steels (C 234). Bar diameter was 19 mm. Concrete slump was 50 to 125 mm. The top and bottom bars were 75 and 230 mm above the sample base [11].

Fig. 16—Deterioration at top of lifts in a dam from which the porous-nondurable concrete was not adequately removed prior to sequential lift placement. The poor quality concrete at the lift surface was caused by excessive bleeding and deteriorated due to frost action.

Corrosion of reinforcement is more likely when bleed water collects under reinforcement steel. Also, if the upper few centimetres of a deep concrete section become significantly porous due to excessive bleeding, carbonation, chlorides, air, and moisture would more readily reach the steel, thereby inducing corrosion.

Grouted prestressing strands can experience corrosive attack, particularly corrosion pitting, in the presence of grout bleed water and air. The corrosion occurs in a sealed environment despite the high pH of the bleed water and low levels of chlorides. The corrosion mechanism is most likely a carbonation reaction involving carbon dioxide in the air and calcium hydroxide in the grout bleed water. This reaction creates regions of low pH at the surface of the bleed water where the passivity of the steel is lost and corrosion subsequently occurs [31].

Bleeding channels can carry lightweight materials to the surface that can reduce abrasion resistance as well as allow popouts to form. A chemical analysis of bleed water indicates that channeled or uniform bleeding can leach alkalies up from the concrete to near the surface [13]. Upon evaporation of the water, an accumulation of alkalies can develop in the upper few millimetres of the surface. If alkali-aggregate-reactive particles are in this zone, the increased concentration of alkalies can aggravate alkali-aggregate reactivity (AAR), possibly resulting in popouts or surface cracking. A similar accumulation of alkalies can develop if the surface is sealed too early during finishing. Landgren and Hadley [27] found that AAR-induced popouts can be minimized by wet curing as early as possible, protecting concrete from drying prior to finishing, and avoiding hard troweling.

Scaling

The relationship between bleeding and scaling depends upon the placing, finishing, and curing practices. Figure 17 illustrates the relationship between scaling and subsidence. In this particular example, increased degrees of subsidence were formed by increasing the rate of evaporation over the concrete. Contrary to common belief, this figure illustrates that increased bleeding or subsidence can actually improve scale resistance for this nonair-entrained concrete. It must be realized, however, that this concrete was finished after the bleed water had evaporated away from the surface and after maximum bleeding had occurred [14].

The scale resistance of a concrete surface can be jeopardized whenever the plastic material near the surface has its water content changed or its ability to transmit bleed water is

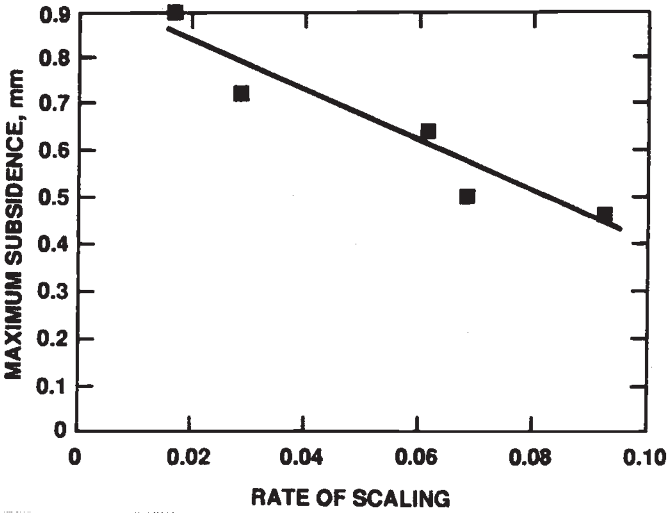


Fig. 17—The relationship between scaling and subsidence. The rate of scaling = numerical scale rating divided by the number of cycles. Higher scaling values indicate more scaling. Concrete was wood floated after maximum subsidence [14].

reduced. Usually, this is achieved by reducing the water-cement ratio or increasing the cement content of the surface material. This can be done by finishing the slab prior to the accumulation of bleed water, or by sprinkling dry cement onto the slab to take up excess water to facilitate finishing.

Steel troweling early seals the surface much more than wood floating. The water rises to the surface and hits the surface stratum of more impermeable cement paste. It cannot penetrate through and merely accumulates under the surface. This accumulation of water either creates a weakened zone of paste or, in some cases, actually creates a water void beneath the surface. Upon freezing, this weakened zone or water-filled void can scale off the surface. Figure 18 illustrates that final finishing prior to the completion of bleeding significantly increases scaling.

Mortar Flaking

Mortar flaking resembles scaling or a flat popout. It is identified by a loss of mortar over flat coarse aggregate particles at the surface. Large flat coarse aggregate particles block the migration of bleed water to the mortar over the aggregate. If rapid evaporation occurs, as with an unprotected surface on a hot windy day, the mortar drys out, shrinks slightly more than the surrounding mortar, and, due to a lack of water for hydration, does not develop adequate strength for frost resistance. Upon repeated freezing in a wet condition, the surface mortar deteriorates and exposes the underlying coarse aggregate, usually with a flat surface parallel to the concrete surface (Fig. 19). Further deterioration to the surrounding mortar usually does not occur, as it received the necessary water for proper strength gain. This condition should not be confused with popouts caused by aggregate that swells excessively upon water saturation or freezing.

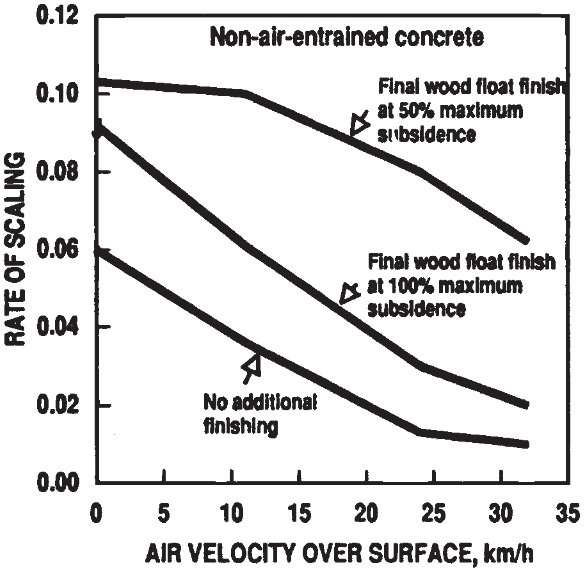


Fig. 18—Effect of time of final finish on scale resistance of concrete after 100 cycles of test. Higher scaling values indicate more scaling. All samples received a wood float strikeoff. Concrete with a final wood finish at 50 % subsidence scaled much more than concrete finished after bleeding stopped or concrete not receiving a final finish [14].

Surface Delamination

Surface delamination here refers to the separation of a large area of surface mortar from the base concrete (Fig. 20). The 1/4- to 1-cm-thick surface delamination can occur in sizes ranging from 10 to 100 cm in diameter. The remaining exposed surface

Fig. 19—Mortar flaking over coarse aggregate (photo courtesy of the Portland Cement Association).

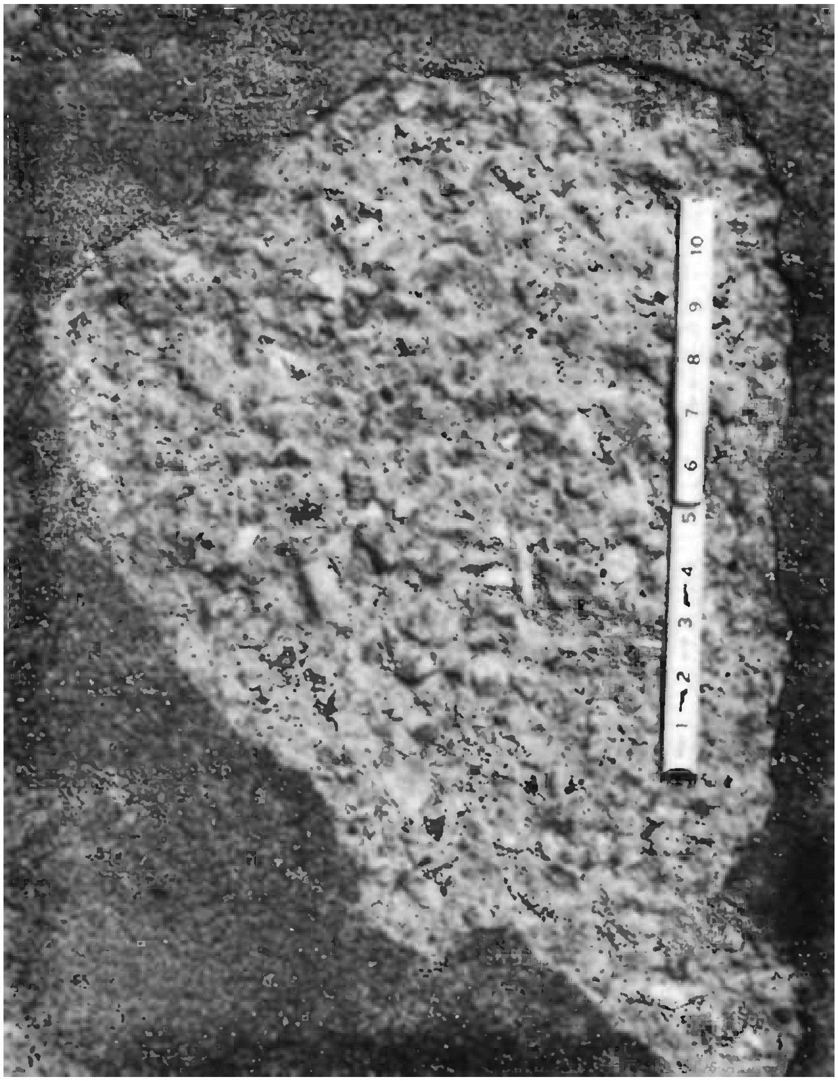


Fig. 20—Delaminated surface caused by early finishing that trapped bleed water under the surface (photo courtesy of The Aberdeen Group).

resembles a scaled surface with coarse aggregate exposed. The cause of surface delamination is the accumulation of bleed water under the surface creating a void or weakened zone (Fig. 21). Upon the freezing of water in the void or weakened zone, the surface delaminates in sheet-like form. In some instances, delamination can occur with interior slabs not exposed to freezing simply because of the large void under the finished surface.

The consolidation of a surface by floating and troweling too early squeezes the water out of the top surface layer reducing the water-cement ratio. The addition of cement to facilitate finishing also reduces the water-cement ratio. These two practices both reduce the settling rate of the surface and make it more impermeable. This allows planes of weakness to develop and bleed water to accumulate under the surface, forming a void. Finishing operations should be delayed as long as the setting time will permit, and the sprinkling of cement on to the surface should be avoided to minimize the risk of developing a plane of weakness or void beneath the surface. Early

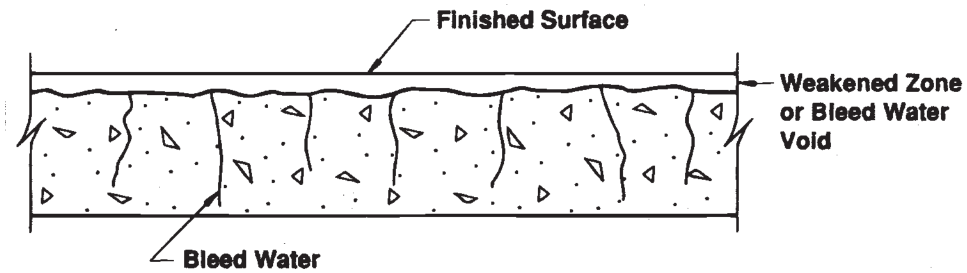


Fig. 21—Illustration of weakened zone or bleed water void under a finished surface.

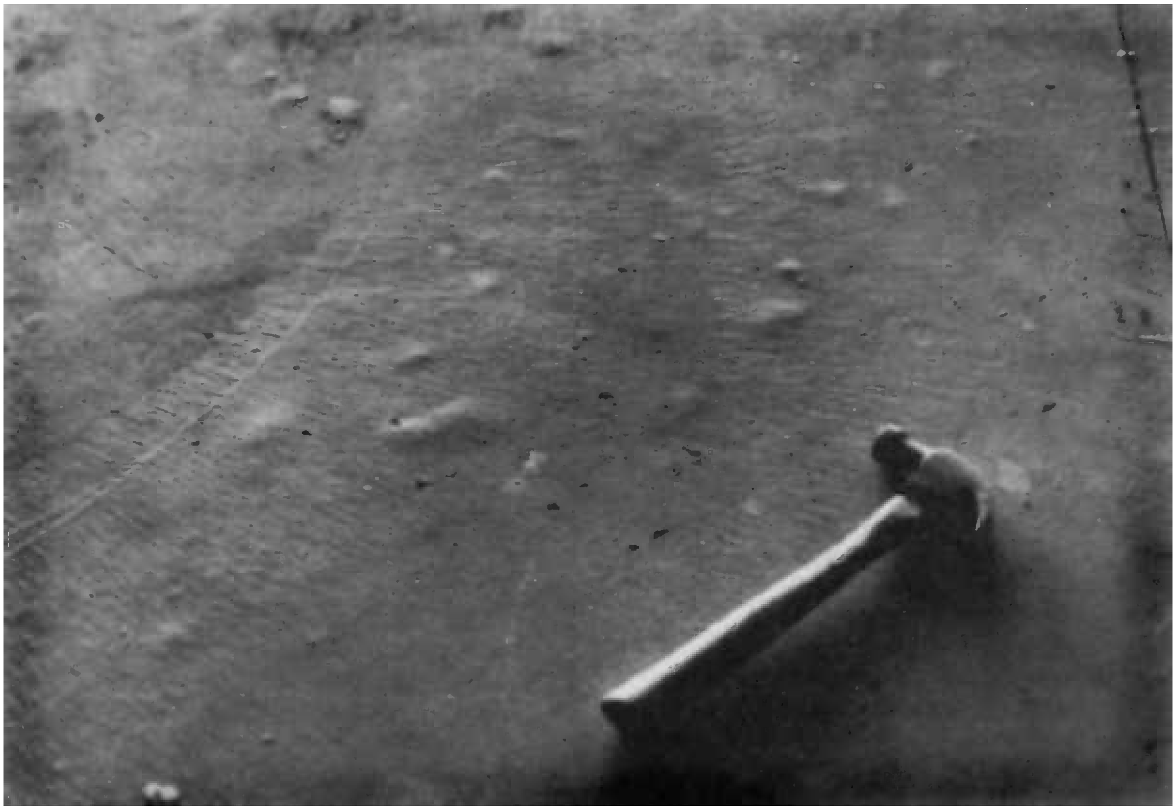


Fig. 22—Blisters (photo courtesy of the National Ready Mixed Concrete Association).

steel troweling is especially prone to trapping bleed water beneath the surface. Jana and Erlin [34] show project examples of delamination caused by trapped bleed water and they present circumstances leading to the entrapment.

Delaminated areas, still intact, can be located by chaining, hammering, or by electro-mechanical sounding procedures all outlined in ASTM Practice for Measuring Delaminations in Concrete Bridge Decks by Sounding (D 4580). Delaminated areas produce a hollow sound upon impact and can often be lifted off the base concrete with a knife or screwdriver.

Blisters

Blisters are small bubbles of water that form under the surface during finishing (Fig. 22). They usually occur during or shortly after steel troweling, but before bleeding has stopped. If punctured while the concrete is plastic, water will usually squirt out. Spaced a few centimetres or decimetres apart, blisters are usually 1 to 10 cm in diameter, 1/4 to 1 cm thick, and visibly rise above the surface. They can form by the accumulation of water under the surface at particular locations–often at the top end of a bleed-water channel (Fig. 23). Formation of blisters is usually an indication that the surface was finished or closed up too early. They are more apt to occur on interior steel-troweled floors. Blisters can also form due to an excess of air in the concrete. An excess of fines or a lack of adequate vibration can also trap air under the finished surface [9].

Surface Appearance

Uniform bleeding on flat work should not affect the color of the surface; however, concretes placed adjacent to one another that have different bleeding rates or different bleeding properties can induce a color change in the surface primarily because of a potential change in water-cement ratio. A concrete that bleeds enough to increase the water-cement ratio at the surface will create a lighter-colored surface. Consequently, a concrete placement with nonuniform bleeding can possibly result in blotchy-colored areas of light and gray. In wall placements, sand streaks can form as the bleed water collects and rises along the form face. As the bleed water moves upward along the form in long bleed-water channels, it washes away some of the paste leaving behind a somewhat sandy appearance (Fig. 24).

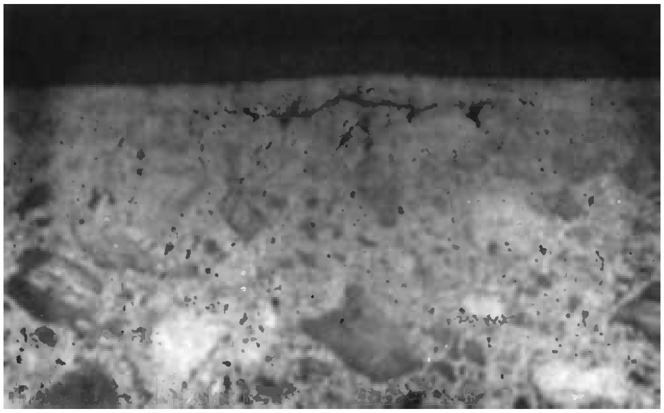


Fig. 23—Cross section of a blister, illustrating the bleed water void under the surface (photo courtesy of the Portland Cement Association).

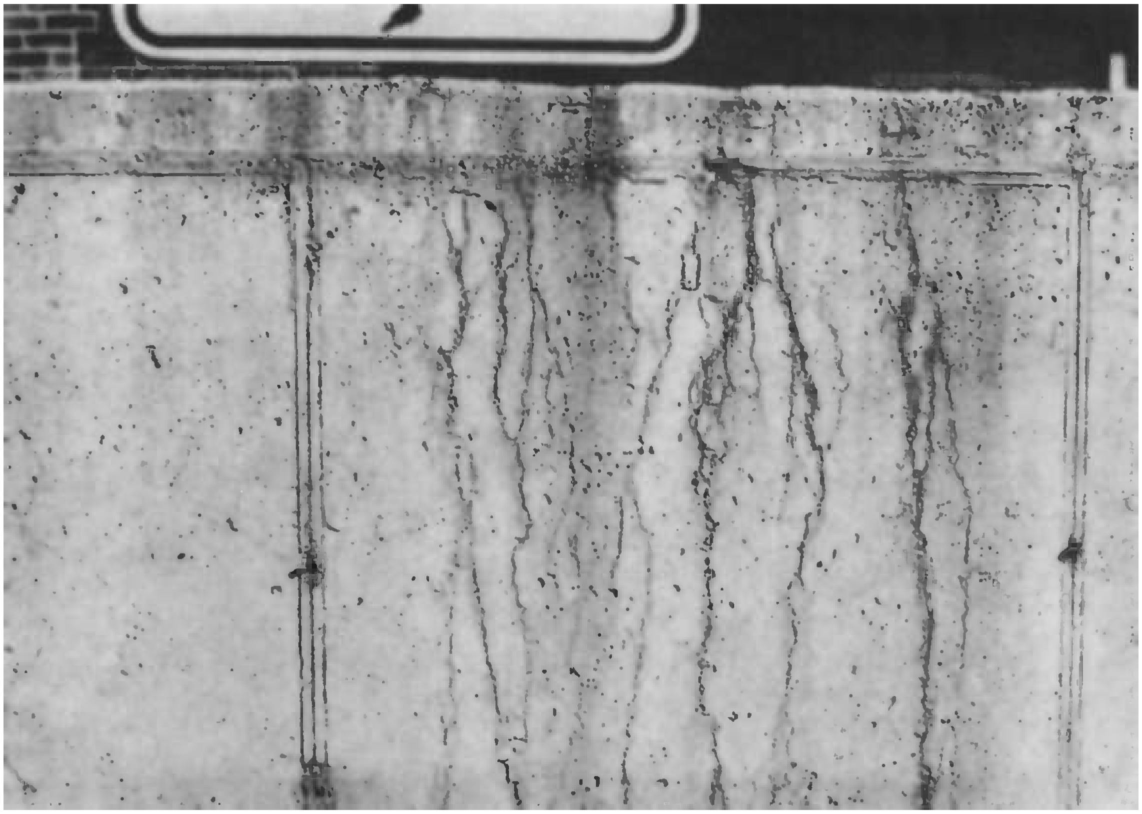


Fig. 24—Sand streaks along a wall caused by excessive bleed water rising along the form (photo courtesy of The Aberdeen Group).

Effects of Ingredients on Bleeding

The individual ingredients and the amount of each ingredient in concrete have a major effect on the bleeding characteristics of concrete. This section will briefly review the effects of some of these common ingredients and their proportions.

Water Content and Water-Cement Ratio

The water content and water-cement ratio predominantly control the bleeding of concrete (Fig. 25). Any increase in the amount of water or the water-to-cementitious material ratio results in more available water for bleeding. Both bleeding capacity and bleeding rate are increased with increased water content. A one-fifth increase in water content of a normal concrete mixture can increase bleeding rate more than two and a half times [3].

Cement

The type, content, chemistry, and fineness of cement can all influence bleeding properties. As the fineness of the cement increases, the amount of bleeding decreases (Fig. 26). Increases in cement content, as it relates to the reduction of water-cement ratio, also reduces bleeding (Fig. 27).

Because the chemical and physical properties of cement are interdependent on one another in how they affect bleeding, it is difficult to isolate the effect of a particular property or chemical compound. Only reactions that occur during the mixing period or bleeding period will affect the bleeding rate. Figure 10 illustrates the effect of cements with different dormant periods on bleeding.

The amount of SO_3 that can be leached from cement in a short time has been found to correlate rather well to bleeding. An increase in SO_3 reduces bleeding [4]. The correlation between water-soluble alkalies in the cement and bleeding is not good. The trend is that an increase in alkalies reduces bleeding; however, other factors such as the precipitation of gypsum and SO_3 content probably have an overshadowing effect.

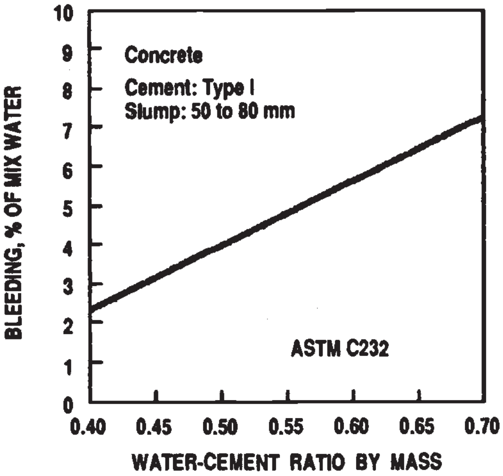


Fig. 25—Relationship between water-cement ratio and bleeding of concrete. Bleeding is expressed as a percent of mix water [15].

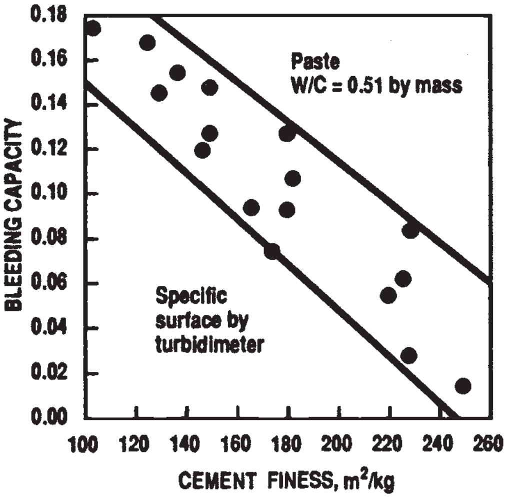


Fig. 26—Effect of cement fineness by Wagner turbidimeter on bleeding capacity of paste. Note that Wagner values are a little more than half of Blaine values [4].

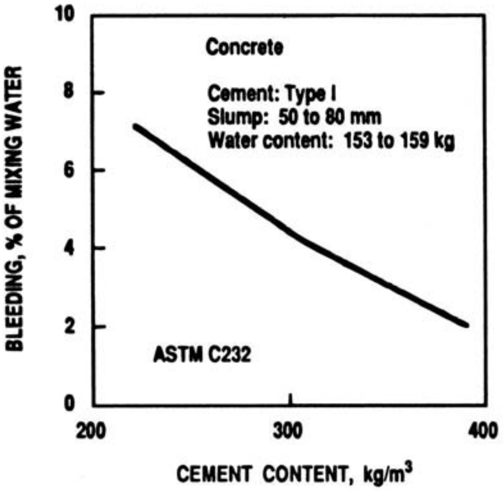


Fig. 27—Relationship between cement content and bleeding of concrete. The increased cement content reflects a decreased water-cement ratio. Bleeding is expressed here as a percent of mix water [15].

The correlation between bleeding and heat liberation demonstrates that bleeding can be reduced by a higher degree of initial chemical reactivity occurring with the cement shortly after mixing. Tricalcium aluminate has a high degree of reactivity and heat-producing capacity and is considered an influence on bleeding properties.

Supplementary Cementing Materials

Fly ash, slag, silica fume, rice husk ash, and natural pozzolans can reduce bleeding by their inherent properties and by increasing the amount of cementitious materials in a mixture. However, each material and different source all have varying effects.

Fly ash usually reduces bleeding. Table 3 compares the bleeding of concretes containing ten different fly ashes with respect to their water reduction and performance in comparison to two controls. Most of the fly ashes reduce bleeding compared to the concretes with cement only. Class C fly ashes reduce bleeding much more than Class F ashes in this study. The ability of fly ashes to reduce bleeding appears to be in their ability to reduce the water demand in the concrete to achieve a particular slump. This is not always the case, as can be

TABLE 3—Bleeding of Concretes With and Without Fly Ash [16][a]

Fly Ash Identification	Class of Fly Ash (ASTM C 618)	Bleeding, % of Mix Water	Bleeding, mL/cm^2 of Surface	Water-Cementing Material Ratio by Mass, W/CM	Change in Mixing Water Requirement Compared to 307 kg/m^3 Control, %
A	C	0.22	0.007	0.40	−7.0
B	F	1.11	0.036	0.42	−2.3
C	F	1.61	0.053	0.42	−2.3
D	F	1.88	0.067	0.45	+4.6
E	F	1.18	0.035	0.41	−4.7
F	C	0.13	0.004	0.40	−7.0
G	C	0.89	0.028	0.42	−2.3
H	F	0.58	0.022	0.44	+2.3
I	C	0.12	0.004	0.42	−2.3
J	F	1.48	0.051	0.43	0
Average of					
Class C		0.34	0.011	0.41	...
Class F		1.31	0.044	0.43	...
Control Mixtures	Cement Content, kg/m^3				
1	307	1.75	0.059	0.43	...
2	282	2.42	0.080	0.48	...

[a] Concretes had a slump of 75 to 100 mm and air content between 6 and 7 %. Test mixtures contained 75 % cement and 25 % fly ash by mass of cementitious material, based on a 307 kg/m^3 cementitious material content. Control mixtures contained no fly ash. Bleeding tested as per ASTM C 232.

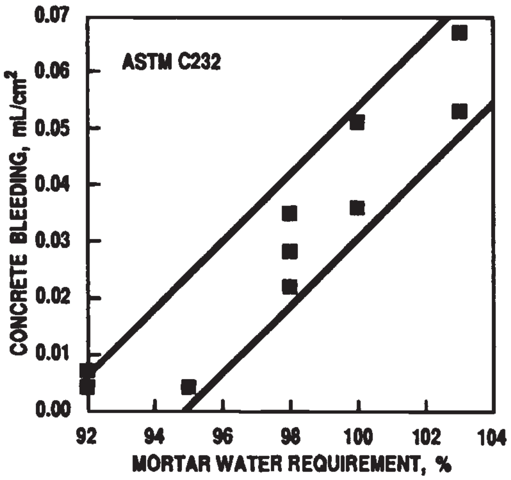


Fig. 28—Comparison of bleeding concrete versus fly ash mortar water requirement of ASTM C 618 [16].

observed with Fly Ash H, which increased water demand and yet still reduced bleeding. Fly Ash J, which has no effect on water, also demonstrated a reduction in bleeding. However, a direct correlation between concrete bleeding and the water requirement of the ASTM Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete (C 618) mortar test exists (Fig. 28). The retardation effect of the fly ashes did not correlate with bleeding [16].

Ground granulated blast furnace slags can increase bleeding capacity and have little effect on bleeding rate compared to mixes with portland cement only. The effect of slag on bleeding is primarily due to the fineness of the slag [17].

Natural pozzolans, such as calcined clay or ground diatomite, usually reduce bleeding. The primary influence is related to the pozzolan's fineness and its effect on water demand. In a study on calcined kaolin clay in mass gravel concrete, pozzolan dosages at 30 and 50 % by volume of cementitious material resulted in bleeding capacities of 9.4 and 6.9 %, respectively. The control mix with 120 kg/m^3 of portland cement had 11.9 % bleeding [18].

Silica fume can greatly reduce bleeding, primarily due to its extreme fineness. Compared to a control with 20 mL of bleeding in samples 40 cm high, one study demonstrated that concretes with silica fume at dosages of 3, 7, and 13 kg/m^3 had bleeding capacities of 12, 8, and 3 mL, respectively [19]. Silica-fume concretes with very low water-to-cementing materials ratios essentially have no bleed water available to rise to the surface. Consequently, such concrete mixtures are prone to plastic shrinkage cracking if proper precautions are not taken to reduce or eliminate surface evaporation while the concrete is in the plastic state.

Rice husk ash (also called rice hull ash) reduces bleeding proportionately with the amount of ash in the paste. The fineness of the material is primarily responsible for the reduction of bleeding [20].

Aggregate

Ordinary variations in aggregate grading have little effect on the bleeding of concrete. This assumes that there is no appreciable change in the minus 75-μm material. Table 4 demonstrates that the specific surface area of the sand, or the fineness, has little effect on the bleeding rate of mortar at four different ranges of water-cement ratio. However, aggregates that contain a high amount of silt, clay, or other material passing the 75-μm sieve can have a significant effect in reducing the bleeding of concrete. This is not surprising as the aggregate represents only a small amount of the surface area within a concrete mixture. For example, consider a concrete mixture with proportions of one part cement to six parts aggregate (coarse plus fine) by mass. The total surface area of the aggregate is only 5 % of the total surface area of the concrete mixture. The surface area of coarse aggregate is essentially negligible [3]. Similar results have been found by other

TABLE 4—Effect of the Specific Surface Area of Sand on the Bleeding Rate of Mortars [3]

Reference Number	Water-Cement Ratio by Mass	Bleeding Rate, 10^6 cm/s	Specific Surface Area of Sand, cm^2/cm^3
47	0.384	30	86
51	0.393	36	99
55	0.402	37	113
59	0.393	32	126
48	0.431	43	86
52	0.443	50	99
56	0.452	47	113
60	0.439	48	126
49	0.477	52	86
53	0.490	63	99
57	0.508	60	113
61	0.490	60	126
50	0.646	114	86
54	0.668	121	99
58	0.693	113	113
62	0.668	112	126

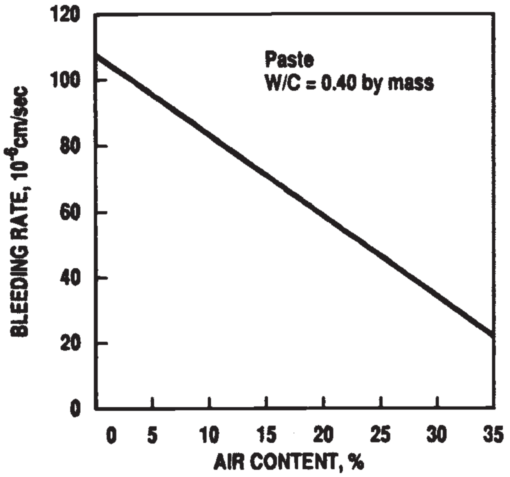


Fig. 29—The effect of entrained air on bleeding rate of paste [3].

researchers, including a negligible reduction in bleeding with reduced particle size [10]. Aggregates that increase water demand, such as crushed rock, tend to increase bleeding due to the higher water content in the mix [32].

Aggregates that significantly increase paste content, such as gap-graded aggregates, can also increase bleeding, especially if the combined aggregate grading is missing the 2.36-mm to 9.5-mm sizes.

Järvenpää [32] determined the relationship between aggregate fineness and pore area and bleeding in conjunction with absorption tendencies and water-retaining capacity of aggregate. As the threshold for the ability of an aggregate to hold mixing water is passed, the bleeding tendency quickly increases.

Chemical Admixtures

The most predominantly used admixtures are air-entraining admixtures and water reducers. In laboratory and field applications, it has been observed that entrained air reduces bleeding. The relationship between air content and bleeding rate for paste is illustrated in Fig. 29. Bruere found that not only do the air-entraining agents reduce bleeding by inducing an air-void system in the concrete, but also the air-entraining admixture itself can slightly reduce bleeding rates [21].

Because water reducers reduce the water content of a concrete mixture, it is expected that they likewise reduce the bleeding. The effect of high-range water reducers or plasticizers is similar to that of normal-range water reducers. A reduction in water content through use of a water reducer results in reduced bleeding. When high-range water reducers are used to reduce the free water content in concrete significantly, they likewise dramatically decrease the bleeding of concretes as illustrated in Fig. 30. These data compare concretes of equal slump.

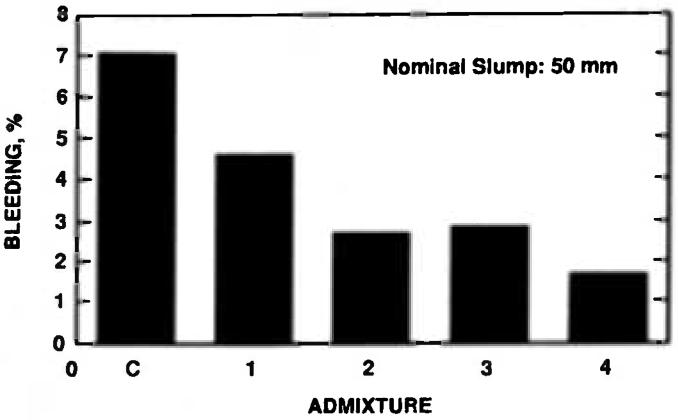


Fig. 30—Effect of high range water reducers on concrete bleeding when used to reduce water content. Mix C is the control and Mixes 1 to 4 contain HRWR admixtures inducing water reductions of 13, 10, 10, and 17 %, respectively. ASTM C 232 bleeding expressed in percent of mix water. Nominal cement content is 223 kg/m^3 and control water content is 157 kg/m^3 [15].

Table 5 illustrates the effect of plasticizers on increasing slump without influencing the water-cement ratio and also compares a high-slump versus low-slump control. At a constant water-cement ratio, the concretes with high-range water reducers have slightly more bleeding than the low-slump control, but significantly less bleeding than the high-slump control. At equivalent water-cement ratios, the flowing concretes with the plasticizers bleed more than the control, but significantly less than the control of the higher water-cement ratio and high slump [22].

Figure 31 illustrates the effect of plasticizers on concrete bleeding with two different cements used to make flowing concrete. All the concretes have the same nominal water and cement contents. The admixture was added to increase slump to between 175 and 225 mm. These mixes had more bleeding

TABLE 5—Bleeding of Concretes With and Without Plasticizers (ASTM C 232 Bleeding Test) [22]

Mix Identification	Water-Cement Ratio by Mass	Water Content, kg/m^3	Slump, mm	Bleeding, % by Mass of Mix Water	Bleeding, mL/cm^2 of Surface
Control 1	0.47	143	75	1.09	0.031
Control 2	0.58	171	215	3.27	0.143
Melamine sulfonate	0.47	143	215	1.59	0.060
Naphthalene sulfonate	0.47	144	225	1.50	0.059

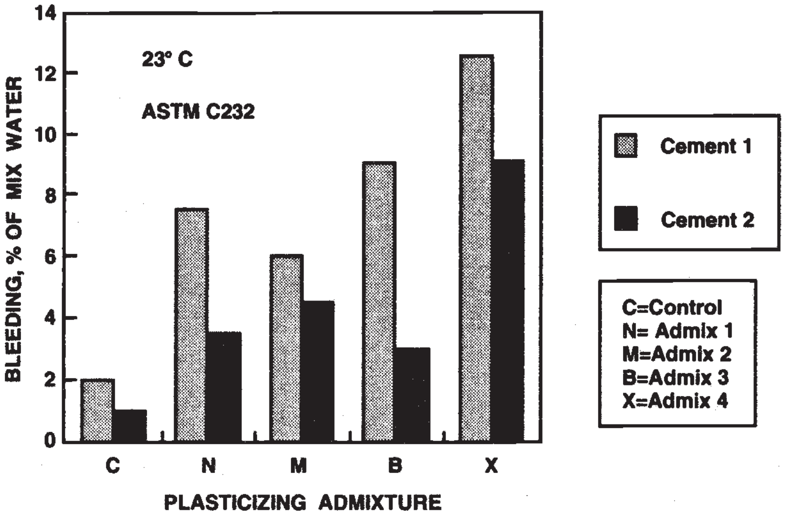


Fig. 31—Effect of plasticizers on bleeding of flowing concrete. The nominal cement, water, and air contents for all mixes were 323 kg/m^3, 161 kg/m^3, and 6 ± 1 %, respectively. The initial slump was 75 to 125 mm and, after the admixture addition, the slump was 175 to 25 mm. The increased fluidity increased bleeding. Mix C is the control and Mixes N, M, B, and X contain different plasticizers [12].

than the flowing concrete in Table 5 because of a higher initial water content. Figure 31 demonstrates that increased fluidity induced by the plasticizer increased bleeding and it demonstrates the effect of different cement and admixture combinations on bleeding. Excessive bleeding in plasticized, flowing concrete can reduce surface durability and deicer-scaling resistance [12]. Excessive bleeding can be avoided by optimal material selection and mixture proportioning.

Calcium chloride is commonly used as an accelerator in nonreinforced concrete. Sodium chloride and calcium chloride both can reduce bleeding significantly. The influence of accelerating salt such as calcium chloride may be largely due to the effect of its acceleration of setting. The effect of calcium chloride on bleeding time and bleeding capacity is illustrated in Table 6. Calcium chloride tends to make cement paste more susceptible to channeling. Little is known about how the different chemical compounds and chemical admixtures affect bleeding, as bleeding is not a required test in ASTM admixture specifications.

Special bleed-reducing admixtures based on cellulose derivatives, water absorbing resins, or various other chemical formulations can significantly reduce or eliminate bleeding. These admixtures make concrete mixtures very cohesive and thixotropic and therefore are very effective at preventing segregation. These admixtures are rarely used for normal construction but are very effective for special applications.

Effects of Placement Conditions on Bleeding

Placement Size and Height

The depth of a concrete placement directly affects the amount of bleeding. As shown in Fig. 32, the amount of bleeding increases with sample depth. This phenomenon has been observed especially in placements of low-cement-content concrete mixtures in deep dam sections, where many centimetres of water can accumulate over a lift just a few metres deep.

The shape of vertical forms can produce sufficient strain in the plastic concrete to induce cracks or zones of weakness

TABLE 6—Effect of Calcium Chloride on Bleeding Rate and Bleeding Capacity [4]^a

ASTM C 150 Cement Type	Admixture Addition	Bleeding Time, min	Bleeding Rate, 10^6 cm/s	Bleeding Capacity, ΔH
I	none	71	163	0.085
	$CaCl_2$	38	146	0.040
II	none	71	139	0.072
	$CaCl_2$	46	103	0.039
III	none	46	129	0.049
	$CaCl_2$	26	89	0.020

[a] Amount of $CaCl_2$ was 1 % of the cement mass and about 2.1 % of water. The water-cement ratio was 0.466 by mass.

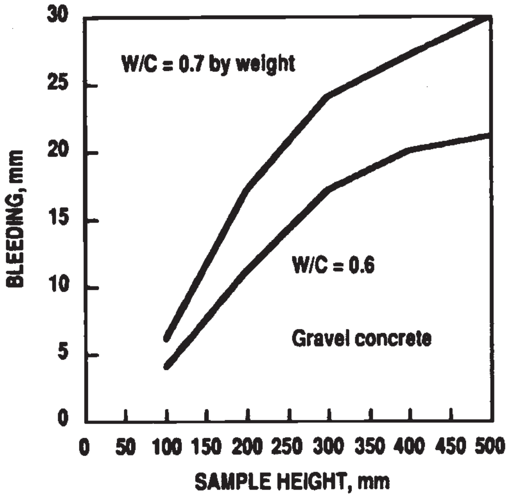


Fig. 32—Relationship between the amount of bleeding and sample height for concrete [10].

due to bleeding and settlement. Deep and narrow placements in which the walls are not parallel are more prone to settlement strain. These situations can create stresses within the plastic concrete upon settlement and bleeding. For example, assume that the settlement along a vertical face is constant. The settlement along an inward incline would be less than that along the vertical face. This would create some possible movement from one side of the form to the other, inducing shear strain and possibly faults to form depending on the angle of the slope, distance between the wall faces, the depth of the placement, rate of filling, and bleeding capacity of the concrete. The opposite situation occurs when the concrete particles settle away from an outward incline where a layer of water can develop and collect along the sloped surface. Such conditions can create localized bleed channels along the form resulting in sand streaks.

Settlement can also form zones of weakness in forms that have areas of significantly reduced cross section. For example, if a T section is filled in one placement, faulting can develop up from the corners of the narrow form. Obviously, the way to avoid this problem is to fill the narrow portion first and then place the rest of the concrete after the concrete in the narrow section has settled. The greater the bleeding capacity of the concrete the greater the tendency of such faults and arches to form. These faults can be eliminated by revibration.

Impermeable Sub-bases

Concrete placed on sub-bases of low permeability such as plastic sheet vapor barriers, bituminous, or clay appear to bleed noticeably more than concretes placed on granular sub-bases. All bleed water must rise to the surface when concrete is placed on an impermeable sub-base. When concrete is placed on a granular material, some of the bleed water can flow out the bottom of the slab into the granular material. Bleeding can be reduced by placing 50 to 75 mm of compacted granular fill on the impermeable surface. This practice also helps control slab curling. Construction in which only parts of a placement are on a vapor barrier will need special care in finishing practices due to nonuniform bleeding in order to provide a uniformly durable surface.

Weather Conditions

Weather conditions can have a significant effect on bleeding. Figure 33 illustrates how the increase in wind velocity and, consequently, rate of evaporation significantly increase the amount of surface subsidence. The wind velocity and increased evaporation greatly increase the capillary force at the surface, pulling the bleed water out of the concrete.

The bleeding rate increases with an increase in temperature; however, the bleeding capacity tends to be nearly constant. In a test on 25 different cements, going from 23.5 to 32°C resulted in a 20 % increase in the rate of bleeding for cement paste. This is primarily due to the decrease in water viscosity with an increase in temperature. In general, there was little change in bleeding capacity with the change in temperature for most of the cements studied [4].

Klieger illustrated that changes in temperature for concrete did not affect the subsidence of the surface [14]. At a wind velocity of zero for nonair-entrained concrete, the subsidence at temperatures of 10, 23, and 32°C was 0.502, 0.413, and 0.454 mm, respectively. The average subsidence was 0.456 mm for this concrete. Whiting found bleeding of concrete at 23 and 32°C to be 2 and 3.2 % by mass of mix water for normal concrete. The addition of plasticizers in some cases reduced bleeding at higher temperatures [12].

Consolidation and Revibration

Surface or internal vibration should not significantly affect the amount of bleeding. However, some studies indicate that a 2- to 3-s vibration period can slightly increase the bleeding capacity; however, the use of an internal vibrator in concretes from 20 s to 10 min tends to reduce bleeding capacity. This is partly due to the reduced volume of the matrix as well as the decreasing degree of dispersion of the aggregate and the expulsion of some of the entrapped air. Normal field vibration would not be expected to greatly affect bleeding [23].

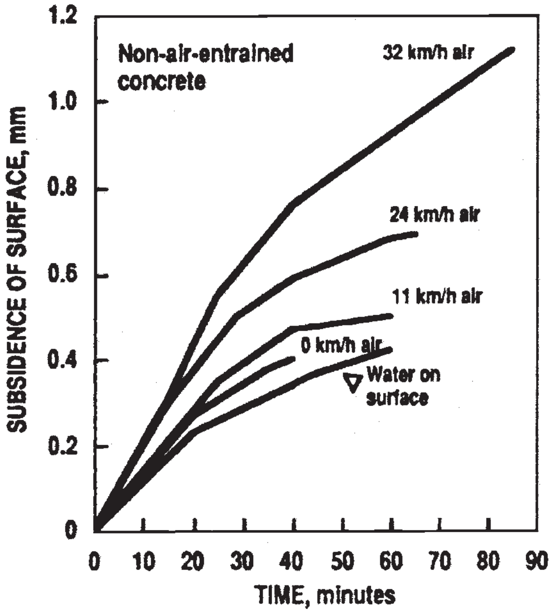


Fig. 33—Effect of wind velocity on subsidence [14].

TABLE 7—Effect of Revibration on Bleeding and Strength of Concrete [24][a]

Interval Before Revibration	Bleeding, % of Mix Water	Compressive Strength, MPa
0	2.9	28
1 h	3.5	32
2 h	3.4	39
3 h	3.2	31
4 h	2.8	29

[a] The cement was an ASTM C 150 Type I. The aggregate was natural sand and crushed trap rock with a maximum size of 25 mm. The cement content was 307 kg/m^3. The slump of the concrete was 75 mm. Compressive strength was estimated by impact hammer.

After concrete has completed its settlement, it can bleed and settle again upon internal revibration of the concrete. This is illustrated in Table 7 where consistent degrees of additional bleeding were obtained for revibration intervals up to 4 h. Also note that the compressive strength was also increased. This would be expected as removal of some of the bleed water would have resulted in a lower water-cement ratio [23].

Remixing

Just as revibration allows bleeding to occur after concrete is revibrated, remixing of the cementitious system after it has settled has a similar effect with little change in bleeding characteristics. Table 8 illustrates that particular cement pastes remixed within the first hour bleed about the same amount as before remixing with only slightly reduced bleeding rates and capacities compared to the first settlement. This demonstrates that concrete in a ready mixed truck for some period of time can, after remixing, resume its normal bleeding characteristics.

Controlling Bleeding

Usually, little attention need be given to controlling bleeding of concretes made of normal ingredients at normal proportions. However, this section will provide some guidance as to adjusting the bleeding to solve particular problems. Limits on the amount of bleeding are not available; however, acceptable bleeding rates can be determined by test and correlated to field experience.

Increasing Bleeding

The easiest way to increase the bleeding is to increase the amount of water in the concrete mixture as well as reduce the amount of the fines in the sand and amount of cementing materials. Bleeding may need to be increased to help prevent plastic shrinkage cracking or to improve a concrete's ability to be finished in dry weather.

Reducing Bleeding

Bleeding may need to be reduced for a variety of reasons, including to facilitate finishing operations, minimize the formation of weak concrete at the top of lifts, reduce sand streaking in wall forms, minimize bleed-related corrosion in grouted tendons, or to stabilize the hardened volume with respect to the plastic volume of the concrete. The most important ways of reducing bleeding in concrete are as follows:

1. Reduction of the water content, water-cementitious material ratio, and slump.
2. Increase the amount of cement resulting in a reduced water-cement ratio.
3. Increase the fineness of the cementitious materials.
4. Increase the amount of fines in the sand.
5. Use supplementary cementing materials such as fly ash, slag, or silica fume.
6. Use blended hydraulic cements.
7. Use chemical admixtures that reduce water-cement ratio or by other means are capable of reducing the bleeding of concrete.
8. Use air-entrained concrete.

Removing Bleed Water

Once the observation of bleed water has become a noticeable problem, there is little time or opportunity to change the concrete mixture, especially once the concrete is in the forms. Therefore, certain techniques can be used to remove excess bleed water from a concrete surface if normal evaporation is not satisfactory. If the concrete is placed within an enclosure, the temperature of the air can be increased and large fans can

TABLE 8—Bleeding Tests on Remixed Pastes [5][a]

Rest Period,[b] min	Bleeding Rate, 10^{-6} cm/s	Bleeding Capacity, ΔH	Duration of Bleeding, min	Time Between Initial Mix and End of Bleeding, min
0	194	0.122	55	55
15	189	0.113	55	70
30	196	0.125	57	87
45	192	0.106	52	97
60	185	0.103	48	108
90	172	0.090	47	137
120	167	0.075	45	165

[a] The water-cement ratio was 0.469 by mass. The schedule for the initial mixing was: 2 min mix, 2 min wait, followed by 2 min mix. The final remixing was 30 s continuously. The depth of the paste was 36 mm. The temperature was 23.5°C. The results for remixed pastes are averages of two or three tests.

[b] This is the period after the initial mixing, at the end of which the final remixing was done.

be used to evaporate away some of the water. However, care must be taken when using this technique to not remove so much bleed water that the evaporation exceeds bleeding, resulting in potential plastic shrinkage crack development.

Vacuum dewatering can be accomplished by special equipment that uses a filter mat placed on the surface of the concrete. As the mat settles with the surface, bleed water is forced up through the filter and the water is removed by a vacuum pump. Because this method consolidates a slab of concrete more than would normally occur due to normal bleeding, the end product is a concrete with a higher strength, lower permeability, and a more abrasion-resistant surface. For vertical applications such as wall forms, special liners can be placed in the form prior to concrete placement that uniformly drain away the bleed water without the formation of sand streaking.

For normal concrete slabs experiencing occasional excessive bleeding that is not removed due to evaporation, a squeegee or garden hose can be used to drag the water off of the surface so that finishing can commence. Finishing should continue only after it has been ascertained that bleeding has nearly or completely stopped. If a finisher waits too long for the bleed water to evaporate away, the concrete can harden and will not be able to be finished. For precast concrete elements experiencing settlement or bleeding problems, a centrifuge can be used to compact the concrete and remove the bleeding by centrifugal force.

Test Methods

Three standard ASTM test methods can be used to analyze the bleeding properties of concretes, mortars, pastes, and grouts. These are ASTM Test Methods for Bleeding of Concrete (C 232), ASTM Test Method for Bleeding of Cement Pastes and Mortars (C 243—discontinued in 2001), and ASTM Test Method for Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory (C 940). Another related ASTM standard is ASTM Test Method for Water Retentivity of Grout Mixtures for Preplaced-Aggregate Concrete in the Laboratory (C 941).

ASTM C 243 is based on a test method published in 1949 [25]. The ASTM C 243 test apparatus is shown in Fig. 34. The sample container is filled with mortar or paste. The collecting ring is then placed to penetrate the sample. The burette and funnel are filled with carbon tetrachloride or 1,1,1-trichloroethane, which are liquids that are denser than water. As the sample settles and bleed water rises through the carbon tetrachloride or 1,1,1-trichloroethane to the top of the burette, bleeding measurements are taken at regular intervals until settlement stops. Bleeding rate is reported as cubic centimetres of bleed water per square centimetre of sample surface per second. Bleeding capacity is reported as cubic centimetres of water per cubic centimetres of paste or mortar.

ASTM C 232 has two procedures—one with vibration and one without. Method A allows the concrete to be undisturbed in a container with a volume of about 0.014 m^3 and the accumulation of bleed water is removed from the surface (Fig. 35). Method B uses a similar sample and apparatus, along with a clamped-on cover, with vibration to help consolidate the concrete mixture. The sample is temporarily tilted when the water is drawn off to facilitate water collection. During the first 40 min, water readings are taken every 10 min and every 30 min thereafter until bleeding stops. Bleeding is reported as millilitres of bleed water per square centimetre of surface, or as a percent of the mix water.

ASTM C 940 is used to test the bleeding of grout, where 800 mL of grout are placed into a 1000-mL graduated cylinder and the cylinder is covered to prevent evaporation. The upper surfaces of the grout and bleed water are recorded at 15-min intervals for the first hour, and hourly thereafter until bleeding stops. The final bleeding is recorded as the amount of bleed water decanted as a percentage of the original sample volume.

ASTM C 941 is used to determine the water retentivity of grout under vacuum atmosphere. The test essentially determines the relative ability of grout to bleed, or retain its water, under pressure.

Several nonstandard test methods have also been used for analyzing the bleeding properties of concrete. One of the first was the Powers Float Method (Fig. 36). The float method was used to measure settlement of bleeding by measuring the subsidence of the small area at the top of the sample. The float consisted of a disk of lucite or bakelite to which a straight glass fiber was mounted like the mast on a sailboat. The disk had a diameter of about 13 mm and a thickness of 3 mm. Movement of the float was measured with a micrometer microscope. Because of the thixotropic structure of the cement paste, the float remains in a relatively fixed position at the top of the paste during settlement. A layer of water is placed on the surface immediately after the float is installed to prevent the development of capillary forces from evaporation. The water depth is about 6 mm deep. The float is used both for paste, mortar, and concrete samples. However, for concrete, the sample diameter was at least 500 mm. The float method was used in most of the early major findings on the bleeding properties of paste and concrete. This test method was used in most bleeding research at the Portland Cement Association in the 1930s to 1950s.

Special Applications

Under some conditions, it is desirable to eliminate the bleeding capacity as much as possible. These are usually situations in which the surface level of the plastic volume of concrete, mortar, or grout must be maintained. This would include, for example, the construction of superflat floors where nonuniform bleeding would interfere with surface tolerance.

In other applications, grout for preplaced-aggregate concrete must have a low level of bleeding in order to provide adequate strength development within the entire mass. Grout used in post-tensioning ducts must minimize the formation of voids due to bleed water in order to prevent corrosion of tendons within the duct. Grout placed under base plates for machinery or column applications must not allow bleed water to form voids. As the grout provides the support for these elements, bleeding must essentially be eliminated. Certain admixtures can be added to grout to prevent such bleeding and actually induce a small amount of expansion to help eliminate bleed-water voids. Often, priority grouts are used for grouting base plates [8].

Mathematical Models

Ever since Powers's work in the 1930s on bleeding, several researchers, including Powers, developed several mathematical models for the bleeding rate and bleeding capacity for paste, mortar, and concrete. Many of these relationships, pertaining to the permeability of the paste, were based on Darcy's law and Poisseuille's law. Stoke's law was also used in empirical rela-

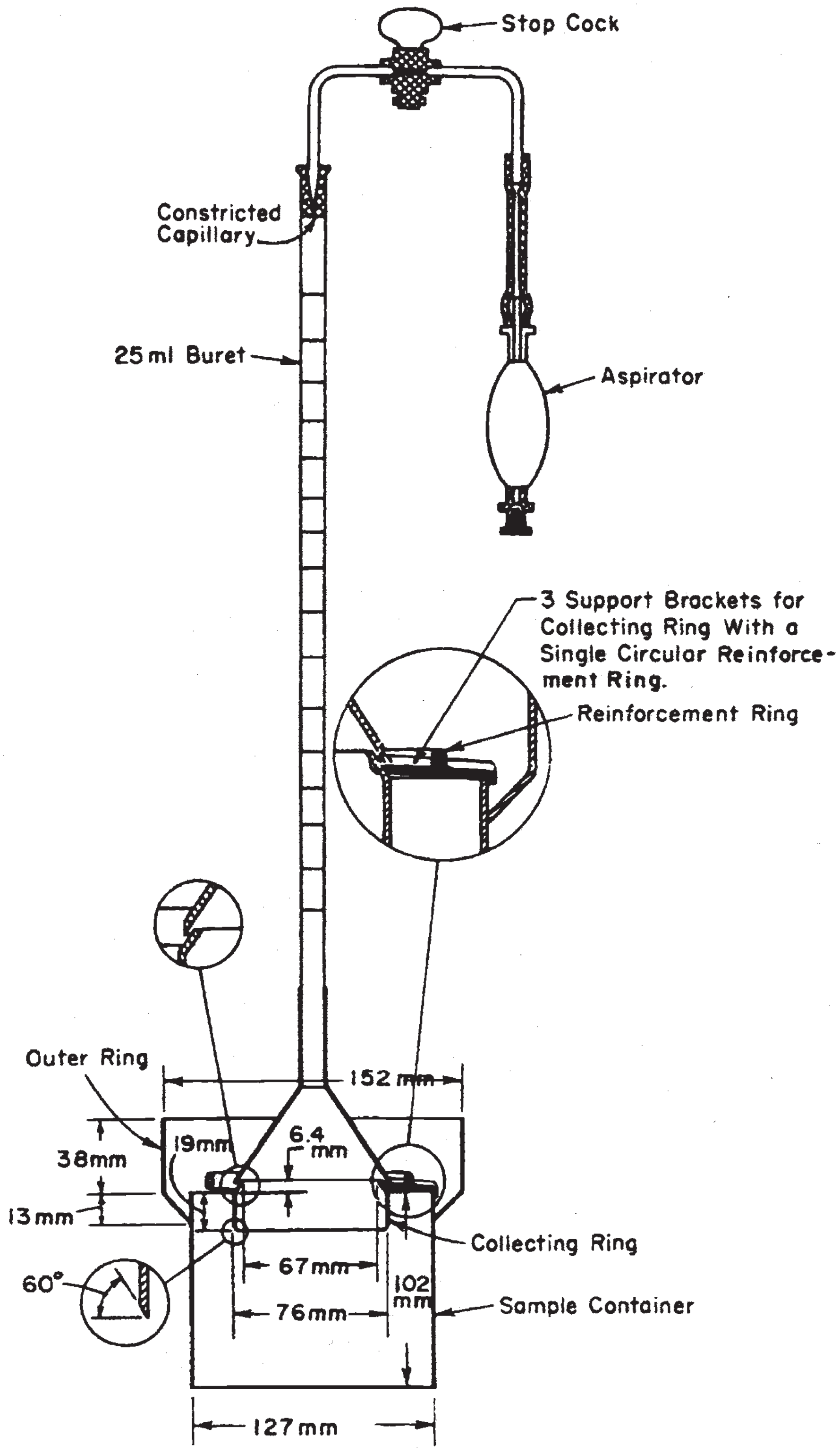


Fig. 34—ASTM C 243 test apparatus to determine the bleeding properties of paste and mortar.

Fig. 35—ASTM C 232 test for bleeding of concrete; Method A without vibration. The container has an inside diameter of about 255 mm and a height of about 280 mm. The container is filled to a height of about 255 mm. The container is covered to prevent evaporation of the bleed water.

tionships. Based on Poisseuille's law of capillary flow, Powers and Steinour developed equations relating to the initial constant bleeding rate of paste. Unfortunately, the bleeding rate diminishes with time. In addition, the early equations to determine bleeding capacity assumed that the bleeding process was not influenced by hydration of the cement.

As opposed to Powers's consideration of bleeding as merely sedimentation, Tan et al. [26] used a self-weight consolidation model to express the relationship between the different characteristics of the bleeding process. By using the consolidation model, Tan et al. were able to obtain very good agreement with the bleeding process, even beyond the period of initial constant bleeding rate.

Järvenpää [32] developed a bleeding model that considers mix proportions, paste content, aggregate shape, aggregate porosity, aggregate surface area, fines surface area, and admixture effects.

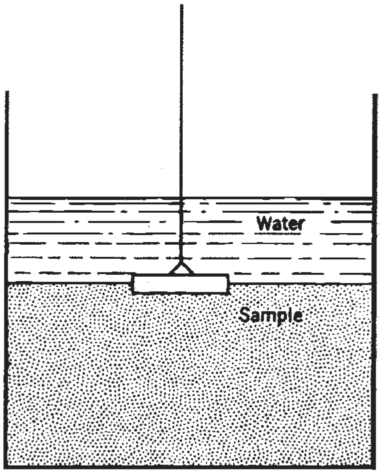


Fig. 36—Powers float test for measuring bleeding.

Conclusions

Bleeding is a fundamental property of concrete. By understanding its influences on plastic and hardened concrete properties and by understanding the effects of ingredients and ingredient proportions on bleeding, bleeding can be economically controlled. With proper control, bleeding should not hinder concrete construction or adversely influence concrete strength or durability.

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PART III
Hardened Concrete

13

Concrete Strength Testing

Celik Ozyildirim[1] *and Nicholas J. Carino*[2]

Preface

THE TOPICS OF STRENGTH AND RELATED TESTING were covered in the previous editions of the *ASTM STP 169* series by C. E. Kesler and C. P. Seiss, "Static and Fatigue Strength" [1]; by C. E. Kesler, "Strength" [2]; by K. N. Derucher, "Strength" [3]; and by P. M. Carrasquillo, "Concrete Strength Testing" [4]. The current chapter augments the topics addressed by these previous authors by introducing new developments since STP 169C was published. This chapter provides information on the nature of concrete strength, preparation of test specimens, test procedures, significant factors affecting test results, strength relationships, nondestructive and in-place strength testing including maturity and temperature-matched curing, strength under combined stresses, and fatigue strength.

Introduction

The most common concrete property measured by testing is strength. There are three main reasons for this. First, the strength of concrete gives a direct indication of its capacity to resist loads in structural applications, whether they be tensile, compressive, shear, or combinations of these. Second, strength tests are relatively easy to conduct. Finally, correlations can be developed relating concrete strength to other concrete properties that are measured by more complicated tests. Caution should be exercised, however, when strength is used to estimate other properties based on empirical correlations. When a nonstrength property is of primary interest, that property should be measured directly. For example, when penetrability is of concern, a test related directly to penetrability should be conducted, rather than relying on a strength correlation that can be misleading.

Strength tests of concrete specimens are used for three main purposes:

- For research.
- For quality control and quality assurance.
- For determining in-place concrete strength.

In research, tests are used to determine the effects of various materials or mixture proportions on the strength of concrete. Strength tests are used to obtain reference values when other characteristics of concrete are being studied, such as abrasion resistance or strength of structural members. In construction, strength tests are conducted on specimens made from concrete taken at job sites, either to determine the adequacy of the mixture proportions developed for the particular job or to check for changes in strength that could indicate quality control problems for the concrete supplier or changes in ambient conditions. When appropriately cured in place, these specimens can also be used to determine when the concrete in the structure is sufficiently strong for application of construction loads, removal of formwork, or application of prestressing. In cases where the strength of the in-place concrete is in doubt, specimens may be cut from existing concrete placements and tested for strength, or nondestructive tests may be performed to estimate the strength from previously established correlations.

Unfortunately, concrete strength is not an absolute property. Results obtained by testing a given concrete will depend on specimen shape and size, specimen preparation, and the loading method. Therefore, existing standard test methods must be followed for reliable results.

Nature of Concrete Strength

This section discusses the nature of concrete strength and those factors involved in the fabrication of the specimens that influence the measured strength. Factors involved in the testing of concrete specimens are discussed in later sections.

Reaction of cement with water forms hardened cement paste, which binds together coarse and fine aggregate to form a solid mass. Hardened paste consists of poorly crystallized hydrates of various compounds, referred to collectively as gel, crystals of calcium hydroxide, unhydrated cement, and air voids [5]. Concrete can be considered as a two-phase composite, consisting of cement paste and aggregate, and its behavior under load is a result of the interaction of the two phases and the interfacial regions between them. Failure of concrete occurs as a result of the development of a network of microcracks that grow in length with increasing load to the point where the concrete cannot support further load. The coarse aggregate particles act as inclusions that can both initiate and arrest crack growth. The latter feature is beneficial in reducing the brittleness of concrete.

Before external load is applied to concrete, fine cracks exist in concrete at the interface of coarse aggregate and cement paste due to mechanical property differences and the occurrence of shrinkage or thermal strains. These pre-existing microcracks are responsible for the low tensile strength of

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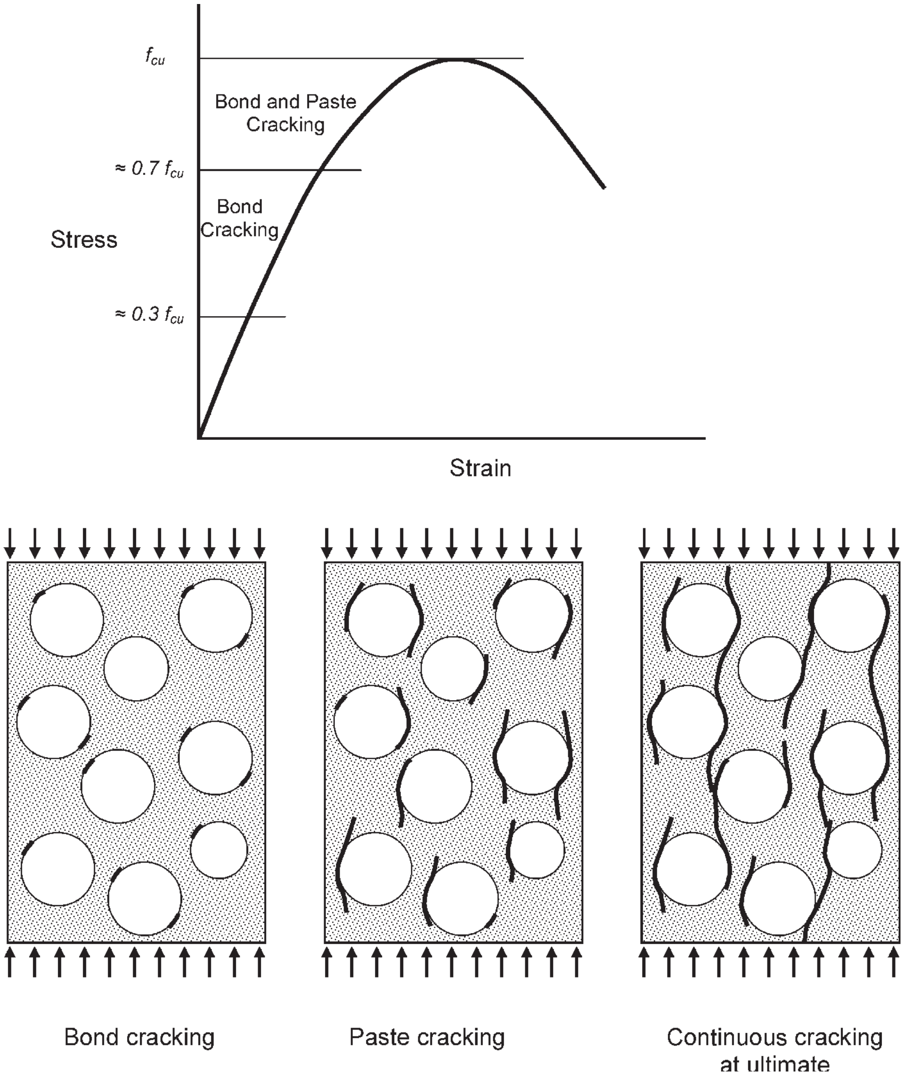

Fig. 1—Stress-strain curve of concrete in compression and stages of microcracking.

concrete. As external load is applied, existing microcracks are stable up to about 30 % of the ultimate load, at which point interfacial cracks begin to increase in length, width, and quantity. When 70–90 % of the ultimate strength is reached, cracks penetrate into the bulk paste leading to continuous larger cracks until the concrete cannot support additional load [5].

The shape of the compressive stress-strain curve of concrete is related to the formation and growth of microcracks. The process of microcracking and its relationship to the stress-strain curve is illustrated in Fig. 1. Up to about 30 % of the ultimate strength, the stress-strain curve is linear. When existing interfacial microcracks begin to propagate, the curve starts to deviate from linear behavior; deviation from linearity increases as more interfacial cracks are formed. When microcracks penetrate into bulk cement paste, deviation from linearity increases at a faster rate. As ultimate strength is approached, interfacial and bulk paste microcracks join to form continuous cracks parallel to the direction of loading. At some point, the extent of cracking is so great that the concrete cannot support additional load, and subsequently the stress required for additional strain decreases.

Based on the above microcracking process, it is clear that the ultimate strength of concrete is related strongly to the strength of the cement paste. Various factors affect paste strength. Probably the most important factor is paste density, which in turn depends highly on the water-cementitious materials ratio (*w/cm*). As the *w/cm* is increased, the density of the paste decreases, and so does its strength. The nature of the cementitious materials also affects paste strength. For example, the cement type affects paste strength, as well as its strength-gain characteristics. The use of ground granulated blast-furnace slag and pozzolans, such as fly ash and silica fume, affect both the paste strength and strength gain. Chemical admixtures can be used to alter paste performance; for example, water-reducing admixtures reduce paste viscosity (make it flow more readily), and other admixtures can be used to regulate time of setting and strength development. The entrainment of air without reducing the *w/cm* will decrease the strength due to the increase in voids in the paste. Finally, curing temperature and moisture conditions have a marked effect on the paste strength. High early curing temperatures, while producing higher early strengths, will reduce strengths at later ages due to the formation of less dense and nonuniform hydration products [5]. Further, hydration will continue only as long as free moisture is available for reaction with the unhydrated cement. Thus, if concrete is allowed to dry prematurely, hydration will cease before desired properties are attained. In poorly cured members, concrete may never reach the strength obtained from standard-cured cylinders at 28 days [6–8].

In most concretes, aggregate strength is higher than the concrete strength and does not adversely affect the strength of concrete. In some lightweight and high-strength concrete, however, aggregate strength may limit concrete strength. The strength of coarse aggregates is determined mainly by mineralogy and density. Beyond this, however, a smaller-sized aggregate may have strength advantages in that internal weak planes may be less likely to exist, and the smaller particles reduce stress concentration effects at paste-aggregate interfaces.

Factors involving both the paste and aggregate will affect the strength of the paste-aggregate interface. The bond between paste and aggregate particles will be stronger for smaller-sized aggregates, which have a higher surface area per unit volume. Also, a rough, angular surface texture such as exists in crushed aggregates increases interfacial bond strength [9]. The use of pozzolans such as fly ash or silica fume results in denser hydration products and increases the strength of the paste-aggregate interface [10,11]. Coatings, such as clay, on the aggregate surface reduce interfacial strength. When concrete bleeds, the rising bleed water is often trapped beneath coarse aggregate particles, thus weakening the interfacial zone.

Other factors that affect the strength of concrete include degree of mixing of the constituent materials and consolidation of the concrete. For a given set of materials, there is an optimum proportion of ingredients that yields the most economical mixture with the desired properties. Once the proportions have been selected, the materials must be mixed adequately to achieve a homogeneous mixture. The concrete must then be placed in the molds and thoroughly consolidated; this can be achieved through rodding or vibration. Inadequate consolidation will reduce strength. Self-consolidating concrete, however, does not require any consolidation effort during placement [12]. Excessive bleeding and segregation of fresh concrete can also lead to reduced strengths, especially in the top portions of test specimens, which will have a higher *w/cm* due to the rise of bleed water. The use of cardboard or plastic cylinder molds in preparing specimens has been reported to result in lower measured strengths compared with the use of rigid steel molds [13,14]. In practice, however, single-use plastic or cardboard molds are used widely for economic reasons. Also, concrete that has been damaged due to mishandling of specimens or inadvertent application of loads may also show reduced strength.

Preparation of Test Specimens

Concrete strength tests are conducted on both molded specimens and specimens cut from existing structures. Although testing procedures are similar, the significance of the information obtained can be quite different depending on specimen preparation and handling prior to testing. Following are brief descriptions of current standard procedures for preparing test specimens up to the time of testing.

Molded Specimens

Current ASTM test methods for measuring strength call for specimens in the shape of cylinders or beams. Preparation of these specimens in the field is governed by ASTM Practice for Making and Curing Concrete Test Specimens in the Field (C 31/C 31M), and under laboratory conditions by ASTM Practice for Making and Curing Concrete Test Specimens in the Laboratory (C 192/C 192M). ASTM Test Method for Compressive Strength of Concrete Cylinders Cast in Place in Cylindrical Molds (C 873) provides procedures for obtaining cylinders cast and cured in concrete slabs. The first two of these standards are discussed in detail in the chapter by Lamond in this publication. ASTM C 873 involves cylinders that are cast in special molds placed within a structural slab, so that the cylinders receive curing identical to that of the surrounding concrete in the structure until the time of test. Specimens made according to ASTM C 873 will be referred to as push-out cylinders.

ASTM C 192/C 192M specifies procedures for preparing test specimens in the laboratory and is generally used in research and mixture proportioning studies. This procedure is considered to be the “ideal” condition for specimen preparation, and will produce the most consistent results due to the high degree of control involved.

Specimens prepared in the field according to ASTM C 31/C 31M have multiple uses. Those given standard moist curing are used to check the adequacy of the mixture proportions as supplied to the job, monitor the producer’s quality control, and indicate changes in materials and other conditions [15,16]. Specimens given curing similar to that of the structural component are used to indicate the in-place strength of concrete prior to form removal or application of construction loads. Even though efforts are made to provide equivalent curing to both the concrete in the structure and the molded specimens, differences in strength are expected due to differences in consolidation and early-age temperature histories. Push-out cylinders (ASTM C 873) are also used to determine in-place concrete strength, but the curing of push-out cylinders is more like that of the structure than for molded specimens stored on the structure according to ASTM C 31/C 31M. Push-out cylinders typically have length-diameter ratios (L/D) less than the standard value of 2, and a strength correction factor has to be applied to the measured strength, as is done for cores.

Another technique to estimate the in-place strength is to use the temperature-matched curing (TMC) technique [17]. In a temperature-matched curing system, thermocouples inserted into the concrete mass monitor the temperature rise as it cures and control the temperature of specimens placed in a water bath or in molds with heating elements. In effect, the ambient temperature surrounding the concrete specimens matches the temperature history of the concrete mass as it cures. Such a curing system could also be used to follow a pre-established temperature history of a structural element. It has been found that temperature-matched curing allows for the best estimation of in-place strength [18].

Specimens from Existing Structures

Procedures for obtaining strength test specimens from existing hardened concrete are specified in ASTM Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete (C 42/C 42M). Drilled cores can be tested either in compression or splitting tension, and sawed beams are tested in flexure. In addition to placing dimensional requirements on specimens, ASTM C 42/C 42M requires that test specimens be comprised of intact, sound concrete, as free of flaws as the particular construction will allow. The standard also specifies moisture conditioning before testing (to be discussed subsequently).

Generally, drilled cores or sawed beams are obtained when doubt exists as to the strength of the concrete as placed. This can be due to low strength test results during construction or signs of distress in the structure. Also, cut specimens are useful if strength information is required for older structures or if service loads are to be increased above original design levels. All other factors being equal, the strength of these specimens is most likely to be representative of the strength of the concrete in the structure. Cutting of these specimens is, however, costly, and the drilling or sawing processes may introduce variables affecting strength test results.

Although the concrete in drilled or sawed specimens is more likely to be representative of that in the structure than molded specimens, one must be aware of various factors that are likely to affect the strength of the concrete samples thus obtained. Excess voids in a particular concrete sample, due to poor consolidation, will cause strength reductions. Further, the process of drilling or sawing of specimens may cause some damage that may affect strength test results, and this factor may become more pronounced as the ratio of cut surface to specimen volume increases [19]. The resultant strength reductions have been reported to be greater in higher-strength concretes [20].

Besides the drilling or sawing processes, the location and orientation of the cut specimens in the structure will affect strength test results. Specimens cut from the top of a concrete placement have lower strengths than those cut from the bottom of the placement due to bleeding, segregation, and curing effects [5,14,19,21,22]. Bloem [22] reported that cores taken from the top of an 8-in. (203-mm) slab tested approximately 5 % weaker than those from the bottom when the slab was subjected to good curing. The difference increased to 15 %, however, when the slab was poorly cured. Studies on high-strength concrete [23] indicated no effect of core elevation on strength test results, which can be attributed to the fact that high-strength concretes generally exhibit little bleeding. Cores drilled horizontally have been shown to yield lower strengths than those drilled vertically [5,14,19,21]. As shown in Fig. 2, this is attributed to the alignment of weak interfacial regions parallel to the loading direction in cores drilled horizontally, due to accumulation of water under coarse aggregate particles due to bleeding.

The loading to which the concrete member has been subjected may also affect the measured core strength. Cores taken from highly stressed regions, where microcracking is likely to have occurred, have lower strength than those from unstressed regions [19,24].

The inclusion of reinforcing steel in the specimen is not permitted by ASTM C 42/C 42M since there are insufficient data to demonstrate the effect of embedded steel on measured strength. The effect of reinforcement on compressive strength is variable. The presence of reinforcement in the tensile region of flexural beams or in splitting tensile strength specimens has pronounced effects on measured strengths.

Moisture conditioning of cores affects measured strengths. Early work showed that specimens tested dry had higher strengths than cores with two days of moist conditioning prior to testing [22]. Recent work on the effect of moisture conditioning practices has shown similar results leading to changes in ASTM C 42/C 42M [25,26]. The moisture conditioning procedures adopted in 2003 are intended to preserve the moisture of the drilled core and to minimize the effects of moisture gradients introduced by wetting during drilling and specimen preparation. Cores are to be placed in sealed plastic bags or nonabsorbent containers after the surface drill water evaporates or within 1 h of drilling. If water is used for subsequent end preparation, the specimens are sealed again after surface drying. Cores are kept in the sealed condition for at least five days after being last wetted and before testing. Unless specified otherwise, cores are to be tested within seven days of drilling.

Compressive Strength Test Procedures

Compressive strength testing of molded concrete cylinders prepared according to ASTM C 31/C 31M or C 192/C 192M is specified in ASTM Test Method for Compressive Strength of Cylindrical Concrete Specimens (C 39/C 39M). Testing procedures for cores (C 42/C 42M) and push-out cylinders (C 873) also refer to ASTM C 39/C 39M for measuring compressive strength. All of these standards specify tolerances on specimen

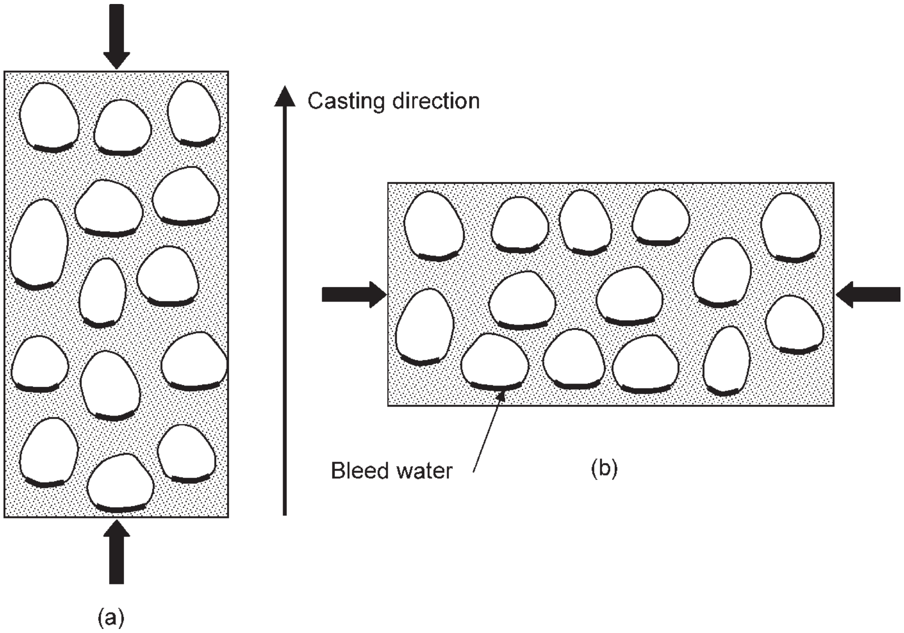

Fig. 2—Planes of weakness due to bleeding: (*a*) axis of specimen vertical and (*b*) axis of specimen horizontal.

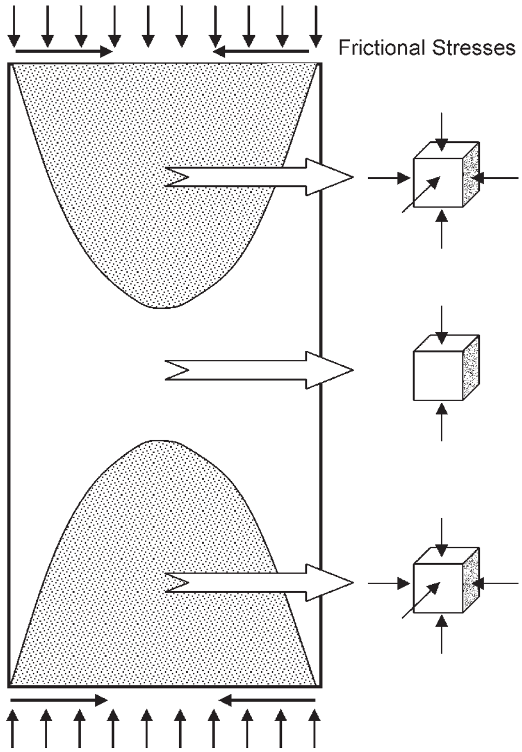

Fig. 3—Frictional restraint at the ends of cylinders results in state of triaxial compression shown as shaded region.

geometry, end conditions, and specimen moisture condition at time of testing. In addition, if the end condition tolerances are not met by the concrete specimens, the test methods require grinding or sawing of the ends to meet the requirements, or capping with bonded caps according to ASTM Practice for Capping Cylindrical Concrete Specimens (C 617) or with unbonded elastomeric caps (to be described) according to ASTM Practice for Use of Unbonded Caps in Determination of Compressive Strength of Hardened Concrete Cylinders (C 1231). All of these factors will influence measured strengths as discussed in following sections.

Ideally, the result obtained from a compressive strength test would be a direct indication of the concrete's ability to withstand a uniaxial compressive force. The stress state in standard compressive strength test specimens is, however, more complex than uniaxial compression. Friction between the bearing faces of the testing machine and the test specimen restrains the specimen laterally, thereby inducing lateral compression in the specimen ends as shown in Fig. 3 [5,14]. The portion of the cross section under triaxial compressions decreases with distance from the specimen ends. At distances from the specimen ends of about 0.8 of the diameter, the triaxial effects are negligible [5]. Thus, for cylindrical or prismatic specimens having an aspect ratio of two, cross sections at mid-height should be free of the effect of the end restraint. Details of this effect are covered in subsequent discussion of the effect of length-diameter ratio.

Factors Affecting Compressive Strength

In order to understand better the significance of the requirements of the test procedures, the effects of various factors on compressive strength test results are discussed.

Effect of Specimen End Conditions

ASTM C 39/C 39 M states that the ends of cylindrical specimens to be tested must not depart from perpendicularity with the specimen axis by more than 0.5° (approximately 1/8 in. in 12 in. or 1 mm in 100 mm), and that the ends must be plane to within 0.002 in. (0.050 mm). If the specimen does not meet these tolerances, the ends shall be sawed or ground to meet those tolerances, or capped in accordance with either ASTM C 617 or ASTM C 1231. ASTM C 31/C 31M and C 192/C 192M on specimen preparation allow for depressions or projections on finished surfaces of cylinders and beams of up to 1/8 in. (3.2 mm). Formed surfaces of beams are to be smooth and plane such that the maximum deviation from the nominal cross section shall not exceed 1/8 in. (3.2 mm) for cross-sectional dimensions of 6 in. (152 mm) or more, or 1/16 in. (1.6 mm) for smaller dimensions. ASTM C 42/ 42M allows projections of up to 0.2 in. (5 mm) from the core ends and requires that ends of cores do not depart from being perpendicular to the specimen axis by a slope of more than 1:8D or (1:0.3D), where D is the average core diameter in inches (or millimetres). The purpose of the latter requirement is to avoid exceeding maximum cap thickness specified in ASTM C 617.

The purpose of specifying end condition requirements of planeness and perpendicularity is to achieve a uniform transfer of load to the test specimen. Surface irregularities will lead to local concentrations of stress even in specimens that are capped to meet the planeness requirements [15]. The effect of cylinder end conditions prior to capping on strength test results has been reported by several authors [27–30]. In general, specimen ends that do not meet the specified requirements prior to capping cause lower strength test results, and the degree of strength reduction increases for higher-strength concretes.

ASTM C 617 covers procedures for capping with materials that bond to the cylinder ends. Freshly molded cylinders may be capped with a neat cement paste that is allowed to harden with the concrete. It is important to keep the cement paste caps moist, since they are susceptible to drying shrinkage and possible cracking. Hardened cylinders and drilled cores may be capped with either high-strength gypsum cement or sulfur mortar. The requirements on the strength of the capping material and the thickness of the caps for concrete strengths below and above 7000 psi (50 MPa) are given in ASTM C 617. Sulfur mortars are permitted if allowed to harden at least 2 h before testing for concrete with strength less than 5000 psi (34.5 MPa). For concrete strengths of 5000 psi or greater, sulfur mortar caps must be allowed to harden at least 16 h before testing, unless a shorter time has been shown to be suitable. The caps shall be plane to within 0.002 in. (0.05 mm), shall not depart from perpendicularity with the specimen axis by more than 0.5°, and shall not be off-center with respect to the specimen axis by more than 1/16 in. (2 mm). Generally, capping materials must be at least as strong as the concrete, unless there are data showing satisfactory performance even though the cube strength of the capping material is lower than the compressive strength of the concrete. This is possible because bonded caps are under a state of triaxial compression and can withstand higher stresses than the unconfined cube strength [31]. Caps on hardened concrete specimens should be approximately 1/8 in. (3 mm) thick, but no more than 5/16 in. (8 mm) thick, and well bonded to the specimen end. Thick caps can reduce the measured strength [32]. Unbonded caps have a thickness of 1/2 ±1/16 in. (13 ±2 mm] and the diameter is not more than 1/16 in. (2 mm) smaller than the inside diameter of the retaining ring. Required hardness of the pads depends on the strength level of the concrete, with higher-strength concrete requiring harder pads. The bearing surface of the retaining rings shall be free of gouges, grooves, or indentations.

The use of sulfur mortar is the most common bonded capping technique. Sulfur capping compounds must be kept molten at a temperature of approximately 265°F (130°C). These compounds have a range of melting temperatures, above and below which they become viscous and difficult to pour [15]. Further, volatilization of the sulfur occurs upon heating; thus, ASTM C 617 restricts the reuse of any material that has been used five times. Retrieval of capping material from specimen ends introduces oil and other contaminants that can reduce the fluidity and strength of the material.

Although bonded capping satisfies the end condition requirements for compressive testing, the capping operation is tedious and can be hazardous in the case of molten sulfur mortar. An alternative is the unbonded capping method covered by ASTM C 1231, which can be used for testing concrete with compressive strengths between 1500 and 12 000 psi. This system, which is illustrated in Fig. 4, consists of elastomeric pads, commonly made of neoprene, confined within metal retaining rings. The elastomeric pads conform under load to the specimen end surfaces, thereby distributing the applied load uniformly. The use of retaining rings is essential to restrict lateral flow of the pads that would otherwise induce lateral tension in the specimen ends, thereby reducing the apparent compressive strength. It has been found that the use of unbonded pads is a convenient and efficient capping method that produces test results comparable to those obtained with bonded caps [33,34]. The elastomeric pads can be reused until physical damage is observed. ASTM C 1231 includes information on the maximum uses of pads and the qualification of unbonded cap systems. Pairs of cylinders are made, and one is tested after grinding or capping while the other is tested with unbonded caps. At least ten pairs of cylinders are made at both the highest and the lowest strength levels for which the unbonded caps are to be used.

The measured cylinder strength of concrete is affected by the capping material when bonded caps are used. Traditionally, it has been believed that the resulting cylinder strength is related to the strength of the capping material. Research has shown, however, that the measured cylinder strength of a given concrete is related more closely to the elastic modulus of the capping material rather than strength [31]. The capping material must have a high elastic modulus to distribute the applied load uniformly to the ends of the specimen. Ideally, the elastic modulus of the capping material should be similar to that of the concrete. In a study of different capping materials [32], it was found that neat cement paste caps with cube strengths of 12 000 psi (85 MPa) and thicknesses up to ¼ in. (6 mm) exhibited strengths similar to specimens with ground ends for concrete strengths up to 17 000 psi (120 MPa). With sulfur mortar caps, however, reduction in strength was observed with 3/16-in. (5-mm) thick caps.

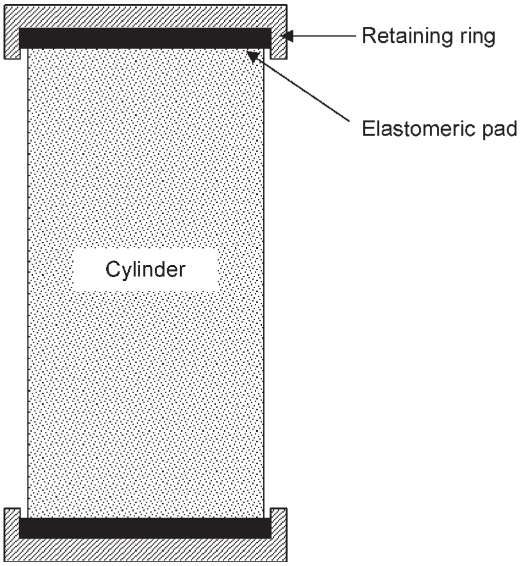

Fig. 4—Unbonded cap system for testing concrete cylinders in compression.

Effect of Specimen Size

Historically, the standard field molded cylinder has been 6 in. (152 mm) in diameter and 12 in. (305 mm) in length. The smaller 4 by 8 in. (102 by 203 mm) cylinder size is permitted when specified and provided that the nominal maximum size of aggregate (NMSA) does not exceed 1/3 the cylinder diameter. The 4 by 8-in. cylinder is being used more commonly. The smaller specimens require less material to make and are much easier to handle: a 6 by 12-in. (152 by 305-mm) concrete cylinder weighs about 30 lb (13.6 kg), compared with about 9 lb (4 kg) for a 4 by 8-in. cylinder. In addition, the use of concretes having increasingly higher strengths requires that testing machines have higher load capacities when 6 by 12-in. cylinders are used. Because 4-in. diameter cylinders have only 44 % of the cross-sectional area of 6-in. cylinders, high-strength concrete can be tested with existing testing machines. In cases of massive concrete placements, such as dams, the use of large aggregate sizes requires the use of larger-diameter specimens in order to maintain a diameter-aggregate size ratio of at least 3 to 1, or wet-sieving to remove larger aggregate sizes.

It is commonly accepted that as specimen size increases, the measured concrete strength and the variation in test results decrease [5,7,14,35–39]. As shown in Fig. 5, the magnitude of the size effect decreases with increasing specimen diameter [7]. The reasoning behind the size effect is that the strength of a concrete specimen will be governed by the weakest part of that specimen, and that the probability of the occurrence of large flaws increases as specimen size increases. Further, it has been reported that the strength difference due to specimen size increases as concrete strength increases [36]. Drilled cores will follow the same trend of increasing strength with decreasing specimen size for larger-diameter cores. For small diameter cores, however, the ratio of cut surface to specimen volume becomes significant, and it is possible that coring damage will cause strength reduction for decreasing diameters below 4 in. (102 mm) [19,40]. A review of available data indicated that 4-in. cylinders, on average, result in about 4 % higher measured strength compared with 6-in. cylinders [41,42]. It has been shown, however, that this strength difference can be reduced if 4-in. cylinders are molded by using two layers instead of three when consolidation is by rodding [43].

Although the testing of smaller specimens is more convenient, precision of strength determination should not be sacrificed. The within-test variability of 4-in. cylinders is about 20 % greater than that of 6-in. cylinders [42]. Equal precision of average strength can be obtained if the number

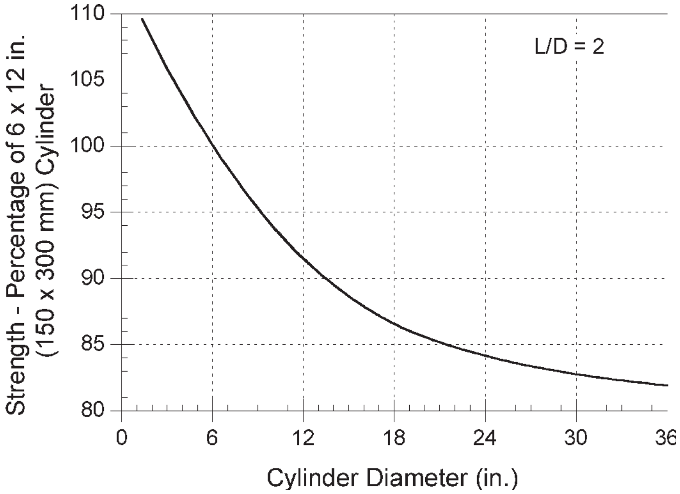

Fig. 5—Effect of cylinder size on measured compressive strength [7].

of 4-in. cylinders is increased to 1.5 times the number of 6-in. cylinders.

Effect of Diameter-Aggregate Size Ratio

Current specifications for molded specimens and push-out cylinders require that the minimum specimen dimension be at least three times the NMSA. Gonnerman [37] reported that test results were satisfactory for specimens with diameter-aggregate size ratio of two. He reported difficulty, however, in consolidating the specimens so that they were homogeneous, and thus recommended the minimum ratio of 3 to 1. For molded specimens, larger-sized aggregates may be removed by hand picking or by wet sieving so that smaller specimen dimensions may be used. It has been reported, however, that the practice of removing larger aggregate sizes from concrete will result in higher compressive strengths [44]. For drilled cores, the preferable condition is that the core diameter is at least three times the maximum nominal aggregate size used in the concrete placement. This condition may be relaxed by the specifier of tests provided the core diameter is at least twice the maximum size of coarse aggregate.

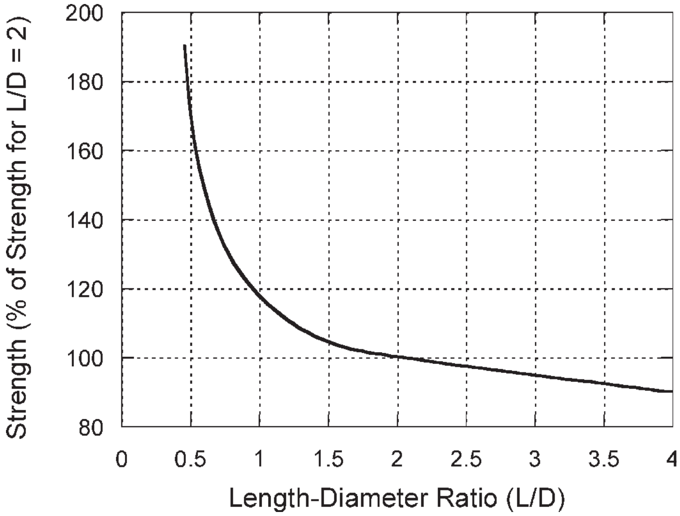

Fig. 6—Relationship between measured compressive strength and length-diameter ratio [7].

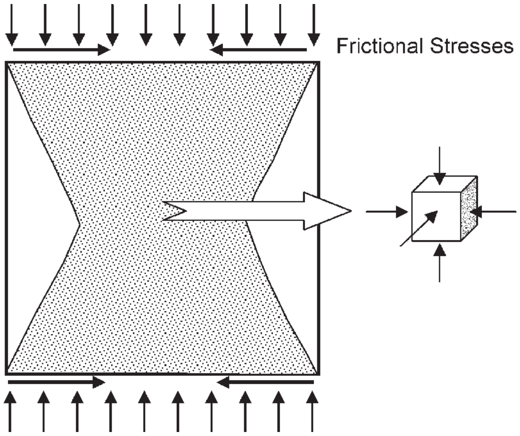

Fig. 7—Overlapping regions of triaxial compressive stresses in specimen with L/D = 1.

Effect of Length-Diameter Ratio

Standard test cylinders have a length-diameter (L/D) ratio of two, but L/D values of capped specimens as low as one are permitted by ASTM C 39/C 39M and ASTM C 42/C 42M. The actual length-diameter ratio will influence the apparent strength of the specimen being tested. As shown in Fig. 6, measured strengths increase as the L/D value decreases [5,14,37,45–47]. This is attributed to the effect of the end restraint due to the friction between the testing machine bearing blocks and the test specimen ends. As L/D decreases, a greater proportion of the specimen is in a state of triaxial compression. For example, Fig. 7 illustrates the conditions in a specimen with L/D equal to one. For L/D values between 1.5 and 2.5, measured strengths are within ±5 % of the strength for L/D = 2. For L/D values below 1.5, measured strengths increase markedly as shown in Fig. 6 [7].

ASTM C 39/C 39M and C 42/C 42M provide correction factors to be applied to strength test results obtained from specimens (molded or cores) having an L/D value between 1 and 1.75. These correction factors are shown in Fig. 8. According to these test methods, the correction factors are applicable to specimens of normal-density concrete, lightweight concretes with densities between 100 and 120 lb/ft^3 (1600 and 1920 kg/m^3), concrete that is dry or soaked at the

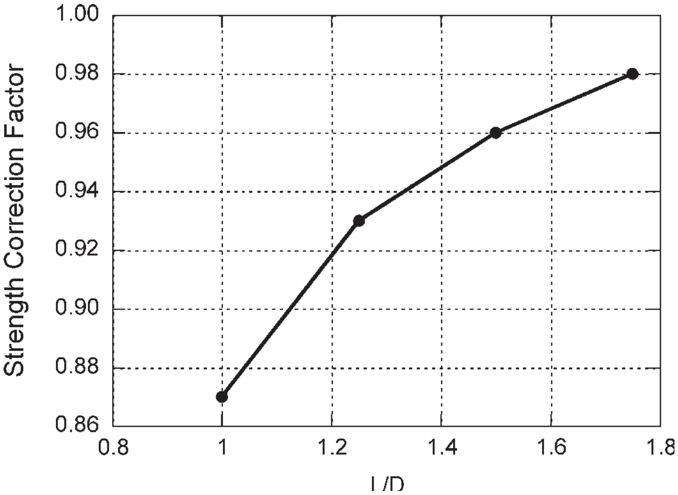

Fig. 8—L/D strength correction factors.

time of testing, and concrete strengths between 2000 and 6000 psi (14 and 42 MPa). For strengths higher than 6000 psi (42 MPa), correction factors may have larger values [48].

Effect of Specimen Moisture Condition

The moisture condition of the specimen at the time of testing can have a significant influence on measured strengths. In general, specimens have 5–20 % lower compressive strengths when tested in a moist condition than they would if tested in a dry condition [5,8,19,21,22,25,49]. The higher strength of dry specimens is attributed to increased strength of secondary bonds within the paste structure. It has been postulated that as a specimen dries, the outer surface attempts to shrink, thereby inducing lateral compression on the specimen interior, which increases its apparent compressive strength [49]. A specimen that is wetter in the outer region will have lower compressive strength.

When choosing the specimen moisture condition for testing, the purpose of the test must be considered, as well as the effect that the moisture condition will have on the test results. Standard-cured cylinders for acceptance testing are tested in a moist condition according to ASTM C 39/C 39M. On the other hand, field-cured cylinders and push-out cylinders are tested in their “as received” condition. In core testing, if results are to be compared with standard-cured cylinder test results, then a moist condition may be preferred; however, if determination of the in-place strength of the concrete is desired, the specimen should be tested as closely as possible in the moisture condition that exists in the structure. ASTM C 42/C 42M specifies moisture conditioning that preserves moisture of the drilled core and provides a reproducible moisture condition that minimizes the effects of moisture gradients [25,26] introduced by wetting during drilling and specimen preparation.

Effect of Loading Direction versus Casting Direction

Molded concrete cylinders are tested parallel to their casting direction. Beams and drilled cores, however, may be tested either parallel or perpendicular to the casting direction, depending on the circumstances involved. In general, specimens tested in the same direction as cast will yield higher strengths than those tested perpendicular to it. As illustrated in Fig. 2, the difference in measured strength is attributed to the occurrence of weak paste-aggregate interfaces aligned perpendicular to the casting direction due to water gain under coarse aggregate particles [5]. Cores tested parallel to the casting direction may have about 8 % higher strengths than those tested perpendicular to the casting direction [5].

Effect of Testing Machine Characteristics

ASTM C 39/C 39M gives required features for the loading apparatus to be used in compressive strength testing. Among these are the capacity for smooth and continuous load application; accurate load measurement; and two bearing blocks, one being spherically seated and one being solid, both of which must satisfy further requirements of surface planeness, minimum and maximum diameters, and other features. Failure to meet these requirements has been shown to reduce strength test results [16,30].

The spherically seated bearing block must be free to rotate to accommodate any small deviation from parallelism of the ends; strength reductions of up to 20 % have been observed with no spherical seating [30]. The spherically seated bearing block must not rotate when the specimen is being loaded, and ASTM C 39/C 39M prohibits the use of heavy grease to lubricate the ball-seat assembly. Placing the specimen off-center with respect to the loading axis by only ½ in. (13 mm) can cause strength reductions of 10 %. A flexible testing machine results in sudden failures of test specimens especially for high-strength concrete [42]. The longitudinal stiffness of the testing machine, however, does not appear to have a significant effect on measured strength [42].

Effect of Loading Rate

The measured strength of concrete specimens increases as the rate of loading increases [5,7,14,16,39,50,51]. Abrams reported that higher-strength concretes were more affected by loading rate, and that the ultimate strength was unaffected by rapid loading up to 88 % of the ultimate load followed by loading at a standard rate [51]. The dependence of ultimate strength on loading rate is thought to be related to mechanisms of creep and microcracking [5,14,39]. This would appear to be in agreement with the observation that when subjected to a sustained load of approximately 75 % of its ultimate capacity obtained using ASTM C 39/C 39M, concrete will eventually fail with no further load application.

ASTM C 39/C 39M requires that test specimens be loaded at a constant rate of movement between the bearing blocks. Prior to 2002, ASTM C 39/C 39M specified the following loading rates:

- For screw-type machines, approximately 0.05 in. (1.3 mm)/min running idle.
- For hydraulically operated machines, a loading platen movement that produces a stress rate between 20 and 50 psi/s (0.14 and 0.34 MPa/s).

Studies showed that at the extremes of the permissible range of loading rate, non-negligible strength differences could occur [42]. On average, the faster loading rate produced about 2.2 % greater strength. These findings lead to narrowing the loading rate range to 35 ± 7 psi/s (0.25 ± 0.05 MPa/s) in the 2004 version of ASTM C 39/C 39M. In addition, the 2004 version revised the requirements for screw-driven or servo-controlled loading machines. The user is required to establish the rate of platen movement that will result in the prescribed loading rate. This will require trial and error until sufficient experience is gained.

The designated rate of movement shall be maintained from about 50 % of the expected ultimate load until the ultimate load is attained. A higher rate of loading is not prohibited up to a load that is about 50 % of the expected ultimate load. The higher load rate shall be applied in a controlled manner so that the specimen is not subjected to shock loading. The loading rate should not be adjusted as the load approaches the ultimate strength.

Significance of Compressive Strength Test Results

The compressive strength of concrete is of primary importance in structural applications because design procedures require this property. As has been discussed, many factors affect the measured compressive strength of concrete test specimens. There are also differences between the conditions that exist within a structure and those within a test specimen. These include restraint, loading conditions, and long-term effects such as creep and shrinkage. Thus results from standard compressive strength tests may not represent in-place behavior. Tests of standard-cured specimens are used as a basis for quality control of batching, mixing, and delivery operations; determination of compliance with specifications; and for evaluating effectiveness of admixtures and other constituent materials.

Tests of field-cured specimens are intended to provide information on the in-place concrete. There are, however, limitations on the ability of field-cured test specimens to be representative of in-place concrete. Temperature-matched curing eliminates some of these limitations, but not all of them because molded specimens are not consolidated the same as in-place concrete.

Drilled cores provide representative samples of in-place concrete. Several factors, however, contribute to the uncertainty of measured core strength as being truly representative of the in-place strength. These factors include, among others, presence of moisture gradients resulting from water-cooled drilling or saw cutting of ends, undefined damage introduced by the core removal process, and differences in size and L/D value compared with standard molded specimens.

Tensile Strength Test Procedures

There are currently no standardized test procedures for determining the direct tensile strength of concrete, that is, the strength under uniaxial tension. This is due to the difficulty involved in inducing pure axial tension within a specimen without introducing localized stress concentrations. Knowledge of the tensile strength of concrete is important because it determines resistance to cracking. Therefore, several test procedures have been developed to indicate indirectly the tensile strength of concrete. These include:

- ASTM Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78)
- ASTM Test Method for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293)
- ASTM Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens (C 496)

Some agencies have adapted ASTM Test Method for Direct Tensile Strength of Intact Rock Core Specimens (D 2936) to measure direct tensile strength of concrete.

Flexural Strength Testing

ASTM C 78 and ASTM C 293 are similar in that they both involve testing a simply supported prismatic beam. For both, failure initiates in the tensile faces of the beams. As shown in Fig. 9, they differ, however, in the loading configuration. ASTM C 78 specifies application of load to the specimen at the third points along the span, resulting in maximum bending moment in the middle third of the specimen. ASTM C 293 specifies load application at midspan, which is the only cross section subjected to maximum moment.

For both test methods, the beam is to be tested on a span that is within 2 % of being three times its depth. Sides shall be at right angles to the top and bottom faces, and surfaces in contact with the loading or reaction blocks should be flat. The specimens should be tested on their sides as molded. If 1-in. (25-mm) or longer gaps in excess of 0.004 in. (0.1 mm) exist between the specimen surfaces and the loading or reaction blocks at no load, the specimen surfaces should be ground or capped. If the specimen surfaces are within 0.015 in. (0.38 mm) of being plane, leather shims may be used. The loading apparatus must apply load perpendicular to the face of the beam without eccentricity, and reactions must be parallel to the direction of load application. Further requirements are specified for the testing apparatus to ensure the supports and loading blocks are free to move to maintain uniform distribution of load over the beam width as tested. The first half of the load may be applied rapidly; afterwards, the loading rate is required to produce a tensile stress between 125 and 175 psi/min (861 and 1207 kPa/min) in the extreme fiber of the beam. The performance characteristics of fiber-reinforced concrete are determined using the flexural test. The load-deflection curve that is obtained can be used to calculate the residual strength or toughness at specified values of midspan deflection. Flexural testing of FRC is, however, discussed in the chapter by Tatnall in this publication.

Splitting Tensile Strength

ASTM C 496 gives requirements for the testing apparatus, test specimen geometry, and load application for determining splitting tensile strength of cylindrical specimens. The testing machine should meet the requirements of ASTM C 39/C 39M. The cylinder is placed on its side and subjected to a diametric compressive force along its length. If either the upper or lower

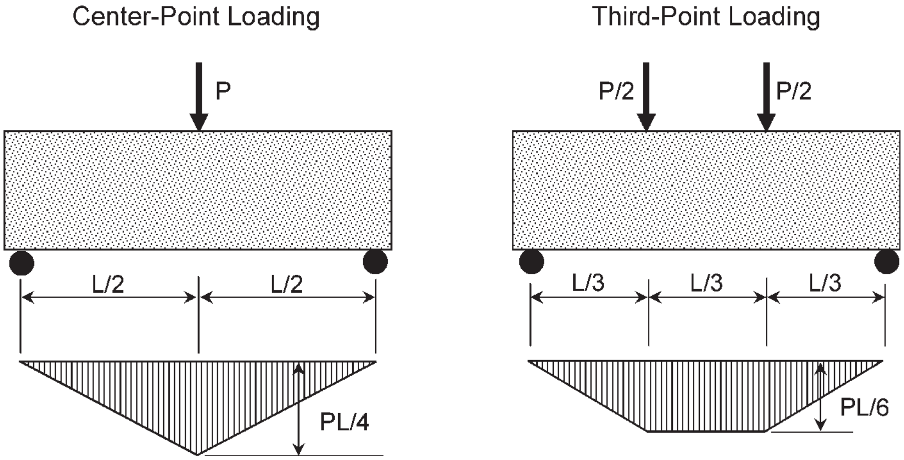

Fig. 9—Bending moment diagrams for center-point loading and third-point loading.

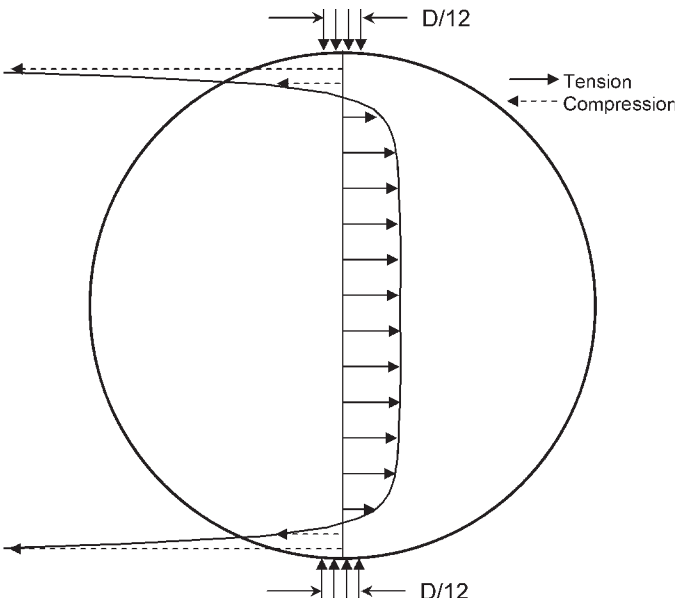

Fig. 10—Stress distribution on diametrical plane of cylinder for load distributed over a width of 1/12 cylinder diameter [52].

bearing block of the testing machine is shorter than the cylinder, a bearing bar or plate shall be used that is at least as thick as the distance from the edge of the machine bearing block to the end of the cylinder, at least 2 in. (51 mm) wide, and plane to within 0.001 in. (0.025 mm). Plywood bearing strips at least as long as the cylinder and 1/8 in. (3.2 mm) thick and 1 in. (25 mm) wide are placed between the specimen and the loading faces to accommodate minor surface irregularities. The load is applied so as to produce a spitting tensile stress rate between 100 and 200 psi/min (690 and 1380 kPa/min).

Failure of the cylinders occurs along a vertical plane containing the specimen axis and the applied load. As shown in Fig. 10, the loading configuration of this test method induces a nearly uniform tensile stress perpendicular to the vertical plane over approximately three-fourths of the specimen diameter. The actual stress distribution on the vertical plane is affected by the width of the uniformly applied load [52]. The value of the uniform tensile stress at failure is the splitting tensile strength and is approximated by the following equation:

$$f_{sp} = \frac{2P}{\pi L D} \tag{1}$$

where

f_{sp} = splitting tensile strength
P = maximum load during test
D = cylinder diameter
L = cylinder length

The regions of the specimen in the vicinity of the loading strips are subjected to large compressive stresses. Tensile rather than compressive failure occurs because the stress state at the loading strips is triaxial compression, allowing the concrete to resist the high compressive stresses [52].

Direct Tension Test

A proposed test method for direct tensile strength of concrete or mortar specimens is an adaptation of an existing standard test method for rock cores, ASTM D 2936. In it, cylindrical test specimens are loaded in axial tension through the use of metal caps bonded to the specimen ends. The direct tensile strength is determined by dividing the axial load at failure by the specimen cross-sectional area. Care is required during specimen preparation to ensure application of a tensile load with minimum eccentricity. This test procedure is not widely used for concrete specimens and ASTM Subcommittee C09.61 is not pursuing standardization at this time (2004).

Factors Affecting Flexural Strength

The formulas in ASTM C 78 and ASTM C 293 for computing flexural strength (modulus of rupture) are based on several assumptions that are approximations when testing concrete beams to failure [53]. One assumption is that the concrete behaves as a linear-elastic material throughout the test, which is not true at stresses approaching failure. The flexural stress equations apply to long, shallow beams, whereas the actual test specimens are short and deep. The failure stress calculated using the two test methods is higher than the actual extreme fiber stress due to the simplifying assumption that the stress distribution over the depth of the beam is linear. It is likely, however, that compared with the variability inherent to concrete strength, this approximation is not significant. Various other factors have been found to affect flexural strength test results obtained using either third-point or center-point loading. These will be discussed.

Effect of Specimen Dimensions

ASTM C 78 and ASTM C 293 require the test specimen to have a span of three times its as-tested depth. While the standard beam dimensions are 6 in. by 6 in. by 20 in. (152 mm by 152 mm by 507 mm), tested on an 18-in. (456-mm) span, other beam dimensions are permitted depending on maximum coarse aggregate size. The minimum specimen dimension must be at least three times the nominal maximum size of coarse aggregate, and the ratio of width to depth as-molded must not exceed 1.5. For specimens prepared in the laboratory, larger coarse aggregate particles may be removed by hand-picking or wet-sieving.

For a constant beam width and test span, the apparent flexural strength of specimens tested in third-point loading decreases as the depth of the beam increases; however, flexural strengths seem to be independent of beam width for a given depth and span [54]. For a constant beam cross section, the effect of span length is unclear. Kellerman [55] reported that strength decreased for both center-point and third-point loading as span length increased. Reagel and Willis [54], on the other hand, reported no effect of span length on strength for third-point loading.

Effect of Specimen Size

It is commonly agreed that as the size of the test specimen increases, flexural strengths decrease [54–57]. This has been found to be true for both center-point and third-point loading, and is due to the size effect as was discussed for compressive strength test specimens. Variability of test results decreases with increasing specimen size [57].

Effect of Coarse Aggregate Size

For the same *w/cm*, the flexural strength of concrete beams is higher for smaller coarse aggregate sizes. This was found to be true even when the use of the smaller coarse aggregate resulted in a mixture with a higher *w/cm* to maintain workability [55]. This effect should be considered when removing larger aggregate particles from concrete by hand-picking or wet-sieving. Variability of test results is lower with smaller coarse aggregate size.

Effect of Loading Rate

As with compressive strength, the apparent flexural strength of specimens increases with loading rate [56]. A linear relationship between flexural strength and the logarithm of applied stress rate has been observed.

Effect of Moisture Condition

Flexural strength test results are sensitive to the specimen moisture condition at time of testing. When specimens are tested in a drying condition, the apparent flexural strength is lower than when tested in a saturated condition [5,14,58,59]. Strength reductions of up to 33 % have been reported [58]. When the surface of the specimen is allowed to dry rapidly, it attempts to shrink, but this shrinkage is restrained by the specimen core. This induces tensile stresses in the surface and, if the induced tensile stresses approach the tensile strength of the concrete, cracks will develop in the outer surface of the specimen. When cracks exist due to drying, they act as stress concentrators and also reduce the effective cross section of the test specimen. If cracks do not develop, the tensile stresses in the near-surface concrete due to drying act as a preload condition; that is, the tensile stresses due to drying and those due to applied test load are cumulative, thereby resulting in a lower applied load to cause failure. The drying condition discussed occurs rapidly under normal ambient conditions if care is not taken to keep the specimen surfaces moist. Due to the significant effect of surface moisture condition on test results, flexural strength specimens that are used to indicate the strength of the concrete in-place should be cured under conditions similar to the concrete structure, but tested in a saturated condition. Note that the effect of drying on flexural strength test results is opposite to that for compressive strength test results.

Effect of Center-Point Versus Third-Point Loading

As was discussed previously, the main difference between ASTM C 78 and ASTM C 293 is the location of load application (refer to Fig. 9). In center-point loading (ASTM C 293), the load is applied at the specimen midspan. In third-point loading (ASTM C 78), the load is applied at the third points along the test span. For the latter case, the middle third of the beam span is subjected to maximum bending moment, and thus maximum extreme fiber stress. In center-point loading, however, only the cross section at midspan is subjected to maximum moment and maximum extreme fiber stress. The probability of having weak concrete in a region of highest stress is lower with the center-point loading compared with the third-point loading. As a result, for a given beam size, flexural strengths obtained from third-point loading are lower than those obtained from center-point loading [55–57]; differences of 15 % are not unusual. Variability is also lower for third-point loading.

Sometimes fracture of test specimens tested in center-point loading occurs at a location other than midspan. Since the bending moment distribution along the beam span varies linearly from zero at the support to its maximum at midspan, fracture at a location other than midspan corresponds to a lower extreme fiber stress than exists at midspan. Thus, when failure stress is calculated using the bending moment at the fracture plane, the flexural strength of the specimen is lower than when calculated using the bending moment at midspan [55,57]. ASTM C 293, however, does not require making note of the location of fracture. ASTM C 78, on the other hand, requires that if fracture occurs outside of the maximum moment region but within a distance of 5 % of the span length, the flexural strength is based on the bending moment at the location of the fracture plane. If fracture occurs outside of the maximum moment region by a distance of more than 5 % of the span length, the test results are to be discarded.

Factors Affecting Splitting Tensile Strength

In theory, the application of a line load perpendicular to the axis of a cylinder and in a diametrical plane produces a uniform tensile stress perpendicular to that plane. For actual testing of a concrete cylinder there are several departures from the theoretical case [52]. First, the theory applies to a homogeneous material, which concrete is not. Secondly, concrete is not linear elastic as is assumed in the analysis. Third, the load is not applied along a line, but rather is distributed within a strip. The strip loading results in large compressive stresses perpendicular to the diametrical plane, near the surface of the specimen under the loading strips. Nevertheless, the splitting tensile strength test is reasonably easy to conduct, and results provide comparative values, even if direct tensile strength values are not obtained. Several factors that affect splitting tensile strength test results are discussed.

Effect of Specimen Length and Diameter

For a given diameter, cylinder length does not seem to affect test results, other than possibly reducing variability for longer specimens [52]. Cylinders having a diameter of 4 in. (102 mm) were observed to have splitting tensile strengths that were roughly 10 % higher than those obtained from cylinders having 6-in. (152-mm) diameters [52,60]. Variability of test results decreases with increasing specimen diameter.

Effect of Bearing Strips

ASTM C 496 requires the use of bearing strips made of 1/8-in. (3-mm) thick plywood that are 1-in. (25-mm) wide and at least as long as the cylinder. The purpose of these strips is to conform to the specimen surface and distribute the load from the loading block. Increasing the thickness of the bearing strips may cause strength reductions [52]. Steel bearing strips cause significant strength reductions, probably due to their inability to conform to the specimen surface. Subcommittee C09.61 has rejected the use of alternative materials to 1/8-in. (3-mm) thick plywood unless comparative test data are provided.

Effect of Specimen Moisture Condition

It is not expected that drying of the specimen surface will affect the measured splitting tensile strength as significantly as it does flexural strength, since the specimen surface contained within the failure plane is subjected to high triaxial compressive stresses. Indeed, it is possible that the effect of drying is more similar to that in compressive strength cylinders, where the restrained shrinkage of the outer surface induces compression in the specimen interior.

Effect of Loading Rate

As with testing of specimens for compressive strength and beams for flexural strength, higher splitting tensile strengths are obtained when the specimens are loaded at a more rapid rate.

Relationship of Flexural Strength to Splitting Tensile Strength

In the flexural strength tests, failure is controlled by the strength of the concrete at the tension surface of the beam, but in the splitting tension test failure can be initiated anywhere in the portion of the diametrical plane that is in tension. Thus, based on the size effect principle, it is expected that splitting tensile strengths would be lower than flexural strength. This has been shown to be the case, with an average ratio of splitting tensile strength to flexural strength for center-point loading of 0.65 [60].

Significance of Tensile Strength Test Results

There is currently no standard test method for determining the direct tensile strength of concrete. Indications of tensile strength are obtained through the use of flexural and splitting tension test methods. Strengths obtained from each test method are particular to that method, and cannot be used interchangeably. The flexural strength test is more similar to the loading encountered in pavements than is the splitting tension test. The former, however, is more sensitive to moisture conditions, and thus more care must be taken to prevent beam surfaces from drying. The splitting tension test subjects the major portion of a diametrical plane to tensile stresses, and thus more closely resembles a direct tension test. When selection of a concrete tension test is being made, the loading of the concrete in the structure should be considered, as well as the expected use of the test results. If the strength is to be used in design calculations, the proper test must be performed to obtain the values required by the design formulas. For example, splitting tensile strength is used in the design of lightweight concrete beams to resist shear [61]. Even so, the results obtained are not direct tensile strengths, and may be more or less sensitive to factors that affect tensile strength. In addition, similar to compressive strength test limitations, conditions in the structure are much more complex than can be taken into account using strength tests on small specimens.

Also the variability of flexural strength test results is high due to sensitivity to details of fabrication, handling, curing, and testing. Thus flexural strength tests may be problematic for acceptance testing of concrete. As an alternative, a correlation between compressive and flexural strength can be developed for a given concrete mixture, and compressive strength testing can be used for control and acceptance of concrete, with flexural strength data used for reference only.

Strength Relationships

Due to the convenience of performing compressive strength tests, empirical strength relationships have been developed so that other strength properties may be estimated from results of compressive strength tests. The relationship between compressive and tensile strength has been found to be influenced by many factors, including concrete strength level, coarse aggregate properties, testing age, curing, air entrainment, and the type of tension test [5]. General relationships between the compressive strength of concrete cylinders and the strengths attained by various tension testing methods are discussed in the literature [5,7,8,14,39,62–64].

The splitting tensile strength follows a power function relationship of the compressive strength. As a result, the ratio of splitting tensile strength to compressive strength is not constant but decreases with increasing strength [65–68].

Nondestructive In-Place Strength Testing

The tests described thus far involve separately made specimens that do not necessarily give direct information about the actual concrete in the structure. Field-cured specimens (ASTM C 31/C 31M or ASTM C 873), cores, and temperature-matched cured specimens [69] are of some help in accounting for in-place conditions; however, such methods either require preplanning, use of specialized equipment, or cause localized damage to the structure. To avoid such limitations, a variety of in-place tests have been developed. Typically called "nondestructive," these tests either do not damage the concrete or result in superficial localized damage. Tests that cause no damage allow for retesting at the same, or nearly the same, location. Thus, changes in properties can be monitored over time. It is important to note that in-place strength tests do not provide direct measures of compressive strength, which means that relationships to other standard test results must be established [70]. Therefore, in-place tests should be accompanied by laboratory testing to develop the strength relationship before use in the field. These in-place tests are discussed in greater detail in the chapter by Malhotra in this publication.

A useful and relatively simple technique to estimate in-place strength is the maturity method, as described in ASTM Practice for Estimating Concrete Strength by the Maturity Method (C 1074). The maturity method provides an approach for making reliable estimates of in-place strength by establishing a relationship between concrete strength, age, and temperature. First, a strength-maturity relationship is developed by laboratory tests on the concrete mixture to be used. Then the temperature history of the field concrete is recorded from the time of concrete placement to the time when the strength estimation is desired. This temperature history is then used to calculate the maturity index of the field concrete. Finally, using the calculated maturity index and the strength-maturity relationship, the strength of the field concrete is estimated. The maturity concept can also be used to estimate later-age potential strength based upon the early-age strength of standard-cured specimens. This application is covered by ASTM Test Method for Developing Early-Age Compression Test Values and Projecting Later-Age Strengths (C 918) and is discussed in the chapter by Carino.

Another commonly used in-place test for strength is the pullout test described in ASTM Test Method for Pullout Strength of Hardened Concrete (C 900). In this method, a metal insert is either placed into fresh concrete or installed into hardened concrete. When the in-place strength is desired, the insert is pulled by means of a jack reacting against a bearing ring. The pullout strength is determined by measuring the amount of force necessary to pull the insert from the concrete mass. These pullout strengths can be related to compressive strength test results. Other in-place tests include the penetration resistance test described in ASTM Test Method for Penetration Resistance of Hardened Concrete (C 803) and the rebound hammer test described in ASTM Test Method for

Rebound Number of Hardened Concrete (C 805). The report of ACI Committee 228 provides guidance on the proper use of these types of in-place tests [70].

Strength Under Combined States of Stress

In structures, concrete members are often subjected to combinations of compressive, tensile, and shear stresses. Thus, it may be important to understand the behavior of concrete under different stress states. ASTM Test Method for Determining the Mechanical Properties of Hardened Concrete Under Triaxial Loads (C 801) provided a standardized method for determining such behavior. According to the test method, cylindrical test specimens are subjected to a lateral confining stress in combination with an axial load. Thus specimens are subjected to triaxial stress states such that two of the three principal stresses are equal. The test method is analogous to the triaxial test used for soil specimens. ASTM C 801 was withdrawn in 2004 because the method was not being used.

A common use of data obtained from triaxial compressive tests is to construct a Mohr failure envelope for determining shear strength as a function of normal compressive stress. The shear strength of the concrete is estimated by drawing a tangent to the Mohr's circles at failure obtained for various combinations of confining pressure and axial stress (see Fig. 11).

Triaxial states of stress are typically of significance in massive concrete structures. Biaxial states of stress are more common. The compressive strength of concrete subjected to biaxial compression is higher than its uniaxial compressive strength, and the tensile strength is not affected significantly by the application of biaxial tensile stresses [5,14,71]. Figure 12 is an example of a biaxial stress failure envelope in which stress combinations that lie within the envelope correspond to no failure. Research results have shown that the degree of confinement at specimen loading surfaces had a significant effect on biaxial stress test results, resulting in biaxial to uniaxial compressive strength ratios in the range of 1.1–3.5 [72]. Biaxial tests conducted using brush platens seem to have removed the effect of end confinement to a great degree. As determined with these end conditions, an average increase in compressive strength with biaxial compressive loading is about 30 %, and occurs at a principal stress ratio of approximately 0.5 [72].

Higher-strength concretes are becoming more common in construction. In biaxial compression tests performed on high-strength concretes, the increase in compressive strength under a biaxial stress state depended on the type of coarse aggregate [73]. As with normal-strength concrete, average strength increases of roughly 30 % above the uniaxial compressive strength were observed, ranging from a minimum of 26 % for trap rock coarse aggregate to a maximum of 34 % for granite coarse aggregate. In addition, it was noted that the proportional limit for high-strength concrete as well as the discontinuity point were at higher stress-strength ratios than for normal-strength concrete, and increased with increasing biaxial stress ratio. The discontinuity point is the stress at which unstable progressive microcrack growth begins. The failure modes of the specimens indicated that fracture was controlled by a limiting tensile strain perpendicular to the plane of loading.

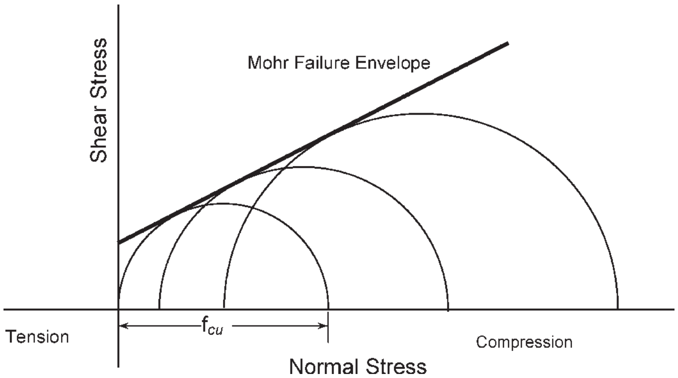

Fig. 11—Shear strength as a function of normal stress obtained from triaxial compression tests.

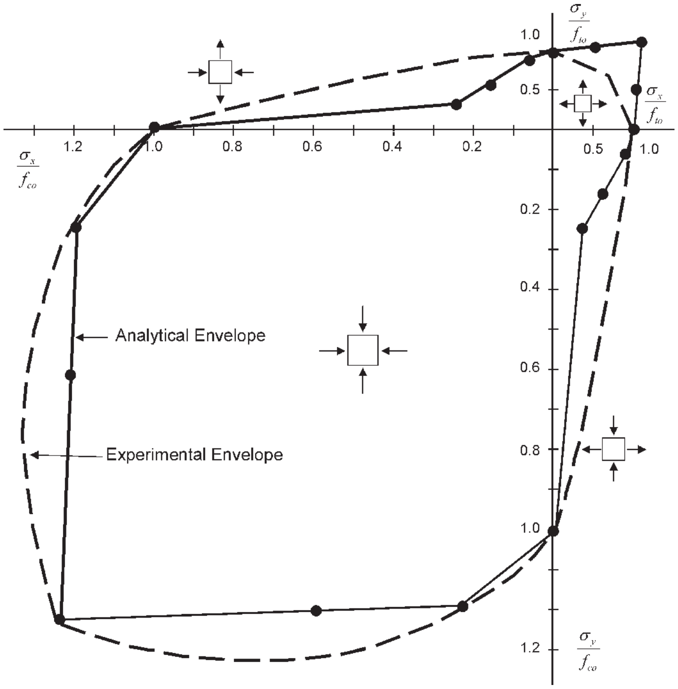

Fig. 12—Biaxial stress failure envelope for normal-strength concrete [71].

Calixto [74] performed biaxial tension-compression tests on high-strength concrete specimens. In general, the application of lateral tension caused a significant decrease in the axial compression capacity of the concrete, whereas the application of lateral compression had a much less pronounced effect on the axial tensile capacity. The increased capacity of concrete under a biaxial or triaxial compressive stress state is significant in that the lateral restraint provided in reinforced concrete structures through such means as steel spirals will enhance the capacity of the concrete. Lateral tension, however, causes a significant decrease in concrete's compressive strength. The higher stress-strength ratios required to initiate progressive, unstable crack growth in higher-strength concretes would seem to provide an additional safety margin in these concretes when subjected to the same sustained stress-strength ratios as for normal-strength concrete.

Fatigue Strength of Concrete

Fatigue strength is the greatest stress that can be sustained for a given number of stress cycles without failure. In numerous structural applications, such as bridge decks and pavements, concrete members are subjected to repeated applications of

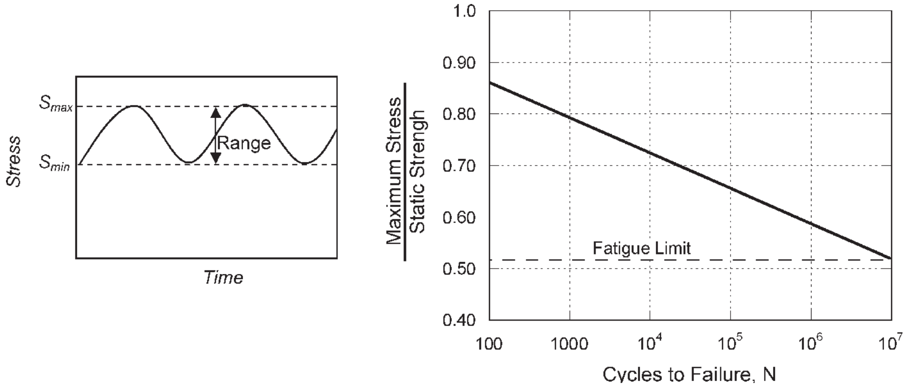

Fig. 13—Schematic of cyclic load history and fatigue strength curve of concrete.

load at a level below the ultimate strength of the concrete. Like most materials, concrete exhibits fatigue behavior; that is, when subjected to cyclic loading of a given level but below its short-term static strength, it will eventually fail. Thus, for structural design purposes, it is desirable to establish the response of concrete to fatigue loading. However, a standardized test method is not available.

Concrete does not have a fatigue limit, that is, a ratio of applied stress to static ultimate strength (stress-strength ratio) below which the concrete can withstand an infinite number of loading cycles [5,14]. Therefore, the fatigue limit of concrete is taken as the maximum stress-strength ratio at which failure occurs only after a large number of cycles, usually 10^7. In normal-strength concrete, the fatigue limit thus defined is approximately 55 % of its static ultimate strength.

The fatigue strength of concrete is sensitive to several factors [75], most of which are associated with the details of the applied loading. The number of cycles of loading to failure depends on the level of cyclic stress applied to the specimen. As shown in Fig. 13, as the stress-strength ratio is increased, the number of cycles to failure decreases [2]. Also, the range of maximum to minimum cyclic stress has an effect. As the difference between the two stresses increases, the number of cycles to failure decreases.

The sequence of loading cycles is also significant. Miner's rule, which states that the effects of cyclic loads are cumulative, does not strictly apply to concrete. Rather, the fatigue life, the number of cycles to failure, of a specimen is different if it is subjected first to high stress-strength ratios followed by low stress-strength ratios, than if subjected to the reverse sequence. Cycling below the fatigue limit increases both the fatigue strength and the static strength by 5–15 % [5,14,76]. When the frequency of load application is low, the fatigue life is shortened compared with a higher frequency of load application. This is probably due to mechanisms of creep and crack propagation. Rest periods of up to 5 min during fatigue testing also increase the fatigue life; increasing the length of the rest period beyond 5 min has no additional beneficial effect.

Specimens subjected to a stress gradient also exhibit enhanced fatigue life over those subjected to a uniform stress distribution, possibly because the lower stressed regions inhibit crack growth. Finally, specimens tested in a moist condition exhibit lower fatigue life compared with those tested in a dry condition.

In biaxial compression fatigue tests on high-strength concrete, Nelson et al. [75] reported that biaxial compression decreased the fatigue life for maximum stress levels below 76 % of the ultimate strength; the fatigue limit decreased with increasing biaxial stress ratio. Similar to normal strength concrete, the fatigue life of high-strength concrete increases with decreasing maximum stress-strength ratio and stress range, and wet specimens fail at less cycles compared with dry specimens. Cyclic loading of the specimens below the fatigue limit increased the uniaxial static strength on the order of 40 %. Whereas normal-strength concrete is reported to have a fatigue limit of approximately 55 % of its static ultimate strength, the corresponding value for high-strength concrete was found to be slightly lower, in the range of 47–52 %.

Structures such as bridge decks and pavements undergo numerous load cycles under normal service conditions. Thus, it is important to understand the response of concrete to cyclic loading. Over 100 references are given in the ACI report on considerations for design of concrete structures subjected to fatigue loading, including references on biaxial stress states and high-strength concrete [77]. Furthermore, since factors such as the magnitude of applied load, the stress state, loading frequency, duration of rest periods, and concrete moisture condition are largely unknown under actual service conditions of the structure, information obtained from laboratory studies can only give general information about a very complex phenomenon.

Summary

This chapter has reviewed the standard practices and test methods used to measure concrete strength. These standards are under the jurisdiction of Subcommittee C09.61. The discussion has stressed the limitations of the results obtained from tests on small specimens in estimating properties of the concrete in the structure. Nevertheless, with a thorough knowledge of their limitations and the factors that affect measured strengths, results obtained from correctly performed standard test procedures will provide useful information.

In concrete construction, strength tests have three main purposes: (1) research, (2) for acceptance of concrete and quality control by testing specimens subjected to standard-curing conditions, and (3) for evaluating in-place properties of concrete. While the tests discussed in this chapter appear to be simple, strict adherence to specified procedures and tolerances is necessary for obtaining meaningful results and ensuring reproducibility between laboratories. It is essential that the purpose of the test be defined, and that those factors that will influence test results be understood and controlled in accordance with the applicable standards.

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Prediction of Potential Concrete Strength at Later Ages

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Preface

THIS CHAPTER DEALS WITH METHODS FOR ESTImating the potential later-age strength of a concrete mixture based upon the compressive strength measured on cylindrical specimens at early ages. The results of the cooperative research program leading to the development of ASTM Test Method for Making, Accelerated Curing, and Testing Concrete Compression Test Specimens (C 684) are reviewed. This review is based on Chapter 13 of *ASTM STP 169B*, which was written by M. H. Wills, Jr., and was titled "Accelerated Strength Tests." The earlier text was augmented by the current author in *ASTM STP 169C* by the addition of information on the high-temperature and pressure-accelerated test method that was added to ASTM C 684 in 1989. In addition, the author expanded the chapter in *ASTM STP 169C* by including the basis of ASTM Test Method for Developing Early-Age Compression Test Values and Projecting Later-Age Strengths (C 918). This current version updates changes to ASTM C 684 and ASTM C 918 since *ASTM STP 169C* was published.

Introduction

Rapid construction practices throughout the concrete industry have caused specifying agencies to require assessment of concrete strength at an age earlier than the traditional 28 days after placement. Currently, the later age is still specified for compression tests for acceptance of concrete as delivered to the job site. During the 28-day period between the preparation and testing of the specimens, a multistory building could go up four or more floors. Many consider it precarious for construction to proceed in this manner. Furthermore, expensive and costly delays are encountered when 28-day test results are low, because a field investigation may be necessary to verify the load-carrying capacity of the structure. Further delay is certain if the structure must be strengthened or replaced. Early assessment of concrete quality is, therefore, essential for overall construction economy and safety.

Recognizing the need for alternative test methods for assessing the quality of concrete at early ages, ASTM Committee C9 on Concrete and Concrete Aggregates formed Subcommittee C09.02.09 on Accelerated Strength Testing in 1964. (In 1996, the subcommittee was discharged and jurisdiction of its standards was assigned to Subcommittee C09.61 on Strength Testing.) The subcommittee developed two methods for estimating the later-age strength of concrete specimens based upon early-age strength tests.

One of these methods, ASTM C 684, involves testing cylinders whose strength development has been accelerated by elevated curing temperatures. Depending on the specific procedure that is used, the accelerated strength is measured at ages ranging from 5 h to 48 h. A previously established relationship between accelerated strength and standard-cured strength is used to estimate the later-age strength under standard curing conditions based on the measured strength of accelerated test specimens.

The other method, ASTM C 918, uses the maturity method [1] and a previously established strength-maturity relationship to estimate later-age strength based on the early-age strength of specimens whose temperature history has been measured.

This chapter reviews the background of these two techniques and provides supporting information to the standard test methods to assist persons who are contemplating specifying these procedures in contract documents.

Accelerated Curing Methods

The first assignment for ASTM Subcommittee C09.02.09 was to investigate the needs of the concrete industry and to study the suitability of standardizing several accelerated testing procedures being developed by King [2,3], Akroyd [4], Smith and Chojnacki [5], Malhotra and Zoldners [6], and Smith and Tiede [7]. A positive response to this canvass led to a cooperative test program among nine laboratories to evaluate various procedures (Table 1) involving immersion of specimens in hot or boiling water or autogenous curing in an insulated container to accelerate strength development of concrete [8].

Experimental Program

Each of the accelerated testing procedures was conducted using ASTM Type I (or Type II meeting Type I specifications) and Type III portland cement. Cement contents were 265, 325, and 385 kg/m^3 (450, 550, and 650 lb/yd^3). Sufficient air-entraining admixture and mixing water were used to produce air contents

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TABLE 1—Accelerated Testing Procedures

Procedure	Description
A—Warm water	Cylinders placed immediately in water at 35°C (95°F)
B—Modified boiling	Standard curing for 23 h followed by boiling for 3.5 h
C—Final set and boiling	At final setting (ASTM C 403/C 403M),[a] cylinders boiled for 15 h
D—Autogenous curing	At 1-h age, place in insulated container for 46 h
E—Initial set and hot water, 55°C (130°F)	At initial setting (ASTM C 403/C 403M), place in hot water for 15 h
F—Initial set and hot water, 75°C (175°F)	At initial setting, place in hot water for 15 h
G—Initial set and hot water, 90°C (195°F)	At initial setting, place in hot water for 15 h

[a] ASTM Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance (C 403/C 403M).

from 5.0 to 6.0 % and slumps from 50 to 75 mm (2 to 3 in.). Concretes were made without a retarder or with a normal dosage of a water-reducing retarder. Fine and coarse aggregates were graded to a No. 57 size, that is, 25.0 to 4.75 mm (1 in. to No. 4). Each laboratory used materials available in their locality without interchange among laboratories. In all cases, compressive strengths of the accelerated specimens were measured at ages of one day or two days and were compared with strengths of standard-cured specimens at ages of 28 days and 364 days. Two replicate specimens were tested at each age, and their averages were used in subsequent analyses. All specimens were prepared, cured, and tested according to applicable ASTM standards. All materials conformed to appropriate ASTM specifications.

The nine participating laboratories were asked to conduct Procedures A, B, and C listed in Table 1. Additionally, they had the option of conducting one or more of Procedures D through G (Table 1). Despite the plans of the subcommittee, it was necessary to curtail some aspects of the experimental program. Procedure C, involving the measurement of final setting time followed by boiling, had to be abandoned because it required too much overtime work to conduct. Consequently, Procedures E, F, and G were also abandoned because they also required measurement of setting time and overtime work. Only four of the nine participating laboratories conducted Procedure D (Autogenous Curing), which was optional. However, all laboratories conducted Procedure A (warm water) and Procedure B (modified boiling).

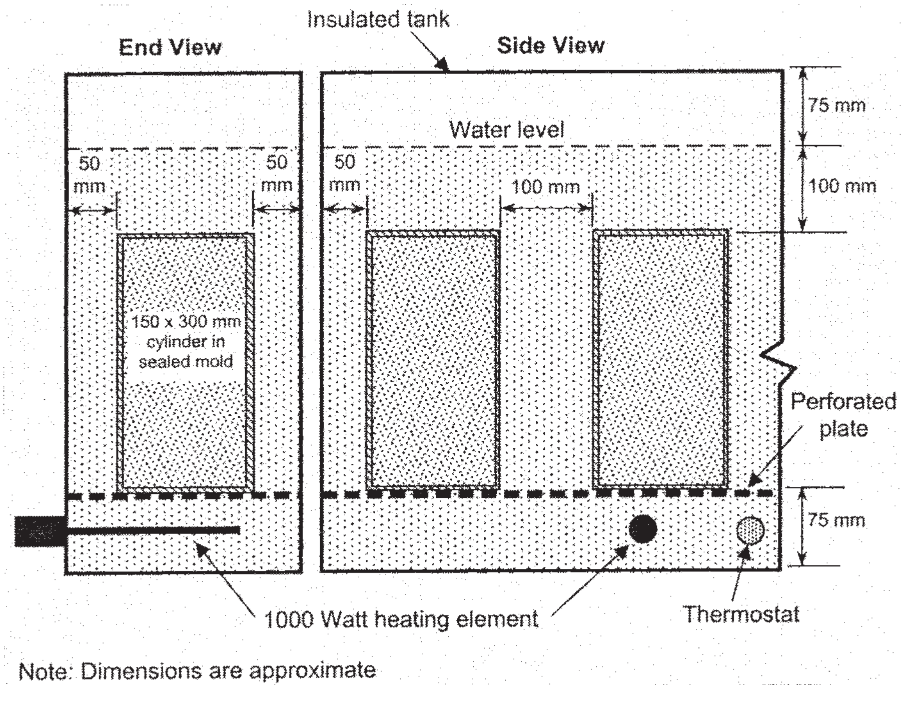
Accelerated Curing Apparatus

The accelerated procedures involving the use of either hot or boiling water were conducted in thermostatically controlled tanks (Fig. 1). The tops of the cylinders were covered by about 100 mm (4 in.) of water. Water volume and heater capacity were sized to prevent an appreciable reduction in the desired water temperature when the specimens were immersed. Specimens were not placed in a tank that already contained specimens.

The autogenous curing container for Procedure D was similar to that used by Smith and Tiede [7] to cure a single 152 by 305-mm (6 by 12-in.) cylinder. It was made by using a plastic container and polyurethane foam insulation (Fig. 2). The polyurethane foam retained the heat of hydration of the cement, which accelerated the strength development of the concrete. Once curing was initiated, the container was not opened until the end of the specified curing period.

Results

The results of the cooperative testing program were summarized by Wills [8], and only the main conclusions are highlighted here. The objective was to establish the nature of the relationships between accelerated and standard-cured strengths, and to examine whether these relationships were affected by factors such as the type of accelerated procedure,



Note: Dimensions are approximate

Fig. 1—Hot or boiling-water accelerated curing tank.

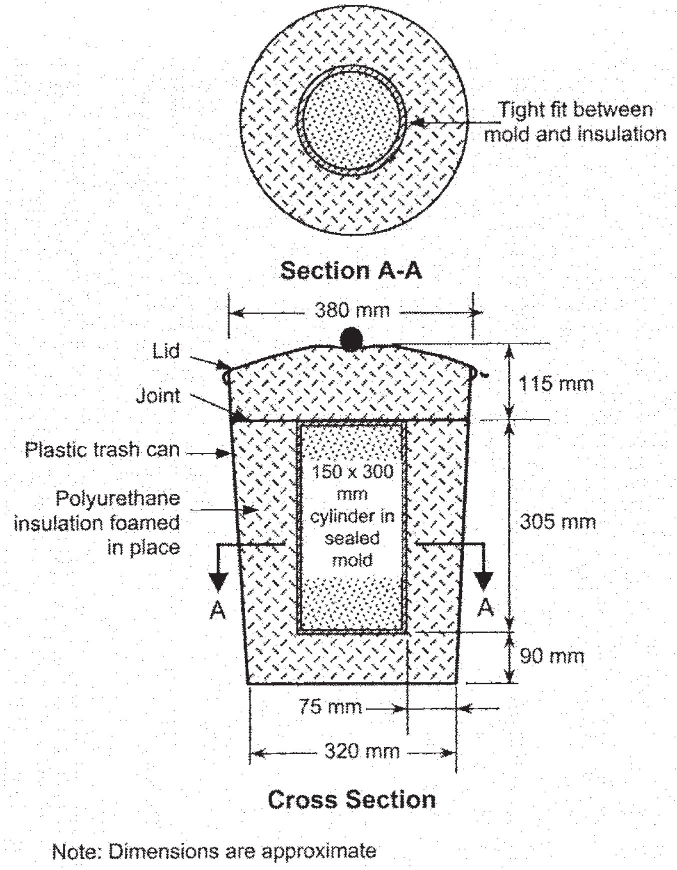


Fig. 2—Autogenous curing container.

cement type, cement content, presence of set retarder, and laboratory. Only the results of Procedures A, B, and D are discussed here, because these procedures were eventually incorporated into the first version of ASTM C 684 in 1974.

Procedure A (Warm Water)

Cylinders were cast in steel molds with tight-closing lids. Immediately after casting, the cylinder molds were immersed in the water bath at a temperature of 33 to 37°C (92 to 98°F), where they remained for a period of 24 h ± 15 min. Sulfur mortar caps were applied to the cylinders and aged at least 1 h prior to measuring compressive strength at an age of 26 h ± 15 min. The warm-water curing increased concrete strengths from 1.1 to 1.6 times those achieved after one day of standard moist curing.

Figure 3 shows the relationships between accelerated strength and 28-day strength for concretes made with Type I cement. Within each laboratory, there were good correlations; however, it appeared that each laboratory obtained significantly different correlations.

Although not shown, the same general trends were observed with concretes made with Type III cement, except that the relationships were at a higher strength level. Since the test procedures were carefully controlled, the results emphasized the effect of local materials, particularly the type of cement, on the relationships between accelerated strength and standard-cured strength. Based upon the good correlations, however, it was concluded that Procedure A can be used with a high degree of confidence to assess concrete quality when the tests are made on mixtures produced from the same materials.

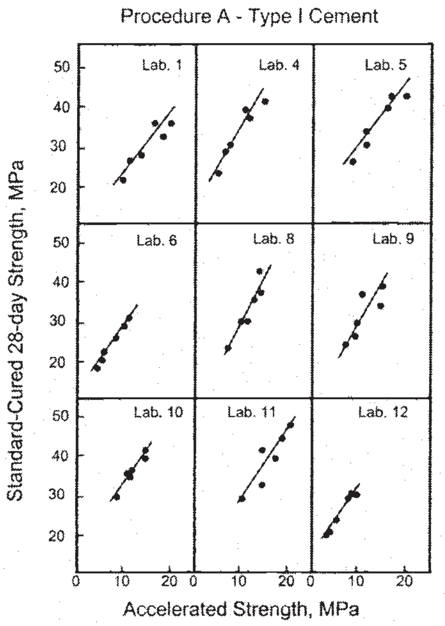


Fig. 3—Relationships between accelerated and standard-cured strengths for Procedure A (Warm Water Method) using Type I cement.

Procedure B (Modified Boiling)

After the cylinders were cast, the sealed molds were placed in a standard moist room. At 23 h ± 15 min from the time of casting, the cylinders, including molds and covers, were immersed in boiling water. Reduction in water temperature was limited to 3°C (5°F), and the temperature was required to recover to the boiling point in no more than 15 min. After boiling for 3.5 h ± 5 min, the cylinders were removed from the molds and allowed to cool for about 45 min. They were capped with sulfur mortar, the sulfur was allowed to age for 1 h, and strengths were measured at an age of 28.5 h ± 15 min. The modified boiling procedure increased concrete strength between 1.1 and 2.1 times that measured after one day of standard moist curing.

Figure 4 shows the results for concretes cured by Procedure B and made with Type III cement. The trends discussed for Procedure A are reemphasized. Most importantly, the laboratories also obtained what seemed to be significantly different correlations. Again, this is attributed to each laboratory using locally available materials. Within a given laboratory, however, good correlations between accelerated strength and 28- or 364-day strengths were obtained [8]. Therefore, Procedure B was also considered to have equal merit in assessing concrete quality.

Procedure D (Autogenous Curing)

Cylinders were molded in light-gage steel molds. One hour after the start of mixing, the molded cylinders were sealed in plastic bags and placed inside the autogenous curing chambers (Fig. 2). They remained there for 46 h, then they were removed from the molds and allowed to cool for 45 min. Sulfur mortar caps were applied and aged at least 1 h before strength was measured at an age of 49 h ± 15 min.

Only four laboratories performed this procedure, but their data were sufficient to justify including autogenous curing in the subsequent ASTM standard. The two-day strengths after autogenous curing ranged from 1.4 to 2.5 times those obtained

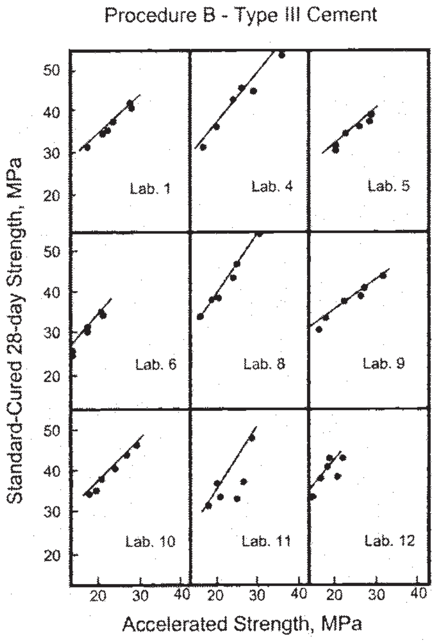


Fig. 4—Relationships between accelerated and standard-cured strengths for Procedure B (Modified Boiling Method) using Type III cement.

after an equal length of standard moist curing, which was the highest level of strength acceleration.

High concrete temperatures at early ages are detrimental to the early hydration reactions of cement and to the developing paste microstructure. With the autogenous curing procedure, the heat of hydration of the cement causes the acceleration but at a low rate of temperature increase, which is beneficial to early hydration reactions. This previous fact, plus the additional fact that higher curing temperatures are attained in this procedure compared with Procedure A, may explain the high values of accelerated strength for Procedure D.

Significance of Test Procedures

The subcommittee was convinced that laboratories, test procedures, and cement types had significant effects on the correlation between accelerated strength and standard-cured strength, and the data were analyzed from that viewpoint [8]. It was assumed that the correlation for each set of conditions could be represented by a straight-line relationship as follows

$$\hat{S}_{28} = B_0 + B_1 S_a \tag{1}$$

where

S_{28} = the standard-cured 28-day strength,
S_a = the accelerated strength for a particular procedure,
B_0 = the intercept, and
B_1 = the slope.

For each laboratory, cement type, and procedure, the best-fit values of B_0 and B_1 were obtained by least squares fitting. The resulting values of B_0, B_1, correlation coefficients (r), and residual standard deviations are summarized in Tables 2, 3, and 4 for Procedures A, B, and D, respectively. The residual standard deviation (s_e) is a measure of the error between the data and the best-fit straight line and is computed as follows:

$$s_e = \sqrt{\frac{\sum_{i}^{n} (S_{ai} - \hat{S}_{28})^2}{n - 2}} \tag{2}$$

TABLE 2—Linear Regression Analysis Results for Procedure A

Laboratory Number	Cement Type	B_0,[a] MPa	B_1[a]	r[b]	RSD,[c] MPa[d]
1	I	13.54	1.120	0.910	2.10
	III	15.65	0.960	0.975	0.86
4	I	19.10	1.515	0.955	1.96
	III	18.96	1.095	0.925	2.76
5	I	15.27	1.375	0.960	1.86
	III	17.86	0.905	0.980	1.07
6	I	14.17	1.525	0.985	0.86
	III	14.31	1.290	0.940	1.93
8	I	9.10	1.985	0.905	2.90
	III	15.44	1.285	0.935	2.31
9	I	16.31	1.320	0.765	3.69
	III	23.48	0.540	0.870	1.59
10	I	19.13	1.440	0.915	1.62
	III	24.20	0.770	0.815	1.28
11	I	14.34	1.475	0.905	2.83
	III	13.76	1.090	0.725	3.62
12	I	17.65	1.440	0.915	1.96
	III	15.65	0.580	0.825	2.07

[a] See Eq 1 for meaning of B_0 and B_1.
[b] r = correlation coefficient.
[c] RSD = residual standard deviation.
[d] 1 MPa ≈ 145 psi.

TABLE 3—Linear Regression Analysis Results for Procedure B

Laboratory Number	Cement Type	B_0,[a] MPa	B_1[a]	r[b]	RSD,[c] MPa[d]
1	I	12.82	1.060	0.970	1.34
	III	16.55	0.910	0.985	0.66
4	I	17.79	1.290	0.965	1.55
	III	13.96	1.145	0.955	2.17
5	I	9.86	1.225	0.975	1.21
	III	14.96	0.840	0.930	1.28
6	I	11.58	1.280	0.970	0.79
	III	13.13	1.050	0.965	1.07
8	I	15.62	1.280	0.810	4.03
	III	14.86	1.220	0.950	2.03
9	I	16.89	1.015	0.865	1.58
	III	19.55	0.780	0.960	1.24
10	I	19.48	1.195	0.950	1.24
	III	17.79	1.000	0.975	0.90
11	I	9.52	1.515	0.975	1.86
	III	13.41	1.095	0.800	3.48
12	I	18.13	1.020	0.860	2.48
	III	18.27	1.143	0.830	3.07

[a] See Eq 1 for meaning of B_0 and B_1.
[b] r = correlation coefficient.
[c] RSD = residual standard deviation.
[d] 1 MPa ≈ 145 psi.

where

S_{ai} = the average accelerated strength of the ith specimens,
$\hat{S}_{28}$ = the estimated 28-day strength corresponding to S_{ai}, and
n = number of pairs of strength values used in the regression analysis.

For example, the regression equation for Laboratory 6 using Procedure A and Type I cement was

$$\hat{S}_{28} = 14.17 + 1.525\, S_a \text{ (MPa)} \qquad (3)$$

Note that the correlation coefficient had a high value of 0.985 and residual standard deviation had a correspondingly low value of 0.86 MPa (125 psi). On the other hand, Laboratory 11 had a much lower correlation coefficient and a higher residual standard deviation. Therefore, the data for Laboratory 11 did not fit its straight-line relationship as well as the data for Laboratory 6.

TABLE 4—Linear Regression Analysis Results for Procedure D

Laboratory Number	Cement Type	B_0,[a] MPa	B_1[a]	r[b]	RSD,[c] MPa[d]
4	I	11.45	1.000	0.990	0.96
	III	3.79	1.230	1.000	0.41
5	I	22.03	1.315	0.780	4.07
	III	22.65	0.875	0.960	1.48
8	I	7.17	1.400	0.950	1.72
	III	9.96	1.125	0.970	1.93
12	I	16.86	0.930	0.900	2.10
	III	9.82	0.810	0.930	1.45

[a] See Eq 1 for meaning of B_0 and B_1.
[b] r = correlation coefficient.
[c] RSD = residual standard deviation.
[d] 1 MPa ≈ 145 psi.

After studying the residual standard deviations, it was concluded that all three procedures were equal in correlating accelerated and later-age strengths. Correlation coefficients were quite high in most cases, as would be expected by the close fit of the data to the linear relationships shown in Figs. 3 and 4 for each laboratory. Further, it was found that accelerated compressive strength correlated with 364-day strength as well as with 28-day strength. Subsequently, the subcommittee started work to develop a tentative test method that included Procedures A, B, and D. The tentative test method was approved in 1971 as ASTM C 684-71T.

Test Precision

While accelerated strengths measured by the nine laboratories were significantly different for a given procedure, the variances (squares of standard deviations) of replicate strength measurements seemed compatible and were therefore pooled across laboratories and cement types for each procedure. The within-batch and batch-to-batch coefficients of variation that were obtained from the data analysis are shown in Table 5 [8]. These values were used to prepare precision statements, according to ASTM Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials (C 670), for the three procedures [8]. Because of the small differences between the resulting statements, they were combined into a single precision statement when ASTM C 684 was published as a tentative test method. The autogenous curing procedure was called Procedure C in ASTM C 684. Note that the precision statements deal only with the repeatability of the measured accelerated strengths. A procedure for determining the precision of the estimated standard-cured strengths is discussed subsequently.

TABLE 5—Precision of Accelerated Strength Tests [8]

Procedure	Within-Batch Coefficient of Variation, %	Batch-to-Batch Coefficient of Variation, %
Warm water	2.9	8.2
Modified boiling	3.0	8.5
Autogenous curing	3.6	8.5

Effect of Cement Chemistry

In order to explain the differences between laboratories, a program was conducted to determine the chemical or physical properties of cement, or both, that affect the one-day accelerated strength of concrete. Similar concrete batches were made using eight Type I cements mixed with the same source of sand and gravel. An attempt was made to hold the slump constant at 75 to 100 mm (3 to 4 in.). Four 152 by 305-mm (6 by 12-in.) cylinders were molded from each batch. Two cylinders were cured according to ASTM C 684, Procedure A, to obtain one-day accelerated strengths and two cylinders were moist-cured at 23°C (73°F) in 100 % relative humidity to obtain standard 28-day strengths.

After all cylinders were tested, the physical and chemical properties of each cement along with the corresponding concrete strengths were analyzed to search for correlations. A strong correlation was found between the sodium alkali (Na_2O) content and the one-day accelerated strengths (Fig. 5).

Several multiple correlations were also examined. For two independent variables, only combinations involving Na_2O produced useful correlations with one-day accelerated strength; moreover, only loss on ignition (LOI) coupled with the Na_2O produced a better multiple correlation than Na_2O alone.

Therefore, the variation between the accelerated strengths for the different Type I cements was attributable mainly to variations in alkali content. Undoubtedly, this caused the principal variation between laboratories in the cooperative test program since each used a different Type I cement.

High Temperature and Pressure Method

In 1978, a different accelerated strength procedure was proposed that could produce test results within 5 h [9]. Acceleration of strength development in the new procedure is achieved by a combination of elevated temperature and pressure. A schematic of the apparatus is shown in Fig. 6. Special molds with heating wires and insulation are used to prepare 76 by 152-mm (3 by 6-in.) cylinders. After three molds are filled, they are stacked in a compression testing frame, a compressive stress of 10.3 ± 0.2 MPa (1500 ± 25 psi) is applied, and the electrical heaters are turned on. The heaters raise and maintain the concrete temperature at 149 ± 3°C (300 ± 5°F). After three hours, the heaters are turned off, the axial stress is maintained, and the specimens are allowed to cool for 2 h. The hardened cylinders are extruded from the molds and tested for compressive strength. Usually, capping materials are not needed because the metal end caps result in sufficiently flat ends.

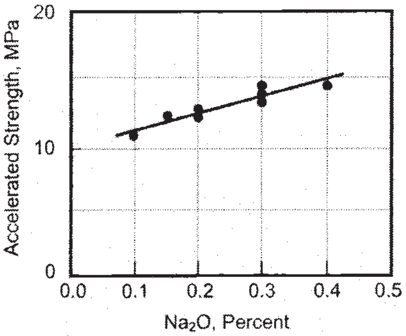


Fig. 5—Relationship between alkali content and accelerated strength using Procedure A.

In 1980, the subcommittee was requested to modify the existing version of ASTM C 684 to permit the high temperature and pressure (HTP) procedure as an alternative to the other methods. Data were provided to the subcommittee to demonstrate that the method resulted in correlations similar to the other methods [10]. The concrete mixtures were made with Type I, III, and IV cements; water-cement ratios (*w/c*) between 0.45 and 0.90; with and without fly ash, air-entraining agent, or water reducer; and with normal-density and low-density (lightweight) aggregates. The accelerated strengths were compared with the strengths of standard-cured 152 by 305-mm (6 by 12-in.) cylinders. The accelerated strengths were between 22 and 90 % of the 28-day strengths.

A task force of the subcommittee compared the residual standard deviations of correlations obtained with the HTP procedure with those obtained by others using the standard procedures. The results of the comparison are shown in Table 6. The relationships for the HTP method were expressed as power functions as follows:

$$\hat{S}_{28} = B_0 S_a^{B_1} \tag{4}$$

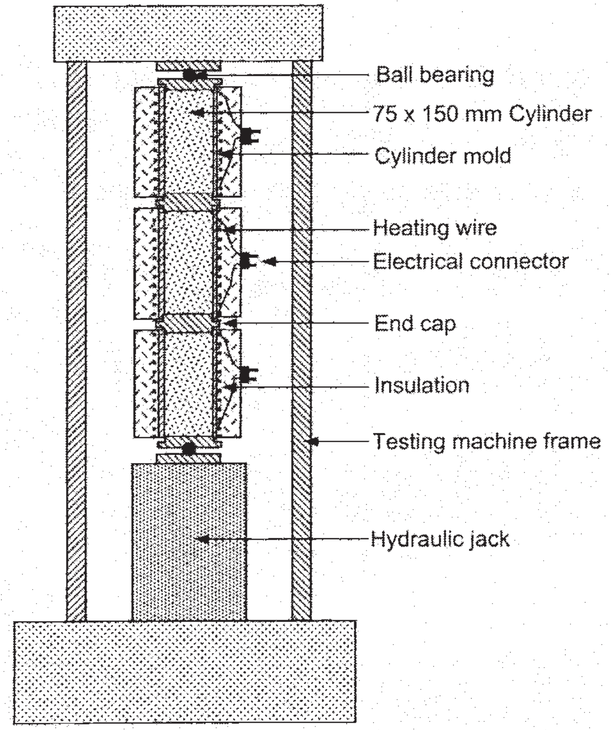


Fig. 6—Schematic of apparatus for high temperature and pressure accelerated curing method.

TABLE 6—Comparison of Accelerated Strength Correlations

Reference	Procedure	Cement Type	Regression Equation	Number of Points	RSD,[a] MPa[b]
Bickley [11]	Autogenous curing	I	Linear	43	2.35
		IV	Linear	147	2.46
		I and IV	Linear	68	2.08
Lapinas [12]	Modified boiling	I	Linear	312	1.82
Roadway and Lenz [13]	Modified boiling	I	Linear	219	3.66
		I	Linear	36	2.34
		I	Linear	76	2.51
		V	Linear	61	1.58
Bisaillon [14]	Autogenous curing	I	Linear	not given	1.48
Bisaillon et al. [15]	Autogenous curing	I	Linear	213	2.30
Malhotra [16]	Modified boiling	I	Linear	336	2.40
		I	Linear	40	2.64
		I	Linear	265	3.54
Nasser and Beaton [10]	High temperature and pressure	I	Power function	171	3.59
		III	Power function	99	3.36
		IV	Power function	65	4.03

[a] RSD = residual standard deviation.
[b] 1 MPa ≈ 145 psi.

Based on the comparisons, the task force recommended that the HTP method should be added to ASTM C 684, because the 5-h accelerated strength correlated reasonably well with standard-cured strength. Subsequently, a member of the subcommittee reported on a comparative study of the HTP method and the modified-boiling method (Procedure B).[2] In this study, mixtures were made with fly ash from different sources, with *w/c* values of 0.4, 0.5, and 0.7, and with an air-entraining agent. The accelerated strengths were correlated with standard-cured, 28-day strengths (Fig. 7). Power functions were fitted to the data. The residual standard deviations were 1.21 MPa (175 psi) and 1.77 MPa (257 psi) for Procedure B and the HTP method, respectively. Surprisingly, the best-fit equations were very similar. This study provided further evidence of the suitability of the HTP method, and steps were taken to incorporate this procedure into ASTM C 684.

During the balloting process, concern was expressed that the hydration reactions due to the high temperature and pressure would not be representative of those due to normal curing. Thus it was felt that the accelerated strength from the HTP method might not be indicative of the potential strength under standard curing. Proponents of the method noted that the data obtained by using a variety of materials showed good correlations. In 1989, the HTP method was adopted as alternative Procedure D in ASTM C 684.

Estimation of Later-Age Strength

To estimate the potential later-age strength from a measured early-age accelerated strength, the laboratory must first conduct enough tests to establish the regression equation and its residual standard deviation. To account for the uncertainty in the regression line, the confidence interval for the line is established [17,18]. Then, for a new accelerated strength, the confidence interval for the estimated average later-age strength can be detemined.

To illustrate the procedure, consider the 12 pairs of accelerated and 28-day strengths given in the first two columns of Table 7. Each number is the average strength of two cylinders.

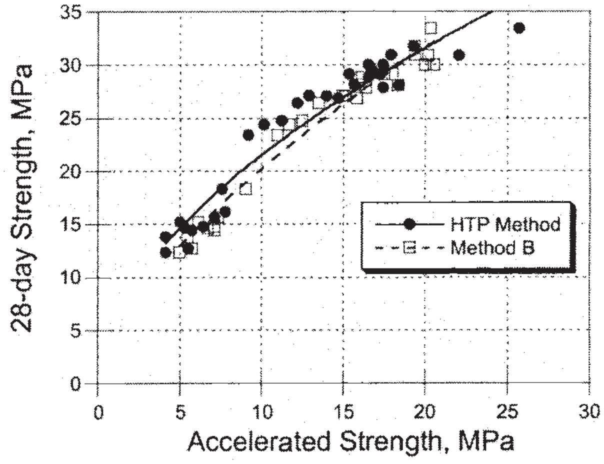


Fig. 7—Comparison of relationships between accelerated and standard 28-day strengths.

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[2] V. M. Malhotra, private communication, Canada Centre for Mineral and Energy Technology, Ottawa, Ont., Canada, 11 Jan. 1985.

TABLE 7—Values Used in Sample Problem to Illustrate Estimated 95 % Confidence Interval for 28-day Strength

Accelerated Strength, S_a, MPa[a]	28-day Strength, S_{28}, MPa	Estimated Strength, $\hat{S}_{28}$, MPa	W_i, MPa	Lower Confidence Limit, MPa	Upper Confidence Limit, MPa
12.06	33.71	33.86	1.78	32.07	35.64
12.15	34.33	33.96	1.76	32.20	35.72
12.96	35.23	34.92	1.54	33.38	36.46
13.85	35.05	35.99	1.33	34.66	37.31
15.19	37.74	37.58	1.09	36.49	38.67
16.09	37.21	38.65	1.03	37.62	39.67
17.08	40.71	39.82	1.06	38.76	40.88
18.15	40.97	41.10	1.21	39.89	42.30
18.24	41.96	41.20	1.22	39.98	42.42
18.42	41.60	41.42	1.26	40.16	42.67
20.12	45.73	43.44	1.67	41.77	45.11
21.28	42.50	44.82	2.00	42.82	46.82
CONFIDENCE INTERVAL FOR ESTIMATED STRENGTH AT ACCELERATED STRENGTH OF 17.00 MPa					
17.00		39.73	1.05	38.68	40.78
16.29		38.89	1.02	37.87	
17.71		40.57	1.13		41.70

[a] 1 MPa ≈ 145 psi.

Using ordinary least squares regression analysis, the best-fit regression equation for the data (Fig. 8) is

$$\hat{S}_{28} = 19.51 + 1.19\ S_a\ (\text{MPa}) \tag{5}$$

The residual standard deviation of the line, s_e, is 1.24 MPa (180 psi).

The 95 % confidence interval for the line [17,19] is constructed by calculating $\hat{S}_{28}$ for selected values of S_a and plotting $\hat{S}_{28} \pm W_i$,

$$W_i = s_e\sqrt{2F}\ \sqrt{\frac{1}{n} + \frac{(S_{ai} - \bar{S}_a)^2}{S_{aa}}} \tag{6}$$

where

W_i = half-width of confidence interval at S_{ai},
s_e = residual standard deviation for best-fit line (Eq 2),
F = value from F-distribution for 2 and n degrees of freedom and significance level 0.05,
n = number of data points used to establish regression line,
S_{ai} = selected value of accelerated strength,
$\bar{S}_a$ = grand average value of accelerated strength for all data points used to establish the regression line,
$S_{aa} = \Sigma(S_a)^2 - (\Sigma\ S_a)^2/n$, and
S_a = values of average accelerated strength for each point used to establish the regression line.

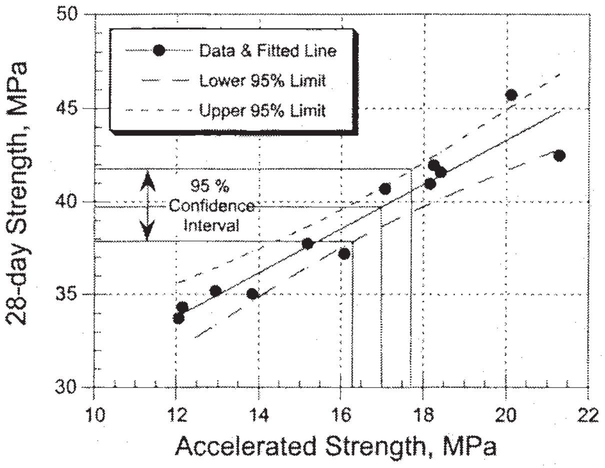

Fig. 8—Confidence limits for the estimated 28-day strength based on a measured accelerated strength and the previously established strength relationship.

The third column in Table 7 lists the estimated average 28-day strengths for the accelerated strengths in Column 1. The value of W_i at each value S_a is listed in the fourth Column of Table 7. Finally, columns five and six list the values of the lower and upper 95 % confidence limits that are shown in Fig. 8. Note that the width of the confidence limits is narrowest when S_{ai} equals $\bar{S}_a$, because the second term under the square root sign in Eq 6 equals zero.

Suppose that the average accelerated strength of two cylinders made from similar concrete is 17.0 MPa (2460 psi). From the regression equation, the estimated average 28-day strength is 39.7 MPa (5760 psi). If the accelerated strength were known without error, the 95 % confidence interval for the average 28-day strength would be 38.7 to 40.8 MPa (5610 to 5920 psi) (see the bottom of Table 7). However, the accelerated strength has an uncertainty that is described by the within-batch standard deviation. Assume that the compressive strengths measured by the specific accelerated test method have a single-laboratory, within-batch coefficient of variation of 3.0 %. Therefore, the standard deviation, s, at an average strength of 17.0 MPa (2460 psi) is 0.51 MPa (74 psi). The 95 % confidence interval for the average accelerated strength of the two cylinders is

$$17.0 \pm z_{0.025}\frac{S}{\sqrt{2}} = 17.0 \pm 1.96 \times 0.51 \times 0.707 = 17.0 \pm 0.7\ \text{MPa}$$

where $z_{0.025}$ is the value from the standard normal distribution corresponding to 2.5 % of the area under the curve. Thus the 95 % confidence interval[3] for the average accelerated strength is 16.3 to 17.7 MPa (2360 to 2570 psi). Projecting the limits of this interval to the lower and upper confidence limits of the regression line results in 37.9 to 41.7 MPa (5500 to 6050 psi) for the approximate 95 % confidence interval for the average 28-day strength. Each different measurement of accelerated strength produces a new confidence interval for the average 28-day strength. A personal computer is recommended for implementing the preceding calculations for routine use.

In 1999, ASTM C 684 was revised to provide explicit guidance on using statistical methods to estimate later-age strength. A new paragraph was added under "Interpretation of Results," which stated that statistical methods were to be used to account for the various sources of uncertainty. It was also stated that all interested parties were to agree on the statistical procedures to be used and the criterion for acceptance testing. It was recommended that estimated strength corresponding to the lower 90 % limit could be used to compare with the specified standard-cured strength. A nonmandatory Appendix X2 was added to provide a recommended statistical methodology based on the above statistical procedure, except that the 90 % confidence interval ($z_{0.05} = 1.645$) was used in the illustrative example instead of the 95 % interval as presented above.

In summary, to estimate the average 28-day strength based upon accelerated strength test results, a procedure is used that accounts for the uncertainty in the regression line and in the measured accelerated strength. It is insufficient to simply use the regression equation to convert the accelerated strength to an equivalent 28-day strength. Additional information on the procedure presented in the example may be found in the references by Moore and Taylor [21] and in Miller [19]. Finally, it is emphasized that a particular regression equation is valid only for a specific test procedure and combination of materials. Each laboratory must conduct enough tests with a given set of materials and a certain procedure to establish the regression line and its confidence limits before estimates of later-age strengths are possible.

Later-Age Strength Estimates Using The Maturity Method

Overview

The other method developed by the original Subcommittee on Accelerated Strength Testing to estimate the later-age strength from early-age tests is based upon the maturity method. Unlike the accelerated strength methods, there are no special curing requirements for this procedure, but it does require measuring the temperature history of the test specimens. The method is an outgrowth of research performed in the late 1960s and early 1970s by Hudson and Steele [22,23] at the West Virginia Department of Highways, and it was adopted as ASTM C 918 in 1980. The basic principles of the maturity method are discussed by Malhotra in another chapter of this publication and a more comprehensive review is also available [1].

The motivation for the development of ASTM C 918 is discussed first. Concrete mixtures cured at standard temperature gain strength predictably, that is, there are fixed relationships between the strengths at early ages and at a later age. Attempts have been made, therefore, to estimate the later-age strength, such as at 28 days, by multiplying the early-age strength by an empirical factor. However, such estimates have not been found to be reliable. As has been explained [22,23], there are two pitfalls to practical application of this simple approach: (1) on a construction project, it is not possible to perform the early-age tests at precisely the specified age, and (2) the early-age temperatures cannot be controlled as accurately as needed. The procedure in ASTM C 918 overcomes these limitations by requiring measurement of actual temperature histories of the early-age specimens and by using a "maturity index," rather than age, to relate to the level of strength development. The meaning of "maturity index" is discussed in the next section.

To use ASTM C 918, the testing laboratory first establishes a relationship between the strength of concrete cylinders and the maturity index. Subsequently, cylinders made from similar concrete are tested at early ages. The maturity index from the time of molding each set of cylinders until the time of testing is recorded. The early-age strengths and the corresponding values of maturity index are then used to estimate the later-age strength based upon the previously established strength-maturity index relationship.

The following provides a brief review of the terminology associated with the maturity method.

Concrete Maturity

In concrete technology, the term "maturity" refers to the extent of the development of those properties that depend on cement hydration and pozzolanic reactions. At any age, maturity depends on the previous curing history, that is, the temperature history and the availability of water to sustain the chemical reactions. In the early 1950s, the idea was developed that the combined effects of time and temperature could be accounted for by using a "maturity function" to convert the temperature history to a "maturity index" that would be indicative of strength development. In practice, the maturity index is obtained by using electronic instruments that monitor the concrete temperature and automatically compute the maturity index as a function of age. The maturity index can be displayed on a self-contained unit or downloaded to a computer. The reader is urged to read the appropriate section of Malhotra's chapter in this publication and ASTM Practice for Estimating Concrete Strength by the Maturity Method (C 1074) for background information on the maturity functions used to compute the maturity index. In this chapter, the "equivalent age at 23°C" is used as the maturity index. While this index is not as

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[3] The 95 % confidence interval is often interpreted to mean that there is a 95 % probability that the true mean falls within the interval. However, the correct interpretation is as follows: If 100 repeated samples are taken from the same population and the 95 % confidence intervals for the mean are computed in each case, 95 of the intervals would include the true mean. The 95 % confidence interval for the regression line has a similar interpretation: If 100 groups of data are taken from the same population and the 95 % confidence intervals are computed for the regression equations, 95 of those intervals would include the true regression line for the population. See Mendenhall and Sincich [20] for further explanations on the proper interpretation of confidence intervals.

well known in the United States as the "temperature-time factor" (expressed in degree-hours or degree-days), equivalent age is the more meaningful quantity and is expected to be used more widely in the future. The equivalent age is the curing age at a specified temperature that would result in the same maturity as has occurred at the actual age under the actual temperature history.

The "strength-maturity relationship" for a specific concrete mixture is used to relate the strength development to the maturity index. The relationship can be used to estimate strength development under different temperature histories, as is covered by ASTM C 1074, or it can be used to estimate the later-age strength under standard curing based upon a measured early-age strength, as covered by ASTM C 918.

Strength-Maturity Relationship

As mentioned earlier, knowledge of the relationship between strength and the maturity index is a fundamental requirement to apply the maturity method. Over the years, a variety of empirical equations have been proposed to represent such relationships. These have been reviewed elsewhere [1] and only the function adopted in ASTM C 918 is discussed here.

In 1956, Plowman [24] proposed that the strength of concrete could be related to the maturity index by the following semi-logarithmic function

$$S_M = a + b \log M \tag{7}$$

where

S_M = compressive strength at M,
M = maturity index, and
a, b = regression constants.

According to Eq 7, strength is a straight line function of the logarithm of the maturity index. This is generally a reasonable approximation for strength development between 1 and 28 days under standard room temperature curing. The parameter a represents the strength at a maturity index of 1 (the logarithm of 1 equals zero).[4] The parameter b, which is the slope of the line, represents the increase in strength for a tenfold increase in the maturity index. For example, if the equivalent age at 23°C increases from one day to ten days, the strength increase equals b. The values of a and b depend on the materials and mixture proportions of the specific concrete [22–24].

Application

To use ASTM C 918 to estimate the potential later-age strength at 28 days, for example, it is first necessary to establish the strength-maturity relationship, that is, the "prediction equation," for the concrete mixture. This is done by preparing standard cylindrical specimens and subjecting them to standard curing procedures. A maturity meter is used to monitor the maturity index of the specimens.[5] Pairs of cylinders are tested at regular intervals (for example, at 1, 3, 7, 14, and 28 days),[6] and the corresponding maturity indexes are recorded along with the average strengths. The values of a and b are obtained by least squares fitting of Eq 7 to the data.

To estimate the later-age strength of a similar concrete mixture based on the results of subsequent early-age tests, the maturity index of the early-age test specimens is monitored from the time of molding until the time of testing. This is conveniently done by casting a "dummy" cylinder into which the sensor of the maturity meter is embedded. The instrumented cylinder is exposed to the same environment as the cylinders that will be tested for strength. The values of the early-age strength, S_m, and maturity index, m, are used along with the previously established strength-maturity relationship to estimate the potential later-age strength.

During a project, the concrete-making materials will usually be the same from batch to batch, and the main purpose of cylinder strength tests is to assure that the specified strength is achieved. The value of a in Eq 7 depends, however, on the water-cement ratio since it represents the strength at a particular maturity index. A change in the value of a shifts the straight line along the strength axis. It is not appropriate to assume that the value of a obtained in the laboratory testing program is applicable to the cylinders being evaluated by early-age testing. It is reasonable, however, to assume that the value of b is applicable, because large changes in w/c are needed to change the value of b [23]. The purpose of the early-age results is to establish the appropriate value of a as follows:

$$a = S_m - b \log m \tag{8}$$

where

m = maturity index at time of early-age test, and
S_m = average early-age strength measured at maturity index, m.

By substituting Eq 8 into Eq 7, one obtains the following "prediction equation" for estimating the strength at a later age:

$$S_M = S_m + b\,(\log M - \log m) \tag{9}$$

where

M = maturity index at the later age when strength is to be estimated, and
S_M = estimated strength at maturity index M.

Again, this derivation assumes that the value of b is not affected by small variations in w/c.

Example

The following example illustrates the application of ASTM C 918. Assume that a set of cylinders are molded in the laboratory and that one of them is instrumented with a maturity meter that computes the equivalent age at 23°C. Table 8 gives the average strengths (two cylinders) that are obtained at different values of the maturity index.

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[4] Note that the value of a depends on the units used for the maturity index. If the maturity index is expressed as the temperature-factor (degree-hours or degree-days), the value of a will be a negative number that has no physical significance. If the maturity index is expressed in terms of equivalent age at 23°C in days, the value of a is the strength at one day of curing at 23°C.

[5] In the original version of ASTM C 918, the measurement of the ambient temperature history was considered adequate. However, revisions in 1993 called for monitoring the actual concrete temperature. The availability of relatively inexpensive maturity meters justifies this more precise approach.

[6] Testing at ages that are related by a factor of two will result in approximately equal strength increments.

TABLE 8—Equivalent Age and Compressive Strength Values Used in Illustrative Example

Age, days	Equivalent Age at 23°C, days	Average Strength, MPa (psi)
1.0	0.91	9.44 (1370)
3.0	3.10	17.09 (2480)
7.0	7.25	21.77 (3160)
14.0	14.5	25.56 (3710)
28.0	27.3	29.28 (4250)

These strengths have been plotted as a function of the logarithm of the equivalent age (Fig. 9). The best-fit equation is

$$S_M = 10.25 + 13.29 \log M, \text{ MPa} \tag{10}$$

Further assume that cylinders fabricated in the field were subjected to "standard curing" as specified in ASTM Practice for Making and Curing Concrete Test Specimens in the Field (C 31/C 31M). After 24 h, two cylinders were removed from their molds, capped, and tested for compressive strength. The average strength of two cylinders was 10.0 MPa (1450 psi), and the corresponding equivalent age was 1.13 days based on a maturity meter in the dummy cylinder. According to Eq 9, the estimated 28-day, standard-cured strength would be

$$\begin{aligned} S_{28} &= 10.0 + 13.29\,(\log 28 - \log 1.13) \\ &= 10.0 + 13.29\,(1.4472 - 0.0531) \\ &= 10.0 + 18.5 \\ &= 28.5 \text{ MPa (4130 psi)} \end{aligned}$$

Interpretation of Results

The intent of early-age tests is to provide an early indication of the potential strength of the concrete sample. It is unrealistic to expect that the traditional standard tests at later ages, such as at 28 days, will be eliminated in the near future. Thus the 28-day strengths of standard-cured specimens will probably continue to be measured. By keeping records of estimated and measured 28-day strengths of companion specimens from the same batch, one can continually correct and improve the slope, *b*, of the prediction equation by using the following relationship [22]:

$$b = \frac{\sum (S_{28} - S_m)}{\sum (\log M_{28} - \log m)} \tag{11}$$

where

S_{28} = measured standard-cured 28-day strength,
M_{28} = maturity index corresponding to standard curing for 28-days, and
S_m = measured early-age strength at maturity index m.

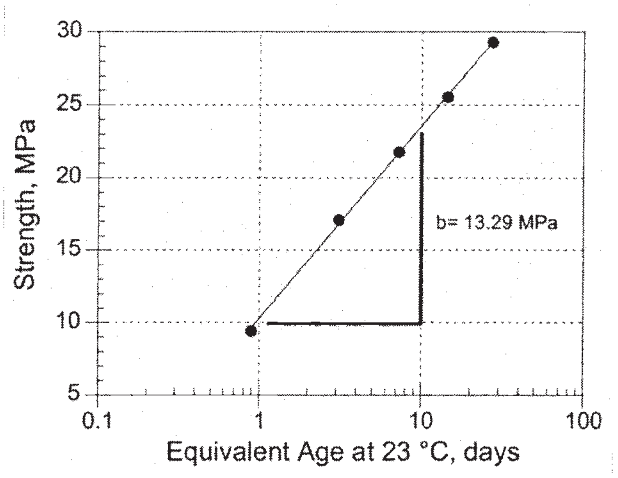

Fig. 9—Example of a strength versus maturity index relationship; the slope is used in the prediction equation to estimate later-age strength based upon early-age strength.

In addition, the differences between the estimated and measured 28-day strengths can be used to calculate a confidence limit for future estimates, and thereby establish an acceptance criterion for future early-age results. First, the average difference, $\bar{d}$ between the measured and estimated 28-day strengths is computed

$$\bar{d} = \frac{\sum_{i=1}^{n} (S_{28} - S_M)}{n}$$

$$\bar{d} = \frac{\sum_{i=1}^{n} d_i}{n} \tag{12}$$

where

S_M = the estimated 28-day strength,
S_{28} = measured standard-cured 28-day strength,
d_i = the difference between the i^{th} pair of strength values, and
n = number of pairs of strength values.

The value of $\bar{d}$ is the "bias" of the prediction equation, and it should be close to zero if the value of b is updated as new data are accumulated. The standard deviation for the difference between the measured and estimated strengths is calculated as follows

$$s_d = \sqrt{\frac{\sum_{i=1}^{n} (d_i - \bar{d})^2}{(n-1)}} \tag{13}$$

The upper 95 % confidence limit for the average difference between the measured and estimated 28-day strength is

$$K = \bar{d} + t_{0.95,n-1} \frac{s_d}{\sqrt{n}} \tag{14}$$

where

$t_{0.95,n-1}$ = value from the t-distribution at the 95 % point for $n - 1$ degrees of freedom

From such past data, it is possible to establish an acceptance criterion for the concrete based on the estimated strength of a future early-age test. If the design strength is f_{28}, the concrete represented by the early-age results would be considered acceptable if the following condition were satisfied:

$$S_m \geq f_{28} + K \tag{15}$$

Before the 1993 version of ASTM C 918, information on interpretation of results was included in the non-mandatory appendix. In the 1993 revision, this information was moved into the body of the standard. The definition of the factor K in the 1993 version differed from Eq 14. In the 1997 revision of ASTM C 918, Eq 14 was introduced, and ASTM C 1074 was referenced for terminology related to the maturity method. Minor changes were made in the 2002 revision of ASTM C 918.

Precautions

The prediction equation given by Eq 9 assumes that concrete strength is a linear function of the logarithm of the maturity index. Before implementing ASTM C 918, the user should verify that this assumption is valid. As was stated, past experience shows that the linear equation is usually adequate for strengths up to 28 days. There is no reason, however, why the procedure cannot be applied to estimate strength at ages later than 28 days. If the laboratory results reveal that the linear approximation is not applicable, the basic principle of ASTM C 918 can still be applied provided an appropriate equation is used to represent the actual strength-maturity relationship. Examples of equations that may be applicable are given in Carino [1].

Summary

This chapter has discussed the basis of two test methods to estimate the potential later-age strength of concrete specimens tested at early ages. The results of these early-age tests can provide timely information on the concrete production process for quality control. These procedures attempt to overcome the inherent deficiencies of the traditional practice, which relies on strength tests at ages of 28 days or later, to judge the adequacy of concrete batches.

One of the test methods (ASTM C 684) involves subjecting the early-age test specimens to specific curing conditions that accelerate strength development. Four alternative accelerated curing methods are specified that permit strength determinations at ages ranging from about 5 h to 48 h after preparing the specimens. Prior to using these measured accelerated strengths to estimate the later-age strength, laboratory testing is required to establish the relationship between the accelerated strength and the standard-cured strength. Typically, the standard-cured strength is measured at 28 days, but other ages are possible, such as 56 or 91 days, depending on the job specifications.

The cooperative testing program leading to the initial development of ASTM C 684 was reviewed. Analysis of the data from that program resulted in the following conclusions [8]:

1. Significantly different relationships were obtained by the nine laboratories due to the use of different local materials.
2. Different types of cements resulted in different relationships within the same laboratory.
3. The omission or addition of a normal amount of chemical retarder in the concrete did not affect the relationships.
4. Batch-to-batch variations due to mixing on different days did not have significant effects on the relationships.
5. Correlation of accelerated strength with 28-day and 364-day standard strength ranged from good to excellent.
6. For each cement type, accelerated strength correlated with 364-day strength as well as with 28-day strength.

Subsequent tests showed that significantly different strength relationships were obtained by the laboratories because of variations in alkali content of the cements. The cooperative study showed that accelerated strength testing by a single laboratory, using the same procedure and materials, results in an assessment of concrete quality at ages less than two days that is as reliable as that obtained after 28 days of standard moist curing [8]. Appropriate statistical techniques, as discussed in this chapter, should be used to establish a confidence interval of the estimated potential, later-age strength.

The other method (ASTM C 918) is based on the principle of the maturity method, in which the early-age curing history is converted to a maturity index indicative of the level of strength development. A previously established relationship is also required to estimate the potential, later-age strength based on the measured early-age strength. In this case, the relationship is between strength and the maturity index. Unlike the accelerated strength testing methods, ASTM C 918 does not require a precisely controlled curing procedure, provided sufficient moisture is maintained during early ages to sustain hydration. The specimens are cured according to the standard-curing procedures in ASTM C 31/C 31M. The only additional requirement is that the temperature history of the early-age specimens must be monitored to determine the maturity index (refer to ASTM C 1074) at the time of testing. Early-age strengths can be measured at any age 24 h after molding the specimens.

The basis of ASTM C 918 is research performed at the West Virginia Department of Highways [22,23]. In a statistically designed testing program, it was shown that the potential, later-age strength could be estimated with sufficient precision and accuracy for quality control purposes. This method also requires use of statistical principles for a reliable lower-bound estimate of the potential strength.

In summary, test methods are available to overcome the deficiencies of quality control programs based on the traditional 28-day strengths. The potential benefits arising from early-age testing should be considered carefully prior to setting up the quality control program to monitor the concrete production process on major construction projects.

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15

Freezing and Thawing

Charles K. Nmai[1]

Preface

THE SUBJECT OF FREEZE-THAW DURABILITY of concrete was covered in all four previous editions of the ASTM Special Technical Publication on significance of tests and properties; the chapters on this subject in *ASTM STP 169, ASTM STP 169A, ASTM STP 169B,* and *ASTM STP 169C* were authored by C. H. Scholer and T. C. Powers, H. T. Arni, H. Newlon, Jr., and H. Newlon, Jr. and T. Mitchell, respectively. Because little has changed in this subject area since the publication of *ASTM STP 169C,* this chapter includes only minor changes, that is, an update of the reference list and a few revisions in the text, to the version that appeared there.

Introduction

The purpose of this paper is to discuss the significance of currently standardized testing procedures for evaluating the resistance of concrete to weathering under service conditions. The published literature on the subject is extensive and only a somewhat superficial treatment can be given in a brief paper. The reader should consult the similar papers in earlier editions of this special technical publication by Scholer [1,2], Powers [3], Arni [4], and Newlon [5]. A monograph by Woods [6] presents a comprehensive picture of durability, and one by Cordon [7] discusses freezing and thawing in detail. Perhaps the most comprehensive treatment of the influence of aggregates on resistance to freezing and thawing is that of Larson et al. [8]. An extensive annotated bibliography on concrete durability published by the Highway Research Board (HRB) in 1957 [9] with a supplement in 1966 [10] contains 534 citations. A more current annotated bibliography, with some 600 citations, was published by the Strategic Highway Research Program in late 1992 [11]. The report of American Concrete Institute (ACI) Committee 201 on Durability of Concrete [12] contains valuable recommendations for the production of concrete that provides resistance to the various destructive processes encountered in field exposures. ACI symposia on durability of concrete, in 1975, 1987, 1991, 1994, and 1997 resulted in 17, 111, 70, 66, and 81 papers, respectively, on various theoretical and operational aspects of producing durable concrete [13–17].

In the absence of contact with aggressive fluids or incorporation of aggregates susceptible to detrimental expansion by reaction with alkalies in cement, the resistance of concrete to weathering is determined by its ability to withstand the effects of freezing and thawing in the presence of moisture.[2]

The testing of concrete for resistance to freezing and thawing had its genesis in similar tests on building stones reported as early as 1837 by Vicat [18]. It was only natural that the advent of "artificial stone" in the form of portland cement concrete would result in corresponding evaluations. Few published tests from the 19th century survive, but when freezing equipment became commercially available for food processing in the 1930s, the reporting of such testing increased. With the recognition that tests conducted in a variety of freezers designed for other purposes naturally gave variable results, several agencies such as the U.S. Bureau of Reclamation, the Corps of Engineers, the Bureau of Public Roads (now the Federal Highway Administration), the Portland Cement Association, and the National Sand and Gravel Association (now the National Aggregates Association) constructed specialized equipment for testing concrete by freezing and thawing. Descriptions of some of this equipment will be found in reports by the agencies [19–24]. These efforts marked the beginning of a systematic quest for standardization and an understanding of factors influencing the resistance of concrete to freezing and thawing.

There are currently two standard test methods under the jurisdiction of ASTM Committee C9 on Concrete and Concrete Aggregates intended to aid in evaluating the resistance of concrete to freezing and thawing. In addition, several petrographic procedures are standardized that provide invaluable information for predicting the resistance of concrete to freezing and thawing and for interpreting the results of exposure in either the laboratory or under field conditions. Two older freezing and thawing methods have been discontinued. The presently standardized methods as well as those discontinued have evolved over more than 60 years and reflect the inevitable compromise between the need for rapid assessment of resistance to weathering and the difficulty of translating the results from accelerated laboratory testing to the varied conditions encountered in field exposures.

Of the currently standardized methods, ASTM Test Method for Resistance of Concrete to Rapid Freezing and Thawing (C 666) covers the exposure of specimens to cyclic freezing and thawing. This test, the most widely used of those available, is a consolidation of two earlier methods (ASTM C 290 and C 291) and provides for two procedures: Procedure A in which both freezing and thawing occurs with the specimens surrounded by water, and Procedure B in which the specimens

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[2] Attack by aggressive fluids and cement-aggregate reactions are treated elsewhere in this publication.

freeze in air and thaw in water. ASTM Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals (C 672) provides a procedure for evaluating the effect of deicing chemicals on concrete and the effectiveness of modifications of the concrete or of surface coatings in mitigating the detrimental influence of such chemicals. Two of the initially approved standards were discontinued in 1971. These methods, ASTM Test for Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (C 292) and ASTM Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310), both of which provided for a single cycle every 24 h, were dropped because of lack of use. Another standard and a recommended practice were discontinued in 2002, also because of lack of use. These are ASTM Test for Critical Dilation of Concrete Specimens Subjected to Freezing (C 671), which employed a single cycle of cooling through the freezing point with specimens that are continuously wet, and the ASTM Practice for Evaluation of Frost Resistance of Coarse Aggregates in Air-Entrained Concrete by Critical Dilation Procedures (C 682).

While much is still to be learned, an extensive body of knowledge has been developed that permits evaluations of concrete to aid in minimizing premature deterioration from environmental factors.

While an extensive discussion of the theory and historical development of freezing and thawing tests is beyond the scope of this summary, a brief treatment is necessary. The reader should consult the cited references for additional information on these subjects.

Historical Evolution

As early as 1837, Vicat, in his famous *Treatise on Calcareous Mortars and Cements*, reported the results from experiments by Brard [25] to distinguish the building stones that were injured by frost from those that were not [18]. These tests were conducted "by substituting for the expansive force of the congealing water, that of an easily crystallizable salt, the sulfate of soda." In 1856, Joseph Henry reported testing, by 50 cycles of freezing and thawing, samples of marble used in the extension of the U.S. Capitol [26].

During the two decades before and those after the turn of the 20th century, various tests of stone, concrete, brick, and other porous materials were reported. In 1928, Grun [27] in Europe and Scholer [28] in the United States reported results from accelerated freezing and thawing of concrete in the laboratory. Following Scholer's initial paper, accelerated laboratory testing greatly increased. In 1936 [29], 1944 [30], and again in 1959 [31], the Highway Research Board (HRB) Committee on Durability of Concrete reported results of cooperative freezing and thawing tests designed to identify factors that influence resistance of concrete to freezing and thawing. The first two cooperative test series were conducted before any methods were standardized. The 1936 series concentrated on the influence of cement using mortar prisms incorporating ten commercial cements. The 1944 series of tests used concrete specimens. Both of these series emphasized the necessity for carefully regulating the methods of making and curing the specimens, the air content of the specimens, the degree of saturation of the aggregates at the time of mixing the concrete, and the degree of saturation of the concrete at the time of freezing.

In 1951, ASTM Committee C9 formed Subcommittee III-0 on Resistance to Weathering (subsequently C09.03.15 and currently C09.67) with the major responsibility for proposing methods for evaluating the resistance to freezing and thawing of aggregates in concrete and standardizing the methods of tests. Drawing heavily on the results of the HRB cooperative test series and the experience of several laboratories that had developed specialized equipment for conducting freezing and thawing tests, ASTM approved four tentative test methods in 1952 and 1953 that later became standards. These methods were largely representative of the methods and procedures then in use by the membership of Subcommittee III-0. The general characteristics of the methods are reflected in their titles: ASTM Test for Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (C 290), ASTM Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291), ASTM Test for Resistance of Concrete to Slow Freezing and Thawing in Water or Brine (C 292), and ASTM Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310). The two slow tests were adopted to cover tests usually conducted in conventional freezers with manual transfer of specimens between the freezing chamber and thawing tank. These methods were dropped in 1971 because neither was in general use nor required by any other ASTM specification. In 1971, the two rapid tests (ASTM C 290 and C 291) were combined as two procedures (A and B) in a single test (ASTM C 666).

When ASTM C 666 was originally adopted, deterioration of specimens was evaluated only by the resonant frequency method, that is, ASTM Test Method for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215). In 1984, length change was incorporated as an optional, additional means for that evaluation (length change measurements are made with the apparatus described in ASTM Specification for Apparatus for Use in Measurement of Length Change of Hardened Cement Paste (C 490).

In the third cooperative test series, initiated in 1954 and reported in 1959 [31], soon after standardization of the four methods, 13 laboratories participated using three concrete formulations: a concrete with good-quality coarse aggregate and adequate air entrainment, one with good-quality coarse aggregate and deficient air entrainment, and one with poor-quality coarse aggregate and adequate air entrainment.

Arni [4] summarized the general conclusions from the 1944 and 1954 test series as follows:

1. Methods involving freezing in water were more severe, that is, produced failure in fewer cycles, than were those involving freezing in air.
2. Rapid freezing was more severe than slow freezing when done in air but not when done in water.
3. Rapid freezing and thawing in water (ASTM Test C 290) appeared to do the best job of detecting a difference between concretes both of which had high durability.
4. Only the slow freezing in air and thawing in water method (ASTM Test C 310) was able to discriminate adequately between concretes of low durability.
5. In general, the four methods tended to rate different concretes in the same order of durability when there was a significant difference."

One of the difficulties with the four methods that was emphasized especially by the HRB cooperative programs was poor repeatability and reproducibility of results within and between laboratories. Good reproducibility generally was obtained only for concretes that were very high or very low in durability. For concretes in the middle range, a wide spread in results was obtained. While the variabilities were large in the middle range, they were amenable to establishing a precision statement that has been incorporated in ASTM C 666, as discussed later.

As noted earlier, Subcommittee III-0 was formed initially in 1951 with the primary mission of developing freezing and thawing tests to be applied to the evaluation of coarse aggregates. In 1961, the subcommittee reported that standardization of such tests was not warranted because of the high levels of variability associated with the existing methods. However, in an unpublished report, the subcommittee did outline procedures for using ASTM C 290 if evaluations of coarse aggregates were required [32].

Lack of a standardized test method for evaluating resistance to freezing and thawing of coarse aggregates in concrete coupled with theoretically based criticisms of the cyclic methods, particularly by Powers [33], as discussed later, led to the development of methods designed to determine the length of time required for an aggregate to become critically saturated in concrete. Critical saturation was defined as when specimens exposed to continuous soaking and subjected to a cycle of cooling through the freezing point exhibited dilation greater than a specified value. This method was first used by the California Department of Highways [34] and later was refined and extensively evaluated by Larson and his co-workers [35]. The literature survey prepared by Larson et al., as part of the research, is a particularly valuable reference on all aspects of freezing and thawing studies related to aggregates [8]. As an outgrowth of this work, ASTM C 671 was approved in 1971 along with ASTM C 682, which provides guidelines for applying ASTM Test C 671 to the evaluation of coarse aggregates. ASTM C 671 and ASTM C 682 were both dropped in 2002, again because neither was in general use nor required by any other ASTM specification.

More recently, agencies studying "D-cracking" of concrete have generally found a modification of the older procedure (ASTM C 666) to be useful in identifying coarse aggregates susceptible to this type of deterioration [36]. According to Stark [37], for example, aggregates susceptible to causing distress in concrete pavements can be identified by freezing and thawing concrete in water at a rate of two cycles per day. Equipment, specimen preparation, and procedures vary considerably, and no standardized failure criterion is available. Agencies desiring to use these procedures for accepting or rejecting aggregates develop failure criteria by relating the freeze-thaw test results to field performance.

Concern with surface mortar deterioration or deicer scaling, particularly on highway and bridge deck pavements, led in 1971 to standardization of ASTM C 672, which combined features of methods that had been developed and used by various agencies for a number of years. The method uses blocks fabricated to permit ponding of water on surfaces that are subjected to freezing and thawing in the presence of various deicing agents.

Of the methods, ASTM C 666 continues to be the most widely used. Specialized equipment is commercially available for conducting the tests under controlled conditions, but the essential elements of the method are those that have been used for more than a century.

Theoretical Considerations

Cyclic freezing and thawing tests were developed on a pragmatic rather than a theoretical basis. It was assumed that the destruction resulted from the 9 % volume expansion accompanying the conversion of water to ice, and that this process was reproduced artificially in the laboratory environment. Powers and his co-workers, from their comprehensive study of the structure of cement paste in 1945 [38], advanced the hypothesis that the destructive stress is produced by the flow of displaced water away from the region of freezing, the pressure being due to the viscous resistance of such flow through the permeable structure of the concrete. According to this theory, when the flow path exceeds a critical length, the pressure exceeds the strength of the paste. Such flow would occur when the water content exceeds the critical saturation point. This concept is called the hydraulic pressure theory. The theory was amplified in 1949 [39] to explain the beneficial influence of entrained air. Since the resistance to flow at a given rate is proportional to the length of the flow path, the air bubbles were conceived as spaces into which the excess water produced by freezing could be expelled without generating destructive pressures. Powers calculated a critical dimension of the order of 0.25 mm (0.01 in.), a value that was approximately the same as that suggested by Mielenz and his co-workers from experimental studies [40]. Powers's initial studies suggested that the hypothesis advanced by Taber [39] to explain frost heaving of soils did not apply to mature concrete. According to this hypothesis, the stress is produced not by hydraulic pressure, but by the segregation of ice into layers that enlarge as unfrozen water is drawn toward the region of freezing rather than forced away from it.

Studies by Verbeck and Landgren [42], as well as those of Powers [43], make clear that the paste and aggregate should be considered separately when explaining the resistance of concrete to freezing and thawing. This is because the paste not only may become critically saturated by moisture from external sources but also must withstand pressure generated by water expelled from the aggregate particles during freezing.

The temperature at which water freezes in various pores within the paste decreases with the size of the pore so that, even if the concrete is at a uniform temperature throughout, the water will be at various stages of conversion to ice. As the water freezes, the solution in the pore becomes more concentrated. The existence of solutions of various concentrations in the pores of the paste causes unfrozen water to move to the site of freezing in order to lower the concentration made higher at the freezing site than the more dilute solution of the unfrozen water. This flow generates stress somewhat like osmotic pressures, hence, the designation "osmotic pressure hypothesis."

Powers, in a subsequent summary of his and other research [44], concludes that all three of the theories, with some modification, are required to account for the behavior of concrete subjected to freezing and thawing.

When freezing and thawing takes place in the presence of deicing chemicals, localized failures of the exposed surfaces occur that is called "scaling," or surface mortar deterioration. Except where the concentration of deicing chemicals is high enough to cause chemical attack, scaling results from freezing and thawing. Various theories have been advanced to explain the increased severity of the damage as compared with freezing and thawing in water. Browne and Cady [45] have summarized these theories and their own experiments. Although definitive answers have not been obtained, the mechanism is probably most influenced by concentration gradients. In addition to the lowering of the freezing temperature that accompanies increased deicer concentration, flow of water from areas of lower to those of higher concentrations generate stresses such as were described earlier. Verbeck and Klieger [46], in work confirmed by others, found that deterioration was greater for intermediate concentrations of deicing chemical (3 to 4 %) than for lower or higher concentrations (to 16 %). Such findings can be explained by generation of osmotic-like pressures accompanying the movement of water between areas of varying concentrations.

Rapid Freezing and Thawing Tests

In 1971, ASTM C 290 and C 291 were combined as Procedures A and B in ASTM C 666. The method is designated as "rapid" because it permits alternately lowering the temperature of specimens from 4 to −18°C (40 to 0°F) and raising it from −18 to 4°C (0 to 40°F) in not less than 2 h nor more than 5 h. Thus, a minimum of four and a maximum of 12 complete cycles may be achieved during a 24 h period. The conventionally accepted term of testing is 300 cycles, which can be obtained in 25 to 63 days. For Procedure A, both freezing and thawing occur with the specimens surrounded by water, while in Procedure B, the specimens freeze in air and thaw in water. For Procedure A, the thawing portion is not less than 25 % of the total cycle time, while for Procedure B, not less than 20 % of the time is used for thawing. The time required for the temperature at the center of any single specimen to be reduced from 3 to −16°C (37 to 3°F) shall be not less than one half of the length of the cooling period, and the time required for the temperature at the center of any single specimen to be raised from −16 to 3°C (3 to 37°F) shall be not less than one half of the length of the heating period. For Procedure A, each specimen is surrounded by approximately 3 mm (⅛ in.) of water during the freezing and thawing cycles, while in Procedure B, the specimen is surrounded completely by air during the freezing phase of the cycle and by water during the thawing phase. The requirements for Procedure A are met by confining the specimen and surrounding water in a suitable container. The specimens are normally prisms not less than 75 mm (3 in.) nor more than 125 mm (5 in.) in width and depth, and between 275 and 405 mm (11 and 16 in.) in length.

During the early years of testing by freezing and thawing, laboratories constructed specialized equipment. Currently, several manufacturers produce off-the-shelf or custom-built freeze-thaw equipment that meets the requirements of ASTM C 666. Capacities typically range from 18 to 80 specimens, although custom-built units have been designed for up to 120. Some of the equipment is designed for Procedure A only, while the larger units typically can be used for both A and B. Some aspects of these machines have been subject to criticism, but they do meet the needs for rapid testing within practical limits.

Deterioration of specimens is determined by the resonant frequency method, ASTM C 215. The fundamental transverse frequencies are determined at intervals not exceeding 36 cycles of exposure and are used to calculate the relative dynamic modulus of elasticity

$$P_c = \left(\frac{n_1^2}{n^2}\right) \times 100$$

where

P_c = relative dynamic modulus of elasticity, after c cycles of freezing and thawing, %;
n = fundamental transverse frequency at 0 cycles of freezing and thawing; and
n_1 = fundamental transverse frequency after c cycles of freezing and thawing.

The fundamental transverse frequency is determined with the specimens at a temperature of 5.0 ± 1.6°C (41 ± 3°F). Calculation of P_c assumes that the mass and dimensions of the specimens remain constant throughout the test. While this assumption is not true in many cases because of disintegration, the test is usually used to make comparisons between the relative dynamic moduli of specimens and P_c is adequate for the purpose.

The durability factor is calculated as

$$DF = \frac{PN}{M}$$

where

DF = durability factor of the specimen;
P = relative dynamic modulus of elasticity at N cycles, %;
N = number of cycles at which P reaches the specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is less; and
M = specified number of cycles at which the exposure is to be terminated.

Because of the danger of damage to specimen containers and other parts of the equipment, testing usually is terminated when the relative dynamic modulus of elasticity falls below 50 %.

The scope of ASTM C 666 states that "both procedures are intended for use in determining the effects of variations in the properties of concrete on the resistance of the concrete to the freezing and thawing cycles specified in the particular procedure. Neither procedure is intended to provide a quantitative measure of the length of service that may be expected from a specific type of concrete."

Procedure A is currently required in three ASTM specifications: namely, ASTM Specification for Air-Entraining Admixtures for Concrete (C 260), ASTM Specification for Chemical Admixtures for Concrete (C 494), and ASTM Specification for Chemical Admixtures for Use in Producing Flowing Concrete (C 1017). In these admixture specifications, the performance requirement is stated in terms of a "relative durability factor" calculated as

$$DF \text{ or } (DF1) = \frac{PN}{300}$$

and

$$RDF = \left(\frac{DF}{DF_1}\right) \times 100$$

where

DF = durability factor of the concrete containing the admixture under test;
DF_1 = durability factor of the concrete containing a reference admixture (or in the case of ASTM C 494, only an approved air-entraining admixture);
P = relative dynamic modulus of elasticity in percent of the dynamic modulus of elasticity at zero cycles (values of P will be 60 or greater since the test is to be terminated when P falls below 60 %);
N = number of cycles at which P reaches 60 %, or 300 if P does not reach 60 % prior to the end of the test (300 cycles); and
RDF = relative durability factor.

TABLE 1—Within-Laboratory Precisions for Averages of Six Beams Tested in Accordance with ASTM C 666

Range of Average Durability Factor	Procedure A		Procedure B	
	Standard Deviation (1S)	Acceptable Range (D2S)	Standard Deviation (1S)	Acceptable Range (D2S)
0 to 5	0.3	0.9	0.4	1.2
5 to 10	0.6	1.8	1.7	4.7
10 to 20	2.4	6.8	3.3	7.4
20 to 30	3.4	9.7	4.3	12.2
30 to 50	5.2	14.7	6.3	17.8
50 to 70	6.4	18.1	8.2	23.2
70 to 80	4.7	13.4	7.0	19.7
80 to 90	2.3	6.5	3.6	6.8
90 to 95	0.9	2.4	1.6	4.5
Above 95	0.4	1.3	0.8	2.3

ASTM C 260, C 494, and C 1017 require that the relative durability factor of the concrete containing the admixture under test be at least 80 when compared with the reference concrete. The value of 80 is not intended to permit poorer performance than the reference concrete, but rather to assure the same level of performance, with appropriate recognition of the variability of the test method. The value of 80 was established before levels of precision were established for ASTM C 666, but is consistent with the now-established precision values.

One criticism of rapid freezing and thawing tests has been variability of the results both within and between laboratories. In response to the requirement by ASTM for precision statements in all test methods, ASTM Subcommittee C09.03.15 (now C09.67) reviewed the data from major published studies. Statistical analyses of these data, as described by Arni [47], showed that the variability was primarily a function of the level of durability factor of the concrete for the ranges of *N* and *P* normally used. In 1976, a precision statement was added to ASTM C 666 for expected within-batch precision for both Procedures A and B. Values for standard deviation (1S) and acceptable range (D2S), as defined by ASTM Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials (C 670), are given in Tables 2 and 3 of ASTM C 666 for ranges of average durability factor in ten increments and numbers of specimens averaged from two through six. Values for six specimens (as required by ASTM C 260, C 494, and C 1017) are given in Table 1.

These values confirm the long-recognized facts that the variability is less for very good or very poor concrete than for concretes of intermediate durability and that results from Procedure A are somewhat less variable than those obtained from Procedure B. An example of earlier work showing the variability at different durability factors is given in Fig. 1. This figure, from Cordon [7], shows durability factors of concretes containing a variety of aggregates, and with varying cement contents, water-cement ratios, and air contents. For very low and very high air contents, the measured durability factors showed relatively small variations. At intermediate values of air content, a “transition zone” reflected much greater variability of performance. One might conclude that, for intermediate levels of performance as indicated by durability factors, an undeterminable portion of the variability reflects the real variability of concretes with such characteristics and, therefore, uniform precisions would not be expected for the entire range of durability factors.

While ASTM C 666 is specified in the ASTM Specification for Lightweight Aggregates for Structural Concrete (C 330), no minimum durability factor values are given. The specification states that “in the absence of a proven freezing and thawing test satisfactory to the purchaser.” ASTM C 666 is similarly listed as a method of sampling and testing in ASTM Specification for Concrete Aggregates (C 33), but again no minimum durability factors or other requirements are stated.

As noted, the primary measure of deterioration is the relative dynamic modulus calculated from determinations of resonant transverse frequency. Length change is noted in ASTM C 666 as an acceptable additional way of assessing deterioration. Mass loss is also sometimes used for such assessments, while determinations of reductions in tensile or compressive strength are used only infrequently because such testing is destructive.

The question of which measure of deterioration is best to use is complicated by the fact that the different available tests measure different things, and the manner and extent to which the properties measured are related to freezing-and-thawing damage, especially under natural conditions, are matters of disagreement. Thus, the particular measure used often depends on the philosophy of the laboratory using it and on the particular purpose for which the tests are being made. Mass loss measures loss of material or sloughing from the surface of the specimens. Length change is based on the fact that internal damage is accompanied by expansion rather than contraction during cooling or by a permanent dilation after a freezing and thawing cycle, as discussed by Powers [33]. Either mass loss due to sloughing or reduction in resonant frequency may occur without accompanying length change, and, in Procedure A, mass loss often occurs without a reduction in resonant frequency or expansion. Resonant frequency and expansion reflect internal disruptions that are caused by unsound aggregates or deficient air-void characteristics, while mass loss reflects primarily surface mortar deterioration.

Users of ASTM C 666 have not reached a consensus on limiting values for the method’s measurement quantities. A 1987 survey of State and Canadian provincial highway agencies indicated that seven used ASTM C 666 to qualify coarse aggregates for construction on agency projects [48]. The seven had “seven different specification limiting values, based

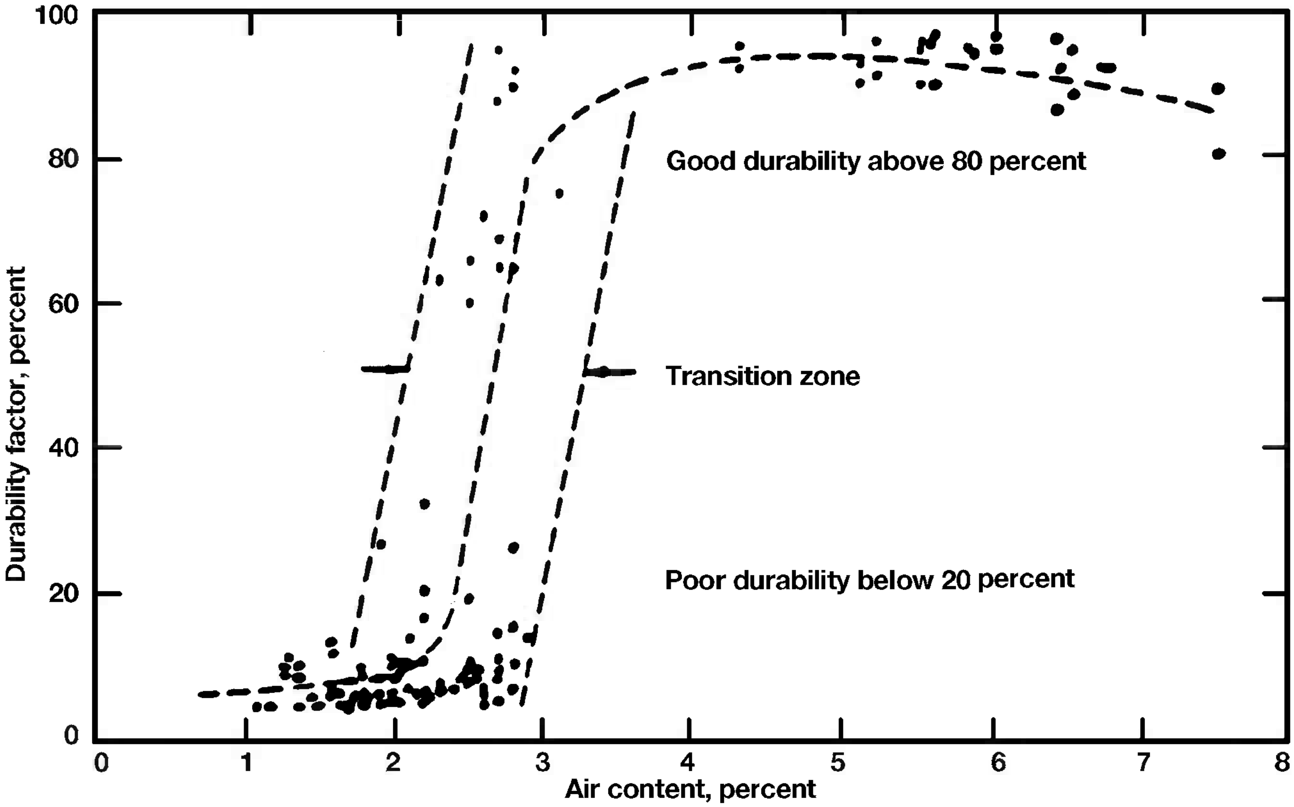

Fig. 1—Durability factors for concretes containing various aggregates, cement contents, and water-cement ratios, as a function of air content (after Cordon [7]).

on the fact that each had a unique way of testing regarding aggregate grading, moisture conditioning, cement contents, air contents, coarse aggregate contents, curing methods and time, cycle length, number of cycles, method of measuring deterioration, failure criteria, and number of specimens constituting a test." Further, three agencies used durability factor, three used length change or another measure of expansion, and one used both for specification limits. A standard practice, which would include research-supported limiting values, is clearly desirable.

One important potential influence on ASTM C 666 results is the effect of the container used to hold the specimen during testing with Procedure A. Rigid containers have the potential to damage specimens and are not permitted. A note explains further. "Experience has indicated that ice or water pressure, during freezing tests, particularly in equipment that uses air rather than a liquid as the heat transfer medium, can cause excessive damage to rigid metal containers, and possibly to the specimens therein. Results of tests during which bulging or other distortion of containers occurs should be interpreted with caution." This situation is particularly noticeable when the specimens are exposed with the long dimension vertical. Ice forms quickly on the open top rather than uniformly along the long dimension of the container. Pressures from continued conversion of water to ice between the specimen and container cannot be relieved, with consequent bulging of the container. Various adjustments have been made to mitigate this problem, including exposure of the specimens in containers with the long dimensions horizontal to provide a larger open area; inclusion of flexible windows, or corners, in containers; or inclusion of a rubber ball in the bottom of containers to absorb the pressures. All of these have been reported to overcome the problem by some and not by others. That the degree of restraint offered by the container influences the number of cycles required to reach a specified level of relative dynamic modulus is clear, as shown by Cook [49], who found that the number of cycles necessary to reach a given level of durability factor was increased dramatically when the specimens were tested in rubber containers as compared with containers made of steel. One agency has minimized the container influence by using cylindrical specimens and surrounding them with rubber boots [48].

Another important influence on the results is the degree of saturation of the concrete and the aggregates both at the time of mixing and throughout the course of the testing. The specifications citing the use of ASTM C 666 require that testing begin after 14 days of moist curing. (A note in ASTM C 666 itself says that the tests should be started when the specimens are 14 days old "unless some other age is specified.") The mixing procedures referenced in ASTM Test Method for Making and Curing Concrete Test Specimens in the Laboratory (C 192) require that coarse aggregates be immersed for 24 h prior to mixing. Thus, the comparatively high degree of internal saturation and the early age at which testing begins result in a relatively severe test when compared with field exposures in which a period of drying normally occurs before exposure to freezing and thawing. Even a brief period of drying greatly improves resistance to freezing and thawing, since it is difficult to resaturate concrete that has undergone some drying. Strategic Highway Research Program researchers [11] confirmed the effect of drying. They found that Procedure B results approach those from Procedure A if the specimens are wrapped in terrycloth during the test; this keeps the outer surface of the concrete moist, that is, at or near saturation, rather than allowing it to dry out during the freezing-in-air portion of the test cycle.

Still another influence on freezing and thawing tests is the use of salt water (typically 2 % sodium chloride in water) as the freezing-and-thawing medium. Some agencies have used salt water since that is the medium encountered in many field situations where water ponds on structures regularly treated with deicers. Use of salt water in Procedure A increases the severity of the test beyond that attained with the use of fresh water.

Philleo has taken ASTM C 666 to task, recommending that it be "modified or replaced by a more realistic standard for judging the acceptability of concrete for field applications" [50]. He points out that most concrete undergoes both drying and curing longer than 14 days before encountering its first freezing. Nevertheless, accommodating the many possible variations of saturation that might be encountered is impractical so that the most consistently reproducible condition is that of continued moist curing.

Because the testing is initiated at a fixed age, considerable variation of strength at the time of exposure to freezing and thawing may be encountered with cements of different strength gain characteristics for concrete with sound aggregates and satisfactory air-void characteristics. There is not a great body of data on the influence on resistance to freezing and thawing of strength at the time that exposure begins. Buck et al. [51] reported tests indicating that, because of its relationship with the amount of freezable water, a given level of maturity (strength) was necessary to provide an acceptable degree of frost resistance as indicated by a durability factor of 50 for concrete containing satisfactory aggregates and entrained air. They cited earlier work by Klieger [52], who reported similar findings in his studies of salt scaling. Consideration of strength at the time of initial freezing is particularly important in testing concrete made with blended cements and pozzolanic supplementary cementitious materials that gain strength more slowly than concrete without such materials. This influence is minimized when the evaluations are made by comparing concretes made with similar materials as required in ASTM C 260 and C 494.

Visual examination of specimens during ASTM C 666 cycling may also give warnings of the likelihood of popout problems in a concrete. Popouts are shallow, usually conical spalls of the concrete through aggregate particles due to internal pressure and can be attributed to defects in the aggregate. The small size of the specimens in ASTM C 666 has been criticized because a large piece of popout-producing aggregate in the center of a relatively small specimen could cause it to fail; in the field, the popout material would presumably only cause superficial surface defects [53].

Cyclic freezing and thawing methods were developed and applied only to laboratory-mixed concrete until 1975, when they were extended to cores or prisms cut from hardened concrete. Experience with testing of specimens from hardened concrete is limited; the results of a 1987 survey of State and Canadian provincial highway agencies gave no indication that any were using anything other than specimens cast to size specifically for freeze-thaw testing [48].

Dilation Methods

In 1955, Powers published a critical review of existing cyclic methods for freezing and thawing tests [33]. He was particularly critical of what he considered unrealistically high freezing rates of from 6 to 60°C/h (10 to 100°F/h) as compared with natural cooling rates that seldom exceed 3°C/h (5°F/h). He also noted the significance of moisture conditioning of aggregates and concretes in the test methods that generally provided for only saturated concrete as compared with natural exposures where some seasonal drying is possible.

He stated that durability was not a measurable property but that expansion that occurred during a slow cooling cycle when the concrete or its aggregates became critically saturated was measurable and would provide an indication of potential resistance to damage by freezing and thawing. He proposed that specimens be prepared and conditioned so as to simulate field conditions and then be subjected to periodic slow-rate freezing and storage in water at low temperature between freezing exposures. Concrete subject to frost damage should reach some critical saturation level, after which it would expand on freezing. The length of time required to become critically saturated would be compared with the field exposures to be encountered. If the period during which freezing would be expected was less than the time to reach critical saturation, then no damage would be expected. As opposed to a single durability factor, the time to critical saturation could be more readily interpreted for various field exposures.

The California Division of Highways was first to report, in 1961, a practical application of Powers's proposed method [34]. They developed specimen preparation and conditioning methods, testing and measuring techniques, and performance criteria. The method was used to evaluate several aggregates for a major highway construction project. The aggregates judged acceptable by the California procedure would have been rejected by other conventionally accepted criteria. The concrete is now more than 30 years old; sections have been overlaid because they are structurally inadequate, but the freeze-thaw performance of all of the concrete has been reported as continuing to be satisfactory.

The procedures proposed by Powers and the method developed in California were extensively studied and refined by Larson and others [35]. In 1971, ASTM C 671 was standardized. This method provides for cylindrical specimens 75 mm (3 in.) in diameter and 150 mm (6 in.) long that are stored in water at 1.7 ± 0.9°C (35 ± 2°F). At two-week intervals, the specimens are cooled in water-saturated kerosene at a rate of 2.8 ± 0.5°C/h (5 ± 1°F/h). During the cooling cycle, the specimen is placed in a strain frame to permit measurement of length change. A typical plot of length change versus temperature is shown in Fig. 2. Prior to critical saturation, the length change will proceed along the dashed curve without dilation. Critical dilation is defined as a sharp increase (by a factor of 2 or more) between dilations on successive cycles. Highly frost-resistant concrete may never exhibit critical dilation. ASTM C 682 was also standardized in 1971. This practice is based largely on the work of Larson and his coworkers. Procedures essentially in accordance with ASTM C 671 have been used by Buck [54], and he found that a specimen that is frost resistant will not show increasing dilation with continuously decreasing temperature; it will show some limited initial dilation as all moist specimens do, but dilation will not continue throughout the cooling. In addition to the ASTM C 671 criterion for critical dilation in terms of increase from one cycle to another, he suggested that a criterion for critical dilation applicable to results of a single test be as follows:

(a) If the dilation is 0.005 % (= 50 millionths) or less, the specimen may be regarded as frost resistant, that is, the dilation is not critical.

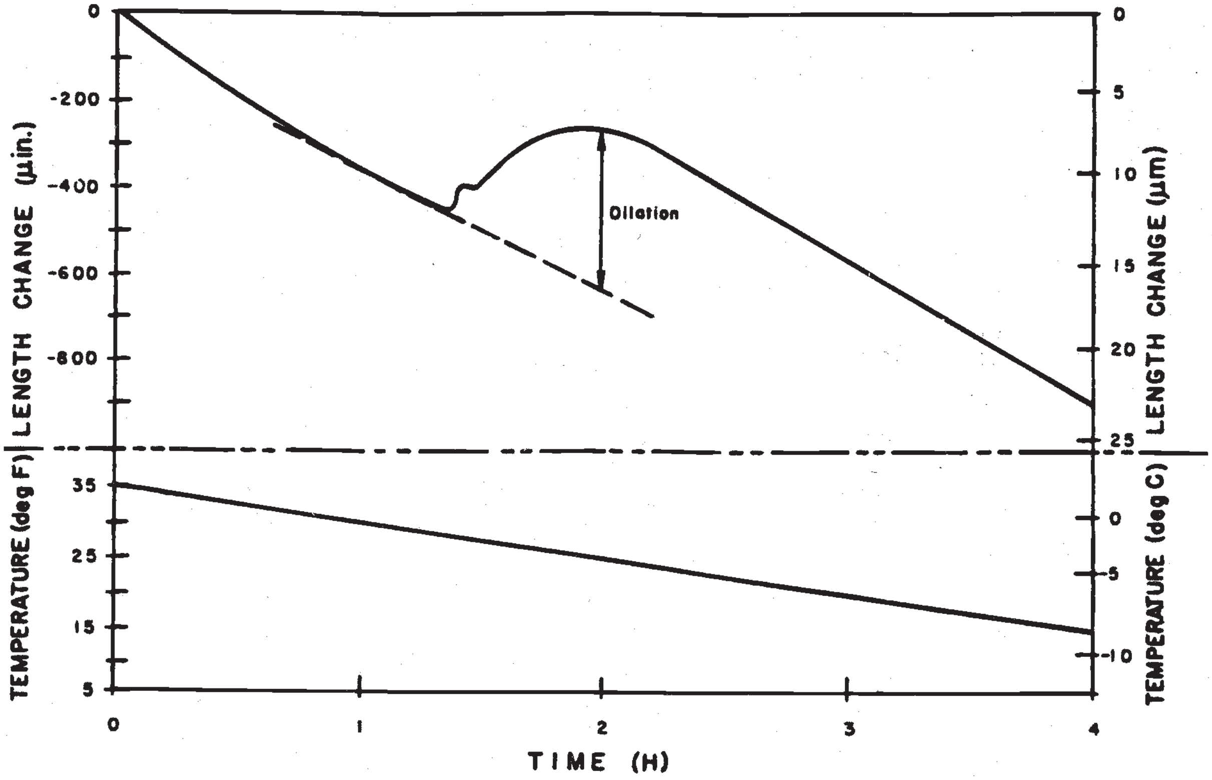

Fig. 2—Typical length change and temperature charts.

(b) If the dilation is 0.020 % (= 200 millionths) or more, the specimen may be regarded as not frost resistant, that is, critical dilation has been exceeded.

(c) If the dilation is in the range between 0.005 and 0.020 %, an additional cycle or more should be run.

ASTM C 671 and C 682 were not used extensively and, consequently, were discontinued in 2002. The 1987 survey of State and Canadian provincial highway agencies indicated that only one was using ASTM C 671 to any extent [48]. This agency has subsequently stopped using the method. Although the apparatus is comparatively inexpensive and larger numbers of specimens can be processed than with ASTM C 666 equipment, significant storage capacity is required. The procedures of ASTM C 682 were extensive and complex, largely because of requirements designed to bracket a broad range of potential exposure conditions. It was suggested that aggregates and concrete be "maintained or brought to the moisture condition representative of that which might be expected in the field." However, it was noted that "aggregate moisture states other than dry or saturated are very difficult to maintain during preparation of specimens. Reproducibility of overall test results is likely to be affected adversely by variability in aggregate moisture." While the complexity of the evaluation procedure limited its general applicability, the procedures may have been justified where large projects or economic consequences of detailed aggregate evaluation were warranted. ASTM C 671 included the statement that it "is suitable for ranking concretes according to their resistance to freezing and thawing for defined curing and conditioning procedures." However, both ASTM C 671 and C 682 warned that the significance of the results in terms of potential field performance would depend upon the degree to which field conditions could be expected to correlate with those employed in the laboratory.

Scaling Resistance

In the early 1960s, it became apparent that the increasing use of deicing chemicals as part of a "bare pavement" policy adopted for the nation's highways was being reflected in widespread surface scaling of pavements and bridge decks. It has long been known that dense, high quality concrete, with adequate entrained air and with adequate curing and a period of drying before the first application of deicing agents, is essential in preventing damage [12]. Widespread scaling demonstrated that all of these requirements were not being met consistently and brought forth a plethora of remedial or preventive products including admixtures, surface treatments, and curing agents. In 1971, ASTM C 672 was standardized. The test is based on the experience of a number of agencies who used blocks that were fabricated to permit ponding of water on one surface and that could be exposed to freezing and thawing in the presence of deicing agents. The test "covers determination of the resistance to scaling of a horizontal concrete surface subject to freezing and thawing cycles in the presence of deicing chemicals. It is intended for use in evaluating the surface resistance qualitatively by visual examination. This test method can be used to evaluate the effect of mixture proportioning, surface treatment, curing, or other variables on resistance to scaling. This test method is not intended to be used in determining the durability of aggregates or other ingredients of concrete." The report of ACI Committee 201 says, on the other hand, the "use of ASTM C 672 will demonstrate the acceptability or failure of a given concrete mixture" [12].

The specimens must have a surface area of at least 0.045 m^2 (72 $in.^2$) and be at least 75 mm (3 in.) deep. The specimens are placed in a freezing space after moist curing for 14 days and air storage for 14 days. Provisions are included for applications of protective coatings if desired at the age of 21 days. The method calls for covering the surface with approximately 6 mm (¼ in.) of a solution of calcium chloride and water having a concentration such that each 100 mL of solution contains 4 g of anhydrous calcium chloride. Modifications of the deicer and application procedures, including freezing of water and addition of the solid deicer, are allowed where there is need to evaluate the specific effect.

The specimens are cycled through a freezing environment for 16 to 18 h, followed by laboratory air for 6 to 8 h. While the method describes laboratory procedures, it has been used for outdoor exposures as well.

The specimens are rated visually according to a scale of 0 (no scaling) to 5 (severe scaling) after 5, 10, 15, 25, and every 25 cycles thereafter. Some investigators have measured the mass of the detritus, but this is not required by the method.

As noted in the method, the ratings are ranks, and as such may not be subjected appropriately to analyses based on the calculation of averages and standard deviations or other techniques that assume continuous distributions. If groups of similar specimens are to be reported or compared with other groups, such nonparametric quantities as the median and range may be used.

Experience with ASTM C 672 generally has been satisfactory for evaluating the variables for which it was developed.

Other Weathering Processes

Other weathering processes that have been suspected of causing deterioration include heating and cooling and wetting and drying. As noted, chemical attack and alkali-aggregate reactions are treated elsewhere in this publication. Although cases have been reported where deterioration was attributed to aggregate with an abnormally low coefficient of thermal expansion [55] or where the freezing and thawing resistance was influenced by aggregates with different coefficients of thermal expansion [56,57], “thermal incompatibility” is now generally believed to have at most a minor effect on concrete durability within the normal temperature range. Elevated temperatures such as are encountered in certain parts of nuclear construction are beyond the scope of this paper.

While wetting and drying induce variations in moisture content that influence the resistance to freezing and thawing, aggregate cracking from excessive drying shrinkage, and an increased concentration of dissolved salts, all of which reduce resistance to weathering, the writers are not aware of cases where alternate wetting and drying per se have caused deterioration.

Summary

The resistance of concrete to weathering in the absence of chemical attack or detrimental cement-aggregate reactions depends on its ability to resist freezing and thawing. Dry concrete will withstand freezing and thawing indefinitely, whereas highly saturated concrete exposed to particularly severe conditions such as hydraulic head or very low temperatures may be severely damaged in a few cycles. This damage is more likely to occur when the air content of the concrete is at the lower end of the recommended range. Research and experience have shown that resistance to freezing and thawing requires a low water/cement (w/c) ratio, an adequate volume of entrained air with the proper void distribution and characteristics, and exposures that reduce the opportunity for critical saturation. While adequate air entrainment with proper air-void parameters protects the paste, it may not overcome the effect of aggregate that is susceptible to damage by freezing and thawing. Direct translation of results from laboratory freezing and thawing tests is difficult at best because of the variety of exposures encountered, but the currently approved standards are very useful when properly conducted and interpreted and have undoubtedly resulted in a significant improvement in the resistance of concrete to weathering from natural forces.

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16

Corrosion of Reinforcing Steel

Neal S. Berke[1]

Preface

THIS VERSION OF CHAPTER 17 IN *ASTM STP 169C* IS an updated version of the chapter written by William F. Perenchio. In *ASTM STP 169B*, this chapter was authored by P. Cady. In *ASTM STP 169A*, this chapter was authored by B. Temper. The first three sections remained as they were with a change in Ref 1 to provide a source of more information on the subject. Substantial changes are included in the following sections to reflect more knowledge that is now available on the use of corrosion inhibitors, epoxy-coated steel, sealers, cathodic protection, and new steels. The main value of the chapter is to provide an introduction to the field and numerous references for a more in-depth understanding. The number of references has increased from 28 to 53 reflecting the increase in information available today.

Note–as in the original versions, the effects of carbonation are not addressed as chloride is the major problem and design for chloride resistance will provide protection against carbonation [1]. Cracking can cause chlorides to reach reinforcement earlier, resulting in corrosion starting at an earlier time, so it should be minimized.

Introduction

Many metals, including reinforcing steel, are normally well protected against corrosion by embedment in high-quality concrete. However, many of the bridges, parking garages, and marine structures in this country have been severely damaged by corrosion of reinforcement and the resultant increased volume. These and other structures have suffered damage due to the penetration of chloride in sufficient amounts to the depth of the reinforcing steel. In the case of bridges and parking structures, the source of the chloride is usually deicing salts. In marine structures, it is seawater. In others, it may be chemical admixtures added to the concrete that contain chlorides for set acceleration.

Cracks are not necessary for chloride penetration into the concrete. Salts are capable of penetrating solid concrete along with the water in which they are dissolved. Variations in the amount of chloride, or moisture, or oxygen at different points along a reinforcing bar, or between different bars, can cause voltage differentials to develop that greatly increase the rate of corrosion. The increased volume due to the development of corrosion products can cause spalling of the concrete cover, and sufficient reduction in the cross section of the bar to render a structure incapable of safely supporting its design loads.

Such chloride-induced corrosion is a serious problem. Durable repair procedures are available; however, all are expensive. Significant improvements in methods of protecting embedded metals from corrosion at the design stage have been made in recent years. This chapter will discuss the mechanisms of corrosion, typical damage caused by it, investigative techniques for evaluating its extent, means of preventing it in new construction, and remedial measures.

Mechanisms of Corrosion

When a metal corrodes, it returns to its natural state, which is usually the oxide or the hydroxide. Metals tend to do this because, in their metallic state, they are at a higher energy level. Materials tend to seek lower energy levels; hence, the tendency to corrode. Few metals (except for the noblest ones: platinum, gold, silver) are ever found in nature in the metallic (elemental) state.

Because of this tendency to return to the state from which they were refined, one might expect that metals would be unusable, quickly disappearing while in service. However, a passive film of tightly adhering corrosion products usually forms on steel in alkaline environments, as in concrete, that protects the remaining steel from the corrosive conditions [1]. Chloride ions can disrupt the passive film through a process known as pitting and this is the initiation process of corrosion of steel in concrete in the presence of chlorides [1]. A good overall treatment of the corrosion of steel in concrete is given in Ref 1.

Any chemical action can be regarded as electrochemical, since it involves transfer or displacement of electrons. However, the term is sometimes applied only to cases of corrosion where anodes and cathodes are some finite distance apart, making the flow of electrical current over measurable distances a part of the process [2]. Corrosion, of course, can take place on reinforcing steel that is exposed only to air and rain. This is, therefore, sometimes termed atmospheric corrosion [3,4]. An electrolyte is not necessary; however, even a thin film of rainwater can act as one, carrying ions and completing the electrical circuit. In this case, the anodes and cathodes are contained in the same bar, separated by only small distances.

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Concrete provides protection for embedded metals, if it is high quality and if the depth of cover is about 2 in. (50 mm) or more. However, if either the quality or the depth of cover, or both, are low enough, corrosion of steel can occur when:

1. the concrete cover becomes carbonated, dropping the pH from about 13 to about 9; or
2. chloride ions have penetrated into the concrete such that the concrete adjacent to the steel contains at least 1.3 lb of chloride ions/yd^3 (0.77 kg of chloride ions/m^3).

In the case of reinforcing steel, if the corrosion develops because the cover concrete has become carbonated, the rate of corrosion is relatively slow. If, however, the corrosion is instigated by the presence of chloride ions, the rate can be many times as fast as atmospheric corrosion [2] and it causes much greater damage. This type of corrosion involves voltage differences and transfer of electrons and electrical current, in addition to various chemical reactions.

Chloride-induced corrosion is a very common cause of concrete deterioration in all types of structures along the sea coast and in northern states where salt is used to remove ice from roads, streets, parking structures, balconies on high-rise buildings, etc. The damage occurs at a more rapid rate in warmer climates because it is due primarily to chemical reactions. Most chemical reactions double in rate for every 18°F (10°C) increase in temperature [3].

A simplified electrolytic cell is shown in Fig. 1. In the figure, a voltage difference has been set up by a low salt content surrounding the iron electrode on the left side and a high salt content on the right. The difference in voltage could be increased by bubbling oxygen (or air) into the left side. It is this difference in voltage that drives the cell and causes the corrosion to proceed rapidly.

The primary reactions that take place at each electrode are shown below the cell. Corrosion, or the conversion of metallic iron into ferrous ions in solution, occurs at the anode, leaving two electrons behind for every iron ion released. These remain within the electrode and, if no suitable cathode is available, will eventually stop the corrosion reaction. This is called anodic polarization. If a cathode is available and is connected to the anode by a material, such as metal or carbon, that is capable of transferring electrons (carrying electrical current),[2] these extra electrons can be shed by the reaction shown. Oxygen and water combine with the electrons to produce hydroxyl ions. The electrical circuit is completed through the solutions surrounding the electrodes and the semi-permeable membrane by the movement of ions such as OH^-, Cl^-, Na^+, K^+, and Ca^{++}.

The common (or now, old-fashioned) flashlight battery is an example of an electrolytic cell that produces a usable product, light. (This is called the Leclanche' cell in physical chemistry textbooks.) The case of the battery is made of zinc metal and the center pole is made of carbon. Between them is a mixture of manganese oxide, pulverized carbon, ammonium chloride, and water. It is of paste consistency rather than a solution, but still acts as an electrolyte. The battery operates by the zinc going into solution and leaving electrons behind, which travel to the carbon cathode in the center of the cell. This flow of electrons excites the filament in the flashlight bulb, producing light.

A more detailed and complete description of electrolytic cells is given by Uhlig [2].

Extending this battery concept to a more practical problem, that of corrosion of reinforcing steel in concrete, consider the schematic diagram in Fig. 2. This figure depicts the typical conditions found in a bridge deck or a floor in a parking structure, in areas where deicing salts are used. The salts typically consist essentially of sodium chloride.

The top layer of steel is termed the anodic steel because this is where most of the corrosion occurs. It becomes anodic because it is much closer to the source of water and chloride ions; therefore, the surrounding concrete becomes inundated with more of each than does the concrete near the bottom steel, which becomes cathodic. Therefore, the iron that makes up the majority of the steel composition goes into solution,

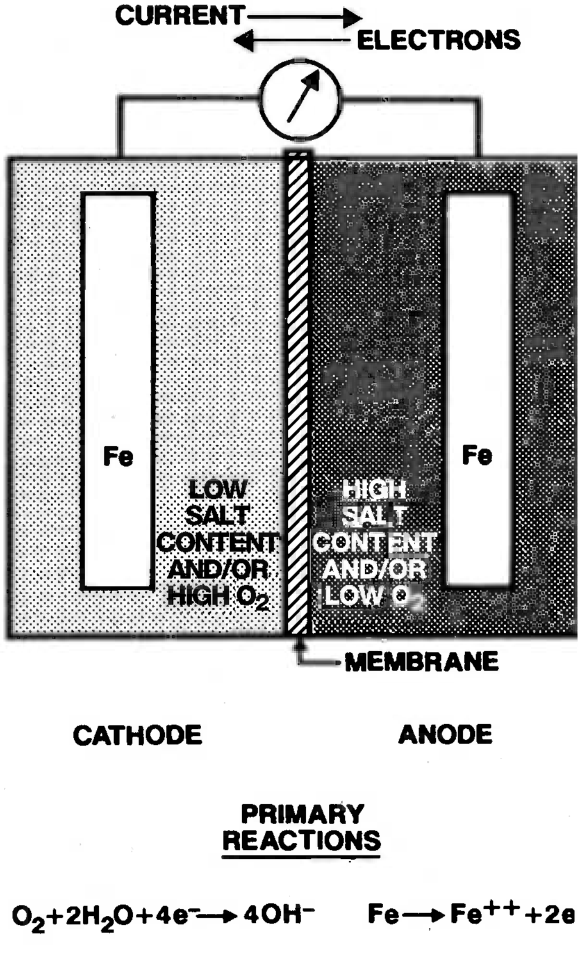

Fig. 1—Electolytic cell.

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[2] Although rubbing two dissimilar metals together was found to attract bits of paper or pith, and sparks had been made to jump gaps, for over a hundred years previously, in 1729, Stephan Grey found he could transfer this "force" along a damp thread several hundred feet long and attract a feather at the other end. Because of this capacity for flow, electricity was thought to be some kind of fluid, or two different fluids. Shortly thereafter, Charles Dufay named these two "vitreous" and "resinous" electricity for the opposite charges produced by rubbing glass or resin.

In 1747, Benjamin Franklin, well known for his electrical experiments, suggested a "one-fluid system," calling it positive or negative depending on the direction of flow. Because he had no way of knowing which direction the current was flowing, he arbitrarily decided it was from positive to negative. One hundred years later, J. J. Thompson demonstrated that electrons flow from negative to positive, but Franklin's convention was so well established by then that we still refer to "current" as flowing in the direction opposite to electron flow. (Taken from Maxey Brooks, "Why is a Cathode Called a Cathode," *Materials Performance*, National Association of Corrosion Engineers, Vol. 30, No. 6, Houston, TX, June 1991.)

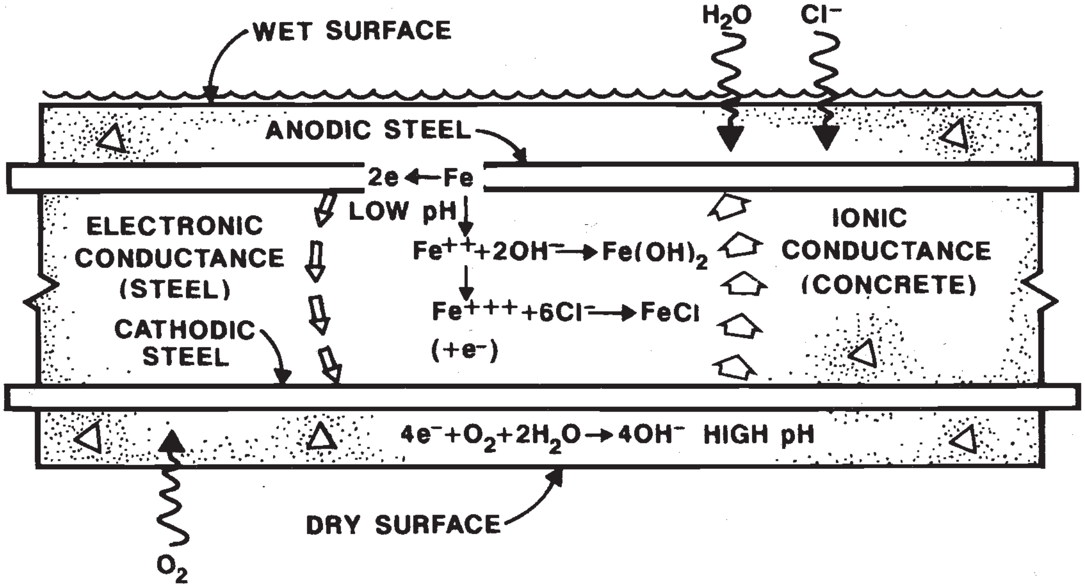

Fig. 2—Electrolytic corrosion inside concrete.

leaving electrons behind. The electrons find their way to the bottom mat by metallic continuity brought about by bent bars, bar chairs, etc. The electrons are then shed into the solution in the pores of the portland cement paste by combining with water and oxygen to produce hydroxyl ions, as indicated by the reaction shown, the same as that in Fig. 1. Ionic conductance within the concrete completes the circuit.

The voltage difference between the top and bottom mats of steel are due in part to differences in concentration of water, chloride ion, and oxygen. As previously stated, the top steel has more water and chloride in the cement pores surrounding it, while the bottom steel has more oxygen because there is less water in the concrete at that depth and below. Oxygen (and other gases) can diffuse into dry concrete much more easily than wet concrete because, with wet concrete, it must diffuse through pore water rather than pore air, a much slower process.

The preceding discussion is presented as a common example of macrocell corrosion. However, it is not meant to imply that separations of anode and cathode are always so great. Microcells can be set up along a given bar, separated only by tiny fractions of an inch. Bits of mill scale can act as cathodes that drive an adjacent anode in the steel [2]. Local chemical or physical differences in or on the metal can also cause small voltage differences. Such localized cells can result in serious pit corrosion.

Damage Caused by Corrosion

Damage to concrete, in the form of cracking, delamination, and spalling, can occur due to atmospheric corrosion; that is, the type brought on by carbonation of the cover concrete. However, this is slow to develop and, depending on the external environment, may never cause serious deterioration. The previous author was asked to visit Tiger Stadium at Louisiana State University to inspect the football stadium, parts of which were 70, 50, 40, and as little as 10 years old. The oldest portions were badly stained by reinforcing steel corrosion products, but very little deterioration was observed. The oldest concrete was also of the poorest quality. Therefore, carbonation progressed rapidly, allowing atmospheric corrosion to take place. But the same high porosity that allowed the rapid carbonation also provided space for the corrosion products, and disruptive pressures did not soon develop. Before carbonation, the steel was passivated by the high pH of the concrete. After carbonation and the resultant drop in pH, the passivation was lost.

Chloride-induced corrosion, by comparison, is usually much more destructive. It does not depend on carbonation and can develop even in good quality concrete. All that is necessary is a reasonable supply of water (75 % humidity appears to be sufficient), and at least the threshold amount of chloride ions at the anode. The cathode must have good access to oxygen. The voltages that develop cause corrosion to advance so quickly that cracking, delamination, and spalling occur in the cover concrete. The deterioration, of course, occurs because the solid volume of the corrosion products is many times the solid volume of the original metal, in the case of iron. This increase in volume is largely due to the production of solids from reaction of the metals with gases or liquids or dissolved solids.

When steel corrodes, it produces many types of corrosion products (collectively called rust), depending primarily on oxygen, chloride, and water availability. Most of the products are amorphous (no crystalline structure determinable by X-ray diffractometry) ferrous (Fe^{++}) and ferric (Fe^{+++}) oxides, hydroxides, chlorides and hydrates, and complexes of these.

The only usual crystalline substance detected is magnetite (Fe_3O_4), but exposure to air and slow drying produces more crystalline oxides. Occasionally, someone new to X-ray analysis interprets the pattern as simply magnetite, ignoring the characteristic "amorphous hump" in the pattern that indicates that the rust is almost entirely amorphous. Crystalline corrosion products such as magnetite occupy two to three times the space that the original iron (or steel) did, while the amorphous products are generally more voluminous and variable, depending on specific conditions.[3]

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[3] William G. Hime, personal communication with William Perenchio.

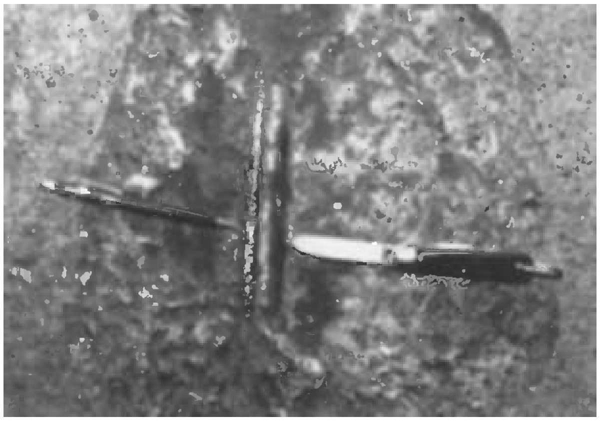

Fig. 3—Concrete spall over a reinforcing bar in a parking garage floor.

Some examples of damage due to chloride-induced corrosion are shown in Figs. 3 and 4. Figure 3 shows a spall over the top reinforcing mat in a parking structure. Such spalls commonly start to occur after 5–15 years of service, depending on concrete cover and quality. Figure 4 shows cracking, leaching, and spalling on the underside of a floor slab in a 27-year-old underground parking structure that had been exposed to deicing salt. When it was built, the technology of corrosion of metals embedded in concrete was in its infancy; therefore, no effective anti-corrosion measures had been taken.

The very large spall near the center of the photo was caused by a 4-in. (100-mm) zinc alloy electrical conduit, which corroded galvanically due to its proximity to the steel. (Galvanic corrosion is discussed in the chapter "Embedded Metals and Materials Other Than Reinforcing Steel" in this volume.) This may not appear to follow the description that attended Fig. 2, and indeed it does not. However, the cross section of this slab did follow that description very well when the structure was much younger. The top surface spalls were "repaired" with open-graded asphalt, which allowed the entry of large quantities of water and salt into the top of the slab, which then found its way to the bottom through cracks. After the electrical conduit corroded away, local anodes and cathodes became established within the bottom mat of steel. These microcells developed rapidly because the distance that ions must travel through the concrete to complete the cell is small; therefore, the total current resistance is small.

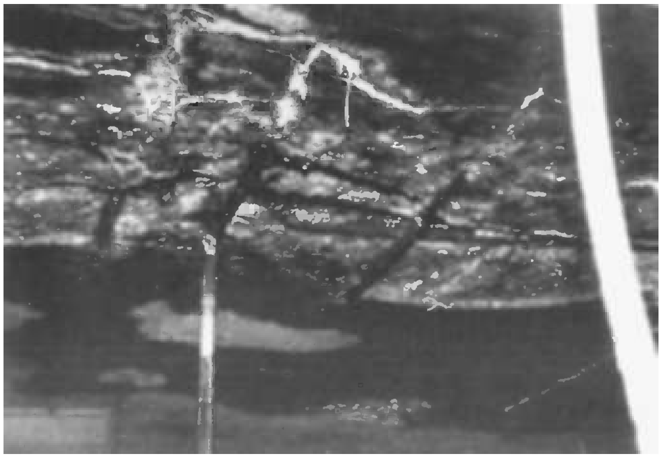

Fig. 4—Severe corrosion of reinforcing steel and an embedded electrical conduit on the underside of a parking garage floor.

The Peculiar Effect of Chloride Ion

Thus far, we have discussed only one ion as an instigator of corrosion, the chloride ion. Corrosion specialists in general agree that any ion of the halogen family would also be destructive. However, chloride ions are the only ones of this group that are normally found in large quantities in concrete.

The peculiar action of the chloride ion is not entirely understood. Some believe that, when the chloride ion concentration becomes large enough, ferrous chloride, or a ferrous chloride complex, is formed on the steel surface, replacing the ferric oxide[4] film that was stabilized by the high pH of the cement paste. Being more mobile (soluble) than the oxide, the chloride salt or complex moves away from the steel, exposing fresh iron to the now-corrosive local environment, and instigating the electrochemical cell. Certainly, the presence of the chloride, and the water that carried it in, increases the current-carrying capacity of the concrete, normally a very poor conductor.

Until the late 1970s, the most commonly used procedure for testing concrete for chloride ion permeability was described in AASHTO T259, "Resistance of Concrete to Chloride Ion Penetration." In this method, concrete prisms are continuously ponded with 3 % sodium chloride solution for 90 days. At the end of this time, the chloride content at several depths is determined. The obvious disadvantage to this test is the time required. ASTM Test Method for Determining the Penetration of Chloride Ion into Concrete by Ponding (C 1543) was approved in 2003 and is an updated version of AAHTO T259.

In 1977, the Federal Highway Administration entered into a contract with Construction Technology Laboratories to develop both a rapid field and laboratory test procedure for determining chloride permeability of concrete. The results were to correlate well with the 90-day ponding test. The rapid test method is now described by AASHTO as the rapid chloride permeability test [6]. A version of this standard was also published in the *1992 Annual Book of ASTM Standards* as ASTM Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration (C 1202). It involves forcing chloride ions to move through a small sample of concrete by applying direct current voltage.

Subsequent work has shown that the rapid method has an excellent correlation with chloride penetration as tested by AASHTO T259, for a wide range of concrete quality and types. However, some concretes that have altered electrical properties can give false readings. In the past version of this chapter it was believed that concrete containing silica fume, for example, is rated at lower permeability than it should be [7–9], presumably because the silica fume is such an active pozzolan. This ties up the normally free sodium, potassium, and calcium ions within insoluble calcium silicate hydrate structures, reducing the capacity of the concrete for conduction of electricity. However, a more detailed look at the test procedure shows that sample

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[4] This stabilized layer causes "passivation" of the steel, but only in an uncarbonated portland-cement paste. According to some investigators, this passivation is due to the formation of a very thin gamma Fe_2O_3 film that prevents further corrosion.

heating occurs, increasing current passed, so that higher permeability concretes look worse than they really are [10]. Thus, the test tends to overestimate the permeability of ordinary concretes due to the heating effect. Very low permeability concretes such as those containing silica fume or other pozzolans at low w/cm, do not heat up over the testing period so their currents don't increase over time. Work is currently under way to develop a modified version of ASTM C 1202 to address the heating and several other issues. Because of such situations, it is best to interpret the test results carefully. The test is an indirect method for determining chloride permeability, done under artificial conditions of high saturation and applied voltage. Its speed, however, makes the method attractive to those who need such information.

A new bulk diffusion test, ASTM C 1556, was issued in 2003. This method allows for a more accurate determination of the diffusion coefficient for chloride into concrete, but requires a minimum of three months to get results. Another new method on water absorption, ASTM Test Method for Measurement of Rate of Absorption of Water by Hydraulic Cement Concretes (C 1585), is now available to add additional ability to model the ingress of chloride.

Precautionary Steps Against Corrosion

Concrete water-cement ratio is especially important in determining the rate of ingress of water, and therefore dissolved salts, into concrete. This ratio above all else in plain concrete is the most important factor [11]. Some recently introduced admixtures and additives are very effective in reducing further the permeability of concrete. These will be discussed in more detail in a later section.

The effect of aggregates on the permeability of concrete is generally to increase it. Because the aggregates occupy approximately three-quarters of the volume of concrete, and their permeabilities can be as much as 1000 times greater than that of a high quality portland cement paste [12], they have a profound effect on the protection concrete offers to reinforcing steel against corrosion. Work [11] has shown the extremely important effects of cover and low water/cement ratios in resisting the ingress of the chloride ion into concrete, as shown in Fig. 5. The reason that 1 in. (25 mm) of cover was relatively ineffective in resisting chloride intrusion is considered to be due to the fact that 3/4-in. (19-mm) maximum size aggregate was used. Because of its intrinsically higher permeability and the high permeability of the paste adjacent to some aggregates, one large aggregate particle can easily "short-circuit" the distance between the concrete surface and the steel. This explains why two inches (50 mm) of cover is so much more effective than one, and three is, comparatively, little better than two.

Other methods of decreasing the permeability of the concrete cover are available. A synthetic latex admixture was first used in a concrete overlay on a bridge deck in Michigan in the early 1960s. Since that time, because the latex proved to be so effective in resisting moisture and chloride penetration, a great deal of work has been done in the laboratory [7] and field to develop the technology of the physical properties of latex-modified concrete.

Latex reduces the permeability of concrete in two ways. The physical presence of a lattice of latex (or rubber) within the concrete presents a more torturous route to water attempting to penetrate the concrete. And the reduction in mix water, made possible by the solids portion of the latex that imparts workability of its own to the concrete, results in lower permeability due to a decreased water/cement ratio paste.

Another method of greatly reducing the amount of water required to make concrete workable is the addition of a high-range water reducer (HRWR) (ASTM Specification for Chemical Admixtures for Concrete C 494, Types F and G). These materials, first identified in the 1930s by Tucker [13], became available in the United States in the mid-1970s, having been developed for use in concrete almost simultaneously in Germany and Japan. They are extremely effective in reducing water requirements. For the first time, commercially prepared concrete could be supplied with water/cement ratios as low as 0.30 by weight, with good workability. All of the physical

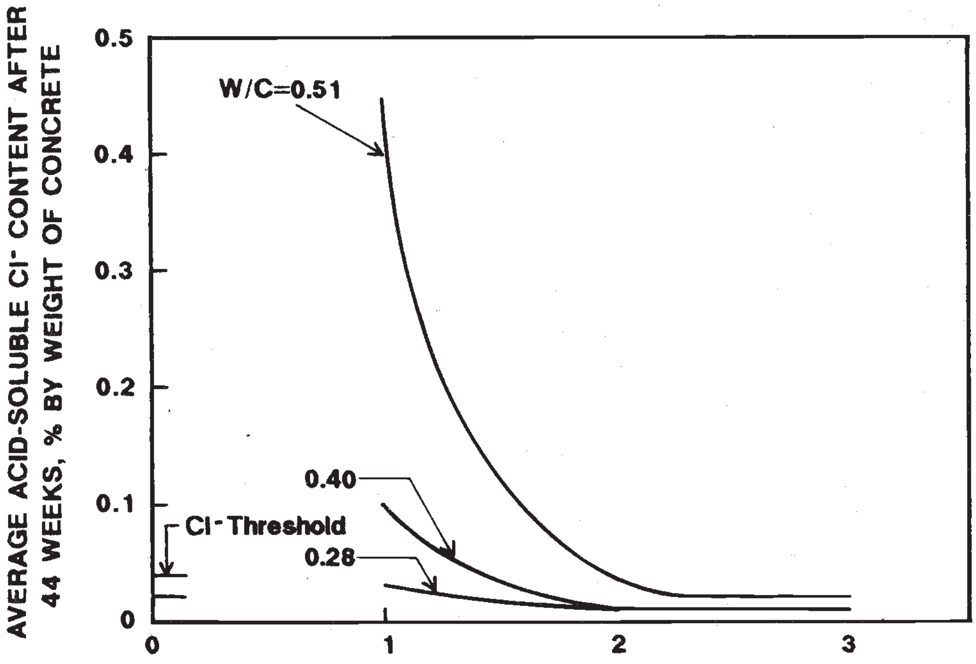

Fig. 5—Chloride content profiles at 44 weeks for different w/c ratio concretes.

properties of concrete with equal proportions, with or without the presence of a HRWR, are essentially identical. Recent developments in HRWRs include the introduction of polycarboxylates, which are more effective at lower dosages [14].

Soon after the HRWRs were introduced, another type of material, solid rather than liquid, appeared. This was silica fume, an extremely fine form of silica ("finer than tobacco smoke") and, therefore, extremely active as a pozzolan, a byproduct of the manufacture of silicon metal and ferro-silicon alloys. Because of its fineness, 50 to 100 times finer than portland cement, used as an additive by itself it would cause such a great increase in concrete water requirement that the material would be of very low quality. However, in combination with a HRWR, concrete can be produced with permeabilities as low [7,8] as that of latex-modified concrete, at a fraction of the cost.

The use of other supplementary cementing materials (SCM) such as fly ash and ground granulated blast-furnace slag (GGBFS) reduce the permeability of concrete [15,16]. Incorporation of such materials in cement paste reduces the pore sizes, resulting in decreased permeability.

Another additive available as an anti-corrosion agent is calcium nitrite. This material acts as an anodic or passivating inhibitor, to increase the chloride threshold level for corrosion initiation [17–19]. Later work showed that it can reduce corrosion rates after initiation [20,21].

Several papers [22–24] suggest using a combination of this calcium nitrite and silica fume. The philosophy was that the silica fume would slow the entry of water and the chloride ion, and the calcium nitrite would slow the corrosion rate after the chloride concentration reached the threshold level at the steel.

Several organic based inhibitors are now commercialized [25,26]. Some of these have dampproofing capabilities that help to reduce chloride ingress. These materials are predominantly amine and amine and fatty acid derivatives.

A different approach to the prevention of penetration of water and dissolved salts into concrete is to apply a surface sealer after the concrete has hardened. A 1981 study [27] investigated 22 materials used for this purpose. A screening test, amounting to nothing more than immersion of 4-in. (100-mm) concrete cubes in salt water for three weeks, was used to determine which of these appeared to have merit. Five were outstanding. These were tested further by subjecting them to wetting and drying, and either freezing and thawing cycles or extremely severe exposure to ultraviolet light. This testing eliminated two materials, leaving a silane, an epoxy, and a methyl methacrylate as the best performers. The silane had the added advantages that it could not be seen, it penetrated about 1/8 in. (3 mm) into the concrete, and it did not restrict the outward movement of water vapor. Silanes can be effective but need to be reapplied on a routine basis (every 3 to 5 years) as found in SHRP [28]. However, one study indicated longer performance [29]. The use of such effective sealers as those identified by this study is sometimes more effective against corrosion than producing a very impermeable concrete [11]. However, as indicated in the SHRP study reapplication is necessary under field conditions.

Another approach to preventing corrosion is to coat the steel. Zinc has been used for this purpose, and is somewhat effective; however, it must be used on all the steel within the structure rather than only that which is expected to become anodic [11,30].

The usual product that results from corrosion of zinc is zinc oxide, which occupies only 50 % more space than the original metal. However, if large amounts of chloride are present in concrete containing the zinc, zinc hydroxy chloride can also form. This occupies about 3.5 times the volume of the metal and, although this is less than the iron products, it is still capable of producing large expansive forces.

Field evaluation of bridge decks reinforced with galvanized reinforcing steel showed good performance. Evaluated bridges were subjected regularly to deicer salts for more than 25 years and chloride concentration measured at the level of steel were scientifically higher than the corrosion threshold for black steel. Remaining coating thicknesses of steel samples taken from the bridges were within the range of recommended coating thickness for new reinforcement [31].

A more widely used material to use for surface protection of the steel is fusion-bonded epoxy [11,17]. Early work showed that, although the epoxy typically has small holes or holidays in the finished coating, and the coating can be damaged by rough handling or bending of the bars, it can do a very good job of protecting the steel, particularly if all the steel in the structure is coated and the number of holes, or holidays, are minimized. Although high corrosion densities may occur at breaks in the coating, the rate of metal loss is moderate. Based upon field experience in Florida and elsewhere [32–34], it is now realized that defects should be minimized and that constant wet conditions can be more severe than laboratory exposures with drying cycles.

The protection systems mentioned above can be combined to provide improved performance. They are especially more effective at lower w/cm values [1].

Prestressed Concrete

Generally, the same factors are involved in the corrosion of prestressing steel as in mild reinforcing steel. However, the same percentage loss of metal can cause catastrophic failures in prestressed structures, while causing nothing more than spalls and delaminations in normally reinforced structures. Also, prestressing steel is subject to stress corrosion cracking, a phenomenon that does not occur in mild steel. This is sometimes brought about, or at least is associated with, the presence of certain ions. Two of these that can be present in concrete are the nitrate and bisulfide ions (NO_3^- and HS^-) [35]. Chloride ions, in this case, often appear to be innocuous [35,36], except at high temperatures.

Cases have been reported [36] where chloride levels have been high in prestressed concrete members, but no corrosion is evident. However, it is generally accepted that corrosion of prestressing steel is instigated by chloride ions.

Research [11,37] has shed some light on this subject. In one study [11], the corrosion threshold of chloride for prestressing steel was found to be approximately six times that for mild steel, or 1.2 % by weight of the cement. Part or all of the reason for the greater resistance to corrosion may be that stearate compounds are used during the manufacturing process. Note that this was the finding of only one study and that chloride induced corrosion in prestressing strand can occur at low chloride levels in the bulk concrete due to cracking or at the anchorage zones for post-tensioned structures [38]. Note that prestressing steel is more susceptible to embrittlement and failure if the concrete or mortar pH drops [1].

A study [37] of pretensioned and post-tensioned prestressing systems evaluated grouts, ducts, and anchorage systems. Highlights of the results were that polyethylene ducts were more effective in shielding the strand from corrosion than steel ducts were, but the plastic tended to wear through at bends during stressing of the steel, exposing the steel to the surrounding concrete. Traditional bare and galvanized steel ducts deteriorated badly under chloride exposure, allowing chloride to enter the grout. Joints in duct material were a serious problem, but it was overcome by the use of shrink-fit tubing. Providing an excellent grout was largely unsuccessful, because of the minimal grout cover possible within the duct at the inside surfaces of bends.

The anchorages proved to be vulnerable behind the traditional dry-pack mortar. These should be coated or covered with epoxy. The epoxy coating on the strand was never breached, even within the wedge grips.

Assessing the Severity of Corrosion in Existing Structures

The simplest technique for assessing the present condition of a structure deteriorated by corrosion is a visual survey [39]. Visual survey data can be used to produce maps that show locations of cracks, spalls, and other features of deterioration. This is usually followed by a delamination survey, done by striking the concrete surface with a metallic object and listening for hollow sounds. The visual and delamination survey results can be used to select areas for the in-depth studies described later, usually those areas that are typical of the worst, moderate, and best conditions.

Half-cell potential surveys are very useful in determining which areas are actively corroding [40]. The action of an electrochemical cell produces differences in electrical potential of the steel. By measuring these potentials with a half-cell and a voltmeter on a grid pattern, diagrams that resemble contour maps can be constructed, with lines connecting points of equal potential, or voltage. Closely spaced lines are typically observed near areas of high corrosion activity. One study [11] has shown that active corrosion is indicated wherever the half-cell potential is more negative than − 0.23 V versus CSE. Figure 6 shows the relationship of half-cell potential to actual corrosion current that was determined in this study. The test method is described in ASTM Test Method for Half-Cell Potentials of Uncoated Reinforcing Steel in Concrete (C 876). However, the oxygen content, usually related to the water saturation, and junction potentials can cause corrosion potentials to vary significantly for essentially the same corrosion rate [41]. Indeed, no corrosion has been found at much lower half-cells than − 0.23 V versus CSE [18]. Thus, half-cell potentials are best used in contour maps and with subsequent autopsy or corrosion rate measurements.

A magnetic rebar locator may be used to assess the amount of concrete cover over reinforcing steel. This instrument is useful in determining the potential for future corrosion in various parts of a structure. It can also be used to locate a near-surface bar for grounding the half-cell voltmeter.

Ground penetrating radar, which is based on electromagnetic wave principle can be used to measure concrete cover more effectively. Cover profiles can be obtained by scanning the radar antenna along the surface of reinforced structures [42].

Concrete Cores

For detailed petrographic examination, chloride ion determination, or compressive strength tests, concrete cores removed from the structure can be useful. The petrographic, or microscopical, examination, ASTM Practice for Petrographic Examination of Hardened Concrete (C 856), can yield information on the quality of the cement paste, degree of curing, stability of the aggregates, air content, and damage caused by chemical attack, freezing and thawing, corrosion, etc.

Chloride Samples

When concrete cores are not removed, powder samples can be taken for chloride analyses. This is usually done with a rotary hammer. Samples are taken at various depths, down to and slightly beyond the depth of the steel. Analysis can be done on an acid-soluble or water-soluble basis. Historically, the acid-soluble technique (AASHTO T260 or ASTM C 1152) has been used, with full awareness of the fact that all of this chloride is not available to support corrosion, due to chemical combination with the cement or because it is tightly held within aggregate particles. The results of water-soluble tests are greatly affected by the degree of grinding of the sample and by the length and temperature of leaching [43]. The

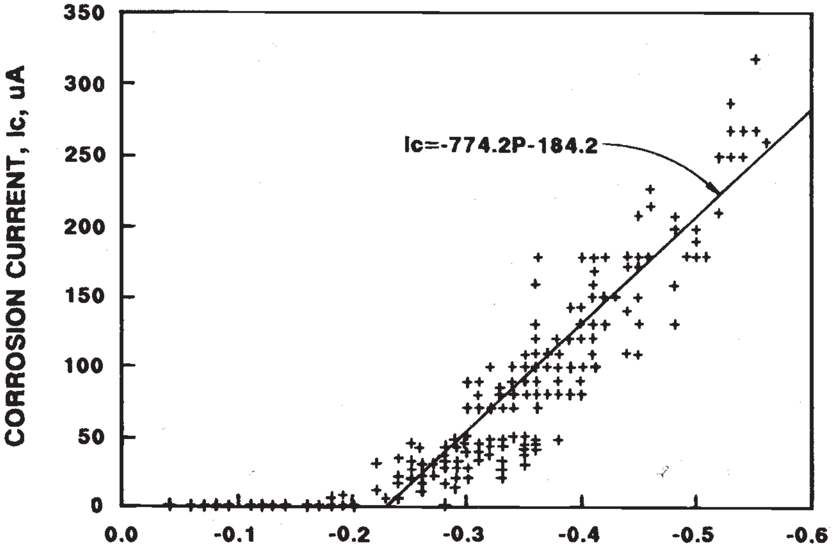

Fig. 6—Relationship between corrosion current and half-cell potential.

currently accepted test for water-soluble chloride is described in ASTM Water-Soluble Chloride in Mortar and Concrete (C 1218).

Repairs to Deteriorated Structures

Bridges and parking structures in areas of deicer use, along with marine structures of all sorts, are constantly exposed to corrosive conditions and, therefore, are the usual structures needing repair and rehabilitation. Typically, these repairs consist of removal of spalled and delaminated concrete (sufficiently below the top reinforcing steel so that it will be encapsulated within the repair concrete), replacement of the concrete, and some means for restricting the entry of water and salt solutions. In most instances, long-term durability of such repairs has not been evaluated. In extreme cases, the structure must be demolished. In others, cathodic protection may be a viable alternate.

After the deteriorated concrete has been removed and replaced, some form of waterproofing must be applied. If it is not, new delaminations will soon form in the original concrete surrounding the repairs. This is called the “ring anode effect.” Starving the concrete for water is the most effective way to reduce the corrosion rate, outside of cathodic protection. Overlays of specialty concretes, such as latex-modified, low-water cement ratio, or silica fume-containing, can be used, but they are expensive. Difficulties in achieving bond with the substrate are sometimes encountered. Thin proprietary membranes of polyurethane alone, or combined with an epoxy-sand wearing course, are also used with good success. However, they are expensive and must be maintained.

A simpler and less expensive method is the application of a penetrating sealer such as a silane or a siloxane. Silanes penetrate the surface more readily [44] and, therefore, are expected to last longer. These materials polymerize within the concrete and also combine with the siliceous portions of the cement and aggregates, becoming chemically bound adjuncts. Because they are inside the concrete, ultraviolet light, a major disintegrator of organic molecules, cannot reach them. Silanes are now available as water emulsions or as 100 % silane, rather than the more traditional alcohol solutions, doing away with volatile organic compound concerns.

Patch areas often fail due to shrinkage of the patching materials as they are restrained by concrete around the patch [45]. Reducing the strength of the concrete in the patch or reducing shrinkage can help to improve performance.

Cathodic Protection

Cathodic protection is truly the only known method of completely preventing corrosion of reinforcing steel embedded in chloride-contaminated concrete. A metal is cathodically protected when sufficient current is applied to polarize the cathodes to the open circuit potential of the anodes [46].

This can be accomplished easily and safely, in constantly wet exposures, with a sacrificial anode system. The technique has been used for many years on concrete piles by the state of Florida, and as part of the overall corrosion protection systems for offshore drilling platforms. An anode of zinc or magnesium is connected to the reinforcing cage through a lead wire, then immersed in the sea. Corrosion of the anode supplies electrons to the steel, preventing its corrosion. A similar system was used in a research project a decade ago [47] to protect a deteriorating interstate highway bridge deck. It was considered a success by the researchers, but the technique has not gained popular acceptance among transportation agencies.

Systems that employ impressed current, while not extremely popular, have met with somewhat better success. The first instance of the use of such a system was in 1974, on a bridge deck in California [48]. Since that time, hundreds of bridge decks have been so treated, but serious difficulties remain [49]. Controlling current densities and maintaining uniform potentials continue to be basic problems, although several viable anode systems have been developed. Impressed current systems have not been put to use commercially on prestressed structures due to the possibility of hydrogen generation [50], which can give rise to hydrogen embrittlement. A general early review of cathodic protection was presented by two British authors [51]. Additional information can be found in a paper by Broomfield and a SHRP Review [52,53], as well as the proceedings from several NACE annual meeting symposia on cathodic protection [54].

New Steels

Stainless steel bars and cladding are now available and ASTM is working on a standard. Results are significantly improved over standard reinforcing bars in testing, but dependent upon which alloys are used [55,56]. Though corrosion is reduced, it is not necessarily prevented [57].

Steel processed to have untransformed nano-sheets of austenite sandwiching dislocated martensite laths is available. There are no carbon containing phases present, so cathodic reaction rates are claimed to be low on this steel. The version with the most promise contains over 8 % chromium [57].

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17

Embedded Metals and Materials Other Than Reinforcing Steel

Bernard Erlin[1]

Preface

THE INITIAL WORK ON THIS CHAPTER WAS DONE by Hubert Woods, a concrete consultant who is now deceased. The chapter was published 27 years ago in *ASTM STP 169A* and, with some modifications, in the subsequent two volumes, *169B* and *169C*. A current search of the literature for the effects of concrete on embedded nonferrous metals reveals little new information than reported previously in this chapter. What has been located is now included.

The basic principals of chemistry and physics do not change. The response of materials to their environment is the same now as it was when this chapter was originally written. And, in spite of lessons learned or, because of a peculiar environmental exposure, a material may perform in an unusual way. An understanding and caution should always be exercised in material usages. Perhaps that understanding and caution has guided the use of embedded materials other than reinforcing steel so that there is little about their adverse performance reported in the literature. If that is the case, then a value of the principals that extend the service life of those embedded materials has become a code of use.

Introduction

Metals other than conventional reinforcing steel are sometimes used in conjunction with concrete. Emphasis will be given here to the possible degradable aspects relative to their use and conditions that may render them unserviceable.

The materials to be described include metals and other inorganic and organic substances. Among the metals are aluminum, lead, copper and copper alloys, zinc, special alloys of iron, monel metal, stellite, silver, and tin. Among the inorganic materials are glass, asbestos, and recycled concrete; and among the organic materials are a variety of plastics and wood and similar cellulose materials. Fiber-reinforced concrete is gaining in use, so information about some fibers has been included.

A general condition necessary for the chemical degradation of any material in concrete is exposure to moisture, and a short discussion on that aspect precedes the discussions of material behavior.

General Condition

Moisture is usually necessary for the chemical degradation of any material. It can be in the form of water vapor, liquid water, or solutions where water is the solvent. Concrete is never completely dry because it contains air voids and capillary voids that hold moisture in vapor form, sufficient in many cases, to maintain a relative humidity of about 80 %, which is the moisture equilibrium of the paste. Furthermore, although a concrete may be relatively dry, carbonation of the portland-cement paste will release water when carbonation occurs, as shown by the following:

$$CaSiO_2 \cdot xH_2O + CO_3 \rightarrow CaCO_3 + SiO_2 + xH_2O$$

Concrete is wet when mixed and completely saturated with water for some time after hardening. There usually is more moisture than needed to hydrate the cementitious materials so there remains water to be lost to the environment. The distance through which free water must move to an external surface where it can evaporate dictates the residual moisture content of the concrete at any given time. The rate of drying also depends on ambient humidity of air in contact with the concrete, but even at low ambient humidities, a long drying time may be needed to lower the concrete humidity to a point where corrosion of embedded materials is slowed dramatically or stopped. Thus, concrete that will eventually be dry may be at an internal relative humidity long enough to cause corrosion of susceptible materials, and, of course, concrete exposed continuously or frequently to a damp environment may remain moist enough to support corrosion. The free moisture in concrete can stay in vapor form or as pore solutions that facilitate transport of soluble chemical substances, such as oxygen, calcium hydroxide, alkalies, sulfates, and chlorides, toward embedded materials and of any soluble corrosion products away from the materials. It also increases the electrical conductivity of concrete, thus aiding any tendency for electrochemical corrosion of metals.

The relatively long drying time of concrete is not usually appreciated. The rates of drying of 15 × 90 × 90 cm (6 × 36 × 36 in.) slabs of normal-weight concrete are shown in Table 1 [1]. The table shows that at an environmental relative humidity

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[1] Petrographer, The Erlin Company, Latrobe, PA 15650.

TABLE 1—Rates of Drying 15 × 90 × 90 cm (6 × 36 × 36-in.) Slabs of Normal-Weight Concrete[a]

Environmental Relative Humidity (RH)	Drying Time to Reach Various Relative Humidities in the Concrete Slab at Middepth, days		
	90 % RH	75 % RH	50 % RH
10	18	80	620
35	30	110	840
50	36	240	…
75	36	…	…

[a] From [1].

of 50 %, 36 days are needed to lower the mid-depth relative humidity to 90 %, and 240 days are needed to reach 75 %. Additional tests show that when the mid-depth relative humidity reaches 90 %, the relative humidity at a point only 1.9 cm (3/4 in.) from an exposed surface is 87 %. The moisture condition of concrete required to support active electrochemical corrosion of aluminum and other susceptible metals is not known with any accuracy; but it seems almost certain that such corrosion could proceed, other conditions being favorable, at 90 % relative humidity and probably also at 60 % relative humidity, though at a much lower rate.

Concrete made with lightweight aggregates dries more slowly than concrete made with normal-weight aggregates. Thicker sections, such as beams and columns, will dry more slowly than thin sections, such as walls or elevated floor slabs. Indeed, today lightweight aggregate is used for internal curing.

Aluminum

Aluminum has been given widespread attention because it has been the cause of problems in the past. These prompted laboratory studies designed to describe conditions necessary for its corrosion and methods for circumventing its distress.

In fresh concrete, aluminum reacts principally with the alkali hydroxides derived from hydration of portland cement. One reaction product is hydrogen gas, and for this reason aluminum powder sometimes is used to form lightweight cellular concrete (an invention usually attributed to Thomas A. Edison, who developed it for insulation purposes). In smaller amounts, the gas provides slight expansion of grout and has been used, for example, for bedding machinery base plates. Aluminum in rod, sheet, or pipe form reacts much less vigorously than the powdered metal because of the lesser surface area exposed to the alkalies, and the reactions will continue until the metal is totally reacted.

Tests carried out by Jones and Tarleton [2] indicate that the corrosion of embedded aluminum can crack concrete under unfavorable circumstances. It has also been shown that the situation can be worse if concrete contains purposefully introduced calcium chloride (chloride ions are a strong electrolyte), and much worse if it also contains reinforcing steel that is in direct contact with the embedded aluminum.

Wright [3] described a case of corrosion of sufficient severity to cause collapse of aluminum conduit in reinforced concrete made using calcium chloride. Extensive concrete spalling over aluminum conduit in the Washington D.C. Stadium [4] and corrosion of aluminum balusters embedded in concrete have been reported [5]. Several dozen additional cases of concrete cracking due to corrosion of embedded aluminum conduit, posts, and window frames in contact with concrete have occurred. In every case, chloride was present. An example of concrete spalled over corroded aluminum conduit is shown in Fig. 1.

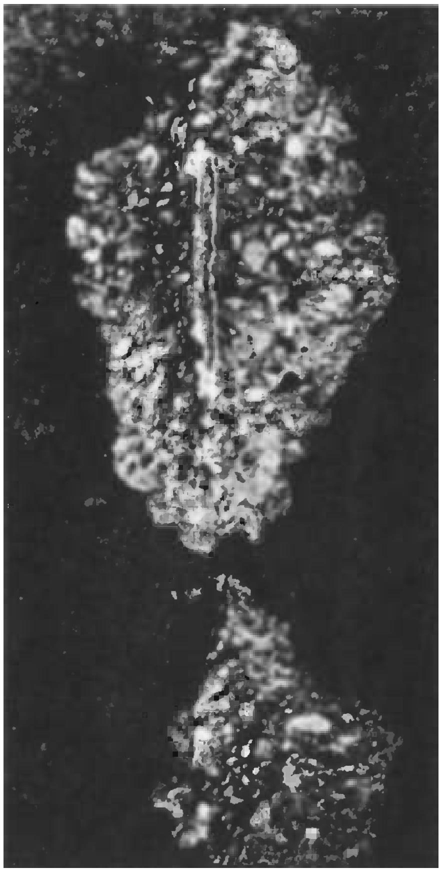

Fig. 1—Concrete spalled by corroding aluminum conduit (from Ref 5).

The laboratory investigations by Monfore and Ost [6] show that calcium chloride concentration, alkali content of portland cement, electrical coupling of steel and aluminum, and the ratio of steel area to aluminum area are all interrelated in the corrosion of the aluminum and the subsequent cracking of concrete. In their studies, 15-cm (6-in.) concrete cubes were prepared using cements of high and low alkali contents in which pieces of nominal 1.25-cm (½-in.) aluminum conduit was embedded 1.25 cm (½ in.) from one face. C-shaped sheets of mild steel were also embedded in the cubes. The steel and aluminum were externally connected in some tests and not connected in others. Various amounts of calcium chloride additions were used. The cubes were removed from their molds at 24 h, coated with a curing compound, and then stored at 23°C (73°F) and 50 % relative humidity for 28 days and observed regularly for cracks. After 28 days, the aluminum pieces were removed, cleaned, and weighed. The principal results of the studies are given in Table 2. Several important findings were noted:

- All cubes that cracked contained calcium chloride.
- Cubes containing 1 % flake calcium chloride ($CaCl_2 \cdot 2H_2O$) by weight of cement did not crack, but metal losses in these cubes were as high as, or higher than, those in

TABLE 2—Corrosion of 6063 Aluminum Conduit Embedded in 15-cm (6-in.) Concrete Cubes Stored at 50 % Relative Humidity for 28 Days[a]

Cement	Cement Alkalies as Na_2O, %	$CaCl_2 \cdot 2H_2O$, % by weight of cement	Ratio of Steel Area to Aluminum Area	Electrodes	Days to Cracking	Loss in Surface Thickness, mils[b]
C	0.24	0	28	uncoupled	no crack	0.16
		2	14	coupled	5	0.92
		2	28	coupled	4	1.2
		4	7	coupled	3	1.6
		4	14	coupled	3	2.3
		4	28	coupled	3	2.4
		0	28	uncoupled	no crack	0.09
		2	14	uncoupled	no crack	0.07
		2	28	uncoupled	no crack	0.10
		4	7	uncoupled	no crack	0.07
		4	14	uncoupled	no crack	0.07
		4	28	uncoupled	no crack	0.04
		0	0	...	no crack	0.09
		4	0	...	no crack	0.04
D	0.89	0	28	coupled	no crack	0.12
		1	3.5	coupled	no crack	0.54
		1	7	coupled	no crack	0.77
		1	14	coupled	no crack	1.0
		1	28	coupled	no crack	0.85
		2	3.5	coupled	3	1.0
		2	7	coupled	3	1.4
		2	14	coupled	4	1.4
		2	28	coupled	4	1.6
		4	3.5	coupled	2	1.5
		4	7	coupled	2	1.7
		4	14	coupled	2	2.2
		4	28	coupled	7	3.3
		0	28	uncoupled	no crack	0.33
		2	14	uncoupled	no crack	0.06
		2	28	uncoupled	no crack	0.06
		4	7	uncoupled	no crack	0.07
		4	14	uncoupled	no crack	0.09
		4	28	uncoupled	no crack	0.08
		0	0	...	no crack	0.17
		4	0	...	no crack	0.05

NOTE—Conversion factor: 1 mil = 2.54×10^5 m.

[a] From [5].

[b] Calculated from weight losses.

other cubes that cracked. (Exposure to a damper environment might have caused cracking to occur.)

- With no calcium chloride and no electrical coupling, the corrosion was greatest with the high alkali cement.
- With a 2 or 4 % calcium chloride addition, and with metals electrically coupled, corrosion was slightly greater when the higher alkali cement was used.
- With calcium chloride and metals electrically coupled, corrosion generally increased with increasing ratio of steel area to aluminum area and invariably increased as the amount of calcium chloride increased.
- Cubes that cracked did so within seven days.

In the case of coupled metals, considerable galvanic currents occurred in the circuit connecting the aluminum and steel. Some measured currents are shown in Fig. 2 as a function of time and calcium chloride content. Current flow progressively increased with increasing calcium chloride contents up to the end of the 28-day tests. When the total electrical flow during 28 days was plotted against the amount of corrosion measured as loss in thickness, straight curves of varying shapes were obtained with the slopes depending on the amount of calcium chloride. The steeper slopes occurred for the higher amounts of calcium chloride.

Tests somewhat similar to those reported by Monfore and Ost were done on 31-cm (12-in.) concrete cubes by Wright and Jenks [7]. With electrically coupled steel and aluminum (area ratio 10:1), cracks did not occur when calcium chloride additions were not used. However, cracks did occur at various ages; after 61 days with 1.1 % flake calcium chloride by weight of cement to eight days with 5.7 % flake calcium chloride by weight of cement.

McGeary [8] also found that: (a) for aluminum conduit electrically coupled to steel, cracks did not occur in the encasing concrete when chlorides were not present; (b) the cause of

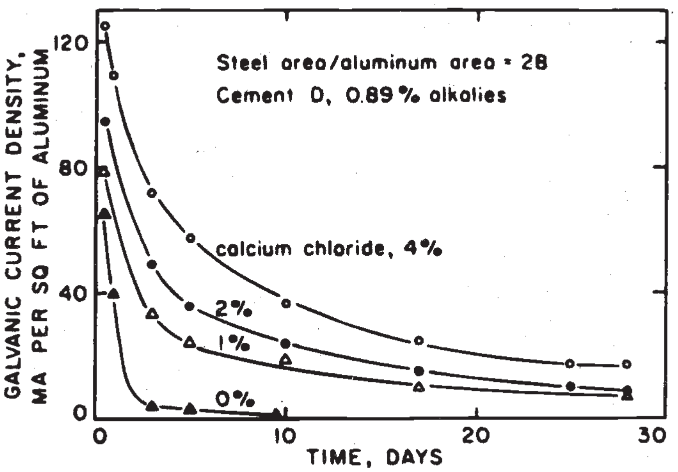

Fig. 2—Effect of calcium chloride on galvanic current (from Ref 5).

corrosion of the aluminum was due unquestionably to galvanic cells activated when chlorides were present in the concrete; (c) corrosion of the aluminum was of the cubic type, which is also referred to as intergranular corrosion; and (d) there was an enrichment of chloride at the aluminum conduit surface.

Tests [6] were done to determine the effectiveness of several different coatings on aluminum embedded in 1.25-cm (½-in.) concrete cubes made with cement having an alkali content of 0.89 % and containing 4 % calcium chloride by weight of cement. The results are shown in Table 3. The data demonstrate that a silicone coating was ineffective; that a lacquer (Lacquer B) prevented cracking within 28 days but permitted some corrosion; and that Lacquer C and Bituminous Coatings A and D were each effective in preventing both corrosion and cracking.

Other tests [8] indicate that the following coatings provide resistance to galvanic corrosion: certain bitumens, epoxies, fluidized-bed plastics, certain metallic pigmented coatings, and alkyd and phenolic materials.

McGeary [8] found the following to be effective repair procedures for concrete cracked because of corroded aluminum: (a) removal of all loose concrete; (b) removal of all corrosion products adjacent to the embedded aluminum surface; (c) patching using epoxy or epoxy polysulfide systems, or both; and (d) engagement of an experienced patching specialist to complete the repairs.

The results of these various investigations and of field experience show that reinforced concrete is likely to crack and spall from corrosion of embedded aluminum if calcium chloride was used, and coatings that insulate the aluminum from the concrete are commercially available and practical for use.

In view of chemical similarities between calcium chloride and sodium chloride, it seems evident that the latter would also facilitate corrosion of aluminum. Sodium chloride is the principal constituent of sea salt, and it therefore seems prudent not to use aluminum in concrete exposed in, or near, seawater. The role of chloride when galvanic cells (e.g., steel and aluminum in electrical contact) are present is that of an electrolyte, a necessary part of a galvanic cell. Other salts that hydrolyze and provide strong electrolytes should also be damaging.

Based upon laboratory studies and field experiences, it is prudent not to use calcium chloride in concrete in which aluminum will be used or where concrete will be exposed to chlorides.

Lead

Lead has a high resistance to certain chemical actions, but in contact with damp concrete it is attacked by the calcium hydroxide component of portland cement hydration and becomes converted to lead oxide or to a mixture of lead oxides. If the dampness persists, the attack will continue. A lead pipe, for example, may be destroyed in a few years. If the lead is electrically coupled to reinforcing steel in concrete, galvanic action may accelerate the attack [9a], in which case the rate of corrosion may be several millimeters per year.

Lead partially embedded in concrete, and thus partially exposed to air, is susceptible to corrosion because of the differential electrical potential that results. In the presence of water, the air-exposed portion becomes a cathode with respect to the embedded portion that becomes an anode. A galvanic cell forms, and corrosion and deterioration of the embedded lead result.

A protective coating or covering should be used when lead pipe or cable sheaths are to be embedded in concrete. Bituminous coatings have been used successfully. Synthetic plastic and other organic coatings or sleeves, which are themselves

TABLE 3—Effect of Protective Coatings on Corrosion of Aluminum Conduit Embedded for 28 Days in 15-cm (6-in.) Concrete Cubes Containing 4 % Calcium Chloride and Steel Electrically Coupled to the Aluminum[a]

Protective Coating Material	Thickness, mil	Days to Cracking	Loss in Surface Thickness, mil[b]
None	...	3	2.5
Silicone	...	2	2.1
Lacquer B	1	no crack	0.47
Lacquer C	2	no crack	nil
Bitumen A	5	no crack	nil
Bitumen D	15	no crack	nil

NOTE—Conversion factors: 1 mil = 2.54 × 10^5 m; 1 in. = 2.5 cm.

[a] From [5].

[b] Calculated from weight losses.

unaffected by damp concrete, are also appropriate. Lead in contact with concrete should be protected by suitable coatings or otherwise isolated from contact with the concrete [9b]. Also, lead and lead salts very strongly retard portland cement hydration.

There appears little, if any, likelihood of concrete itself being damaged by corrosion of lead because of the softness of the metal and its capability for absorbing stress.

Copper and Copper Alloys

Copper will not corrode in concrete unless soluble chlorides are present. Copper pipes are used successfully in concrete except under unusual circumstances where ammonia is present [10]. Very small amounts of ammonia, and possibly nitrates, can cause stress corrosion cracking. Brass wall ties have reportedly failed by stress corrosion, and manganese bronze bolts have sheared below their ultimate strength. However, because such phenomena can occur under circumstances unrelated to concrete, it is not clear what role, if any, the concrete plays. Very little systematic work has been reported on the behavior of copper and its alloys in contact with concrete, probably because these metals have given satisfactory service under such conditions.

Copper, brass, red brass, bronzes, aluminum-bronze, and copper-silicon alloys embedded in concrete have good resistance to corrosion [11,12].

When copper is connected or adjacent to steel reinforcement and an electrolyte such as chloride is present, corrosion of the steel due to galvanic action is likely to occur. Thus, under such circumstances, it is desirable to insulate the copper with suitable coatings.

Zinc

Zinc reacts chemically with alkaline materials, but normally in concrete the reaction is superficial and may be beneficial to bond zinc (galvanized) to steel. The primary chemical reaction with calcium hydroxide is

$$\mathrm{Zn} + \mathrm{Ca(OH)_2} \rightarrow \mathrm{CaZnO_2} + \mathrm{H_2}\uparrow \tag{1}$$

When zinc is used in concrete, it is generally as a galvanized coating for steel. The reaction is self-limiting, and products of reaction are not voluminous; consequently, damaging stresses are not created.

Although good concrete normally provides a nearly ideal environment for protecting embedded steel from corroding, embedded galvanized steel reinforcement has been used in concrete exposed in marine and other environments where chlorides are present (e.g., bridge decks and columns, and docks). Galvanized reinforcing steel has not always been successful in stopping corrosion of the steel, and thus there are conflicting reports about the benefit, if any, of the coating [13–15]. Stark [16] investigated embedded galvanized reinforcement in seawater-exposed concretes that were from 7–23 years old. The concrete cores examined were from mean tide and above high tide zones. Chloride concentrations to depths of 10.1 cm (4 in.) were always exceedingly high at about the level of the galvanized bars [from 1.9–6.4 kg/m^3 (3.2–10.7 lb/yd^3)]. Concrete cover over the galvanized bars was from 5.7–13.3 cm (2¼–5¼ in.). The amount of galvanized coating remaining was 92–100 %. A summary of the data is given in Table 4. Unpublished data by Hime and Erlin for galvanized bars threaded through brick hollow cores packed with mortar of 12-year-old masonry facades containing exceedingly high chloride contents revealed that the galvanize coating was completely corroded. Galvanized wall ties of brick masonry facade construction can undergo similar corrosion when chlorides are present.

TABLE 4—Galvanize Coating Remaining on Reinforcing Bars After Indicated Years of Concrete Exposure to Seawater

Years of Age	Galvanize Coating Remaining, %
7	98
8	100
10	95
10	96
10	99
12	92
23	98

Fig. 3—Corrosion and perforation of galvanized steel from under concrete slab (from Ref 11).

Galvanized corrugated steel sheets often are used as permanent bottom forms for concrete roof, bridge deck, or floor construction. Both satisfactory and unsatisfactory performances have been reported. Figure 3 shows one instance of multiple perforation and corrosion of such sheets under a roof slab [10]. Most of the corroded spots were dry to the touch, but some were moist and had acidic pH values of 2.7–4.8. Analysis of the corrosion protuberances indicated the presence of iron, zinc, and chloride. Further investigation of this and other similar cases showed that in each instance calcium chloride was used in the concrete so that chlorides were in direct contact with the galvanized sheets. This abetted the corrosion by chemical action on the zinc and by increased electrical conductivity of the concrete. The chemical reaction is probably

$$\mathrm{Zn} + \mathrm{CaCl_2} + 2\mathrm{H_2O} \rightarrow \mathrm{CaZnO_2} + 2\mathrm{HCl} + \mathrm{H_2}\uparrow \tag{2}$$

This chemical action produces hydrochloric acid, which explains the observed acidity of the corrosion product. It is probable that this reaction takes place only after local depletion of calcium hydroxide by the reaction expressed in Eq 1. Other reported zinc corrosion products are zinc oxide and zinc hydroxy-chloride.

Admixtures containing major amounts of chloride should not be used in concrete that will be exposed to moisture and contains, or is in contact with, galvanized steel. It is also advisable to keep chloride-containing solutions from permeating the concrete.

Zinc galvanize can corrode when in contact with relatively fresh concrete. The corrosion phenomenon results in pitting of the zinc surface due to reactions of the zinc and alkalies in the cement paste. Thus, zinc-coated steel forms, for example, in precast or cast-in-place concrete, may cause concrete surface disfiguration due to contact of the zinc. Contact of galvanize to steel in concrete in the presence of chloride and other electrolytes can cause galvanic corrosion.

Passivation of zinc by use of chromate dips has been reported to be effective in protecting galvanized products. The dips are solutions of sodium or potassium dichromate acidified with sulfuric acid. Chromate dips on galvanized bars or 400 ppm chromate in the mixing water also prevent hydrogen evolution in fresh concrete.

Concrete in which galvanized reinforcing steel is located close to galvanized forms has a tendency to stick to the forms. A chromate treatment, such as previously described, has been used as a method for avoiding that problem.

Use of corrosion inhibitors, such as calcium nitrite, improves the corrosion resistance of zinc in concrete [10a].

Other Metals

The following metals have been reported to have good resistance to corrosion in concrete: stainless steels, chrome-nickel steels, chromium-aluminum-silicon steels, cast silicon-iron, alloyed cast iron, nickel, chrome-nickel alloys, iron-chrome-nickel alloys, monel metal, stellite, silver, and tin [12]. The resistance of some of these metals to corrosion may be affected by the presence of "corrosion promoters" such as soluble chlorides. Monel metal and Type 316 stainless steel are well known for their resistance to the effects of sodium chloride and other constituents of seawater and should work well in concrete. The 300 series of stainless steel will corrode in the presence of chloride and when temperatures are above 140°F (60°C). Special circumstances might justify the use of these more costly metals.

Nickel and cadmium-coated steel will not corrode in chloride-free concrete if the coatings are continuous [17]. However, the corrosion resistance of these materials becomes questionable if chlorides are present in the concrete or in solutions that permeate the concrete [18].

Glass

Glass sometimes is embedded in mortar or concrete as artificial aggregate [19,20] used for decorative or aesthetic purposes, as reinforcing as a substitute for steel, as wall blocks or tiles, and as frameless windows or lights. Some glasses are reactive with alkalies in portland cement paste and form alkali-silica gel. The resulting expansion may cause severe damage to the glass or the concrete or both [21].

Whenever glass is to be used in concrete, it should always be tested to ensure that it would be chemically stable in the alkaline concrete environment and not cause deterioration due to alkali-silica reaction. For example, waste bottle glass used as aggregate in a decorative concrete facing of outside-exposed pre-cast concrete panels caused sufficient expansion to warp the panels. The warping was due to expansion of the facing, which was bonded to a non-expansive concrete backing. Prisms cut from the panels and tested using accelerated methods in the laboratory had expansions of 0.2 % after three months of testing. The glass aggregate removed from the concrete, tested using the procedures in the ASTM Test Method for Potential Reactivity of Aggregates (Chemical Method) (C 289), was found to be deleteriously reactive. Other reports [21] have also indicated the deleterious behavior of reactive glass.

Before glass is used in portland-cement concrete, it should be tested using the methods provided in the Appendix of ASTM Specification for Concrete Aggregates (C 33), and specifically those given in Paragraph X1.1.2 of ASTM C 289 and Paragraph X1.1.3 of the ASTM Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method) (C 227).

Nondeleterious reactive glass is manufactured and is available for use in concrete.

Wood

Current trends in the use of new or unusual materials in concrete are due to the emphasis on conservation of energy and utilization of wastes and by-products. Among the materials proposed for use in concrete is wood (including bamboo, fibers, bark, jute, cotton, and rice stalks and hulls).

Problems incidental to the use of natural cellulosic materials have included adverse effects of sugars on concrete setting and degradation of fibers due to the high concrete alkalinity. Further, high differential thermal coefficients of expansion of some of these materials and unaccommodative volume changes can cause cracks to develop. These factors are perhaps foremost in precluding the use of many cellulosic materials.

Additional problems incidental to the use of natural-cellulosic materials include swelling upon moisture absorption and subsequent shrinkage after drying, and chemical degradation due to contact with calcium hydroxide solutions. Prior treatment of the cellulosic materials, such as impregnation or coating techniques, or carbonation of the portland-cement paste [22], are possible ways for improving their utilization.

Sawdust, wood pulp, and wood fibers have been incorporated in mortars and concretes, and timbers have been embedded in or placed in intimate contact with concrete in composite constructions. The use of fresh untreated sawdust, wood chips, or fibers in concrete commonly results in very slow setting and abnormally low strength because of interference with normal setting and hardening processes by carbohydrates, tannins, and possibly other substances in the wood. The amount of such substances differs with wood species and from time-to-time with place-of-origin within a single species. Softwoods generally give less trouble in this respect than hardwoods.

Many admixtures and wood treatments have been proposed or used to circumvent the influence of wood constituents on setting and hardening. Addition of hydrated lime to the mixture, in an amount equivalent to one third to one half of the cement by volume, has been found effective in overcoming this action [23]. The treatment is usually effective with mixed softwoods, except when a high proportion of larch or Douglas fir is present.

Five percent calcium chloride by weight of the cement is sometimes added, as well as hydrated lime. With woods of high tannin or carbohydrate contents, the addition of lime with or without calcium chloride is not effective. Other treatments that have been suggested include soaking in sodium silicate solutions, moistening the wood with 1 % sulfuric acid for 4–14 h then neutralizing with "milk of lime," and treating it with 37 %

TABLE 5—Resistance of Plastics to Strong Alkalies [26]

Class of Material	Resistance
Polyethylene	excellent
Polymethyl methacrylate	poor
Polypropylene	excellent to good
Polystyrene	excellent
Polystyrene acrylonitrile	excellent to good
Polytetrafluoroethylene	excellent
Polytrifluorochloroethylene	excellent
Polyvinyl chloride and polyvinyl chloride vinyl acetate (rigid)	excellent
Polyvinyl chloride and polyvinyl chloride vinyl acetate (plasticized)	fair to good
Saran (monofilament grade)	fair to good
Epoxy (unfilled)	excellent
Melamine (formaldehyde)	poor
Phenol (formaldehyde)	poor
Polyester styrene-alkyd	poor
Urea (formaldehyde)	poor

aluminum chloride solution or 50 % zinc-chloride solution in a rotary barrel with beater. A treatment found by Parker [23] to be effective with all sawdust woods evaluated consists of the following consecutive steps: (a) boiling in water, (b) draining and washing with water, (c) reboiling with a 2 % water solution of ferrous sulfate, and (d) draining and rewashing.

Concrete made with wood aggregate has considerably greater volume change on wetting and drying, or simply with changes in external humidity, than concrete made with mineral aggregates. If the element is restrained, drying may lead to cracking. If drying is not uniform, the element may warp. The pretreatments mentioned previously have only a small influence on these volume changes. Various methods have been used to reduce the changes in volume with changes in moisture. Some of these methods involve encasement of the wood particles or of the finished product in a material of low permeability to moisture; but the details of such treatments and the results achieved have not, in general, been revealed.

Timbers embedded in concrete sometimes have been observed to deteriorate. The harm is done by calcium hydroxide, which causes dissolution of lipins, and decomposition, chiefly of pentosans, to a smaller extent of lignin, and least of all to cellulose. The most suitable wood for embedment is said to be pine or fir, preferably of a type with high resin content [24].

Plastics

The use of plastics in concrete and concrete construction has increased significantly. Plastic products are now being used as pipes, conduit shields, sheaths, chairs, waterstops, and joint fillers. Their compatibility with concrete is thus important.

The principal chemicals in concrete that could conceivably attack plastics are calcium hydroxide, sodium hydroxide, and potassium hydroxide, which create a minimum pH of 12.4. The following plastic groups have excellent resistance to these alkalies at 24°C (75°F) [25]: polyethylene, styrene copolymer rubber-resin blends poly (vinyl chlorides), Types I and II, and polytetrafluoroethylene.

Another source, Ref [26], provides information on the resistance of plastics to strong alkalies (Table 5).

TABLE 6—Some Applications of Fibers in Concrete

Type of Use	Type of Fiber
Cast-in-place and precast bridge deck units; overlays; structural bridge deck elements; pavement; dolosse; boats; poles; tunnel linings; rock slope stabilization; highway, street, and airfield pavements; sluices; industrial floors	steel and polypropylene
Maintenance and repairs to dams, slabs, pavements, bridges, culverts, etc.	steel and polypropylene
Industrial floors, slab overlays, pile caps, pavements	steel, glass, and polypropylene
Miscellaneous small precast items (burial vaults, steps, garden units, etc.)	glass and polypropylene
Pipes, sheets, boards, fence posts, panels, piles, building panels	asbestos, glass cellulose, and polypropylene

Fibers

In a broad sense, fibers used for reinforcing concrete are small versions of conventional steel reinforcement, and they provide a similar service. For fibers to be useful and effective, they must enhance the physical attributes of concrete and be durable. Among the desirable characteristics that fibers can impart to concrete are increased flexural strength, increased resistance to fatigue and impact, and increased fracture resistance. Applications of fiber-reinforced concrete are shown in Table 6. A report on fiber-reinforced concrete prepared by the American Concrete Institute [27] provides little information regarding their corrosion characteristics.

Among the materials that have been used as fibers in concrete are steel, glass, polyethylene, polypropylene, nylon, asbestos, and carbon. Organic fibers are considered to rely upon mechanical interlock, and steel and glass upon chemical interactions, to develop adhesion to the cement matrix. Steel fibers have also been produced as crimped or deformed fibers so that they mechanically interlock in addition to chemically bond. Some glass fibers can corrode and also cause embrittlement as discussed in the section on Glass Fibers.

Steel

Good durability of steel-fiber reinforced concrete has been reported [28,29]. However, steel fibers are subject to the same type of corrosion as reinforcing steel, and thus the durability of concrete made with steel fibers and exposed in environments chemically aggressive to the steel may be poor. The deterioration of steel is enhanced, particularly by chlorides. It has been reported that steel-fiber reinforced concrete used for pavements, bridge decks, and other similar usages where chloride-deicing agents are used, can suffer reductions in strength [30,31]. If cracks are present, rusting will be initiated at locations where the fibers are exposed in the cracks. Obviously, chlorides should not be a component of admixtures used in steel fiber-reinforced concrete, and the concrete should have as low a permeability as workability and water-ratio will permit.

In studies of steel-fiber reinforced beams in a simulated seawater environment for eight years, rusting of fibers to depths of 0.34 cm (1⁄16 in.) occurred [32]. Because of the lack of electrical continuity between the steel fibers, electro-chemical corrosion cannot develop on a large scale.

Steel fibers and steel filings are used in proprietary floor toppings to enhance wear characteristics desirable in warehouses. The volume percentage of metal used is significantly greater than for steel fiber-reinforced concrete. Deterioration of surfaces has resulted because of rusting of the fibers. In one such case, chloride equivalent to a purposeful calcium chloride addition (1 % by mass of portland cement) was present in the concrete on which a topping shake containing steel fibers had been applied. This situation points out the desirability of not using calcium chloride, either as an admixture to concrete mixtures or, along with sodium chloride (rock salt), as deicing chemicals.

Glass Fibers

Glass fibers can be sensitive to alkalis in portland cement paste. Early work using glass fibers reflected the adverse effects of alkali-silica reactivity [33].

Glass fibers formulated with zirconia were thought to be resistant to alkali degradation [34,35]. However, the loss of fracture resistance caused by chemical changes within glass-fiber reinforced concrete (which occurs prior to five-year exposures in wet environments) was as dramatic as previously experienced. The embrittlement associated with that exposure is thought to result from reformation of cement hydration products in and tightly around the fibers so that fiber encasement is enhanced and failures are due to fracture rather than pullout of fibers [36]. That explanation does not entirely satisfy all aspects of the situation, however, and further research is needed [37,38]. New glass fibers coated with chemically inert coating have been used in an attempt to overcome the strength-loss phenomenon.

Organic Materials

Polymer fibers, such as nylon and polypropylene, improve impact strength of concrete, but not tensile or flexural strengths, because they have a low modulus of elasticity. The polypropylene fibers are in common use. They increase impact strength and minimize the concrete potential for early cracking, such as that due to plastic shrinkage.

Asbestos

The use of asbestos in portland cement-based products is a thing of the past because of its potential carcinogenic effect. However, asbestos has been used in conjunction with portland cement since about 1900. Asbestos minerals are naturally occurring and include a variety of different materials that fall into two mineral groups: amphibole and chrysotile. The asbestos minerals may differ in composition but have the common composition of magnesium silicate, and alkalies—magnesium and iron may freely substitute for each other.

Although the durability of embedded asbestos fibers is not considered a problem, some corrosion of the fibers may occur after prolonged periods because of chemical reactions with calcium hydroxide in the portland-cement paste. The chemical reactions have been found to be topo-chemical and occur on fiber surfaces and on cleavage planes. Corrosion products are probably magnesium hydroxide, magnesium carbonate, magnesium silicate hydrates, and low lime calcium silicate hydrates [39]. The alteration of the asbestos fibers does not adversely affect properties of the concrete because the alteration is restricted to fiber surfaces.

Concrete

The current emphasis on recycling of materials and the razing of old concrete structures and pavements have resulted in the use of old concrete as aggregates for new concrete. For such use, the old concrete is crushed and graded and used either alone or blended with other aggregates [40–42]. Crushed concrete has had principal use as a base for concrete made with conventional aggregates.

The workability of concrete made with recycled concrete aggregates is about that of concrete made with conventional aggregates. However, its compressive strength is about 75 %, and its modulus of elasticity is about 60 % of that for conventional concrete [43].

Buck reported that concrete made with recycled aggregate had resistance to cyclic freezing [44]. Aggregates for use in concrete must possess certain necessary physical and chemical characteristics, such as those required in ASTM C 33. Because the concrete has seen prior service in a given exposure and environment for lengthy periods does not necessarily mean that it will perform similarly as concrete aggregate in other service and exposures. For example, concrete made using chemically unstable aggregate and used in an elevated structure where the exposure has been dry will probably respond differently when used in a moist environment. Concrete made with a calcium chloride addition, if used in reinforced nonchloride-containing concrete, could promote corrosion of reinforcing steel. These are two examples that demonstrate the need for establishing suitable specifications and requirements to ensure that concrete made with recycled concrete will provide adequate service. Concrete from building construction has been used for fill material as well as for concrete aggregate. Contamination of the crushed concrete aggregate by adhering gypsum plaster from plastered walls can cause internal sulfate attack.

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18

Abrasion Resistance

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Preface

IN PREPARATION OF THIS CHAPTER, THE CONTENTS of the 4th edition were drawn up. The author acknowledges the authors of the first four editions, H. L. Kennedy and M. E. Prior, M. E. Prior, R. O. Lane, and Tony C. Liu, respectively. The current edition will review and update the topics as addressed by the previous authors, introduce new technology that has been developed, and include up-to-date references.

Definitions and Types of Abrasion

ASTM Terminology Relating to Erosion and Wear (G 40) defines abrasion as "wear due to hard particles or hard protuberances forced against and moving along a solid surface." Abrasion resistance, according to American Concrete Institute (ACI) Committee on Cement and Concrete Terminology (ACI 116), is the "ability of a surface to resist being worn away by rubbing and friction" [1].

Wear of concrete surface can be classified as follows [2]:

- Wear on concrete floors.
- Wear on concrete road surfaces due to heavy trucking, and automobiles, with and without studded snow tires or chains (attrition, plus scraping and percussion).
- Wear on hydraulic structures such as dams, spillways, bridge piers, and abutments due to the action of abrasive materials carried by flowing water (erosion).
- Wear on concrete dams, spillways, tunnels, and other water-carrying systems where high velocities and negative pressure are present. This is generally known as cavitation erosion (cavitation).

The first type of wear to concrete floors can vary widely from a rubbing action to high impact and hard-wheel traffic of forklifts. Concrete floor wear is greatly increased by the introduction of foreign particles, such as sand, metal scraps, or similar materials. ACI 302.1R-04, Table 2.1 classifies floors, including design and construction considerations based on the intended use [3]. The second type of wear is caused by a rubbing action, plus an impact-cutting type of wear. This is brought about by the use of chains on automobile and truck tires or metal vehicle wheels. As the wheel revolves, it brings the metal into contact with the concrete surface with considerable impact, a process that tends to cut the surface of the concrete. The third type of wear is primarily grinding and cutting actions. The action of the abrasive particles carried by the flowing water, of course, is controlled largely by the velocity of the water, the angle of contact, the type of abrasive material, and the general surrounding conditions. The fourth type of wear, cavitation, is caused by the abrupt change in direction and velocity of a liquid to such a degree that the pressure at some point is reduced to the vapor pressure of the liquid. The vapor pockets so created, upon entering areas of high pressure, collapse with a great impact, which eventually causes pits or holes in the concrete surface. Discussion of cavitation resistance of concrete is beyond the scope of this paper; therefore, it will not be covered. An excellent source of information on cavitation erosion can be found in Refs 4 and 5.

Factors Affecting Abrasion Resistance

Factors that may affect the resistance of concrete to abrasive action should be considered in the design and construction of concrete surfaces that are to withstand abrasion due to rubbing, scouring, sliding, impact, scraping, attrition, percussion, gouging, or cutting from mechanical or hydraulic forces. Frequently, the failure of concrete to resist abrasion can be traced to cumulative effects such as soft aggregate, inadequate compressive strength, improper curing or finishing, or overmanipulation during finishing of concrete surface. The following discussion of significant factors relative to concrete resistance to abrasion illustrates the importance of the proper selection, composition, and application of concrete based on the specific type of service condition.

Quality of Aggregates

Studies by Liu [6] and Laplante et al. [7] indicated that the abrasion resistance of concrete is strongly influenced by the hardness of its coarse aggregate. The abrasion resistance can be increased appreciably by the use of maximum amount of dense, hard coarse aggregates such as traprock, chert, granite, or metallic aggregate. For example, the abrasion loss of concrete containing limestone aggregate has approximately twice as much as that of the concrete containing chert [6].

Abrasion tests carried out by Liu [6] indicated that no correlation existed between abrasion resistance of concrete and the quality of the coarse aggregate as determined by ASTM Test Method for Resistance to Degradation of Small-Size Coarse

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Aggregate by Abrasion and Impact in the Los Angeles Machine (C 131). As can be seen from Fig. 1, the Los Angeles abrasion losses are approximately equal for soft aggregate, such as limestone, and relatively hard aggregate, such as chert. However, the abrasion losses of the concrete containing these aggregates vary widely; the abrasion loss of the limestone concrete is much more than that containing chert. A similar finding was reported by Smith [8].

Schuman and Tucker [9] pointed out that the shape of aggregate particles regulates the water requirements for placing and finishing and has a direct influence on the abrasion resistance of concrete. Angular to subangular-shaped aggregate is known to improve bond, usually resulting in increased abrasion resistance.

For concrete subject to light-to-medium abrasion, good quality aggregates meeting the requirements of ASTM Specification for Concrete Aggregates (C 33) are generally acceptable. Heavy-duty floors and slabs exposed to more severe abrasive action demand well-graded hard mineral aggregates [2].

Compressive Strength

Witte and Backstrom [10], as researchers before and after them, considered the compressive strength as one of the most important factors responsible for the abrasion resistance of concrete. For the same aggregate and finishing procedure, the abrasion resistance of concrete increases with an increase in compressive strength. For example, as shown in Fig. 2, the average abrasion resistance (the reciprocal of abrasion-erosion loss) for concrete containing limestone increases approximately 44 % as the compressive strength increases from 20.7 MPa (3000 psi) to 62.1 MPa (9000 psi) [6].

However, abrasion resistance is a function of the water-ratio at the surface and of the quantity of quality aggregate, not of the compressive strength of concrete [11]. The 6-in. × 12-in. cylinder tested to measure compressive strength is not a measure of the slab surface strength [12].

Mixture Proportioning

Abrasion test results by many researchers clearly indicated that, for a given aggregate, the abrasion resistance of concrete increases with a decrease in water-cement ratio [6]. A maximum water-cement ratio of 0.45 has been specified by the U.S. Army Corps of Engineers for concrete subject to abrasion in hydraulic structures.

The effects of various admixtures, including air-entraining and water-reducing admixtures and retarders, on the abrasion resistance of concrete could not be established conclusively on the basis of the literature review. The general trend in most studies reflects an increase in abrasion loss proportional to increase in air content for any given water-cement ratio (w/c). As the compressive strength is adversely affected by increased air contents, so will the resistance of concrete to abrasion. Water-reducing admixtures, by virtue of their positive effect on the w/c and consequently on increases in strength, tend to produce a concrete of improved abrasion resistance.

Concrete Types

Studies by Holland et al. [13] and Laplante [7] indicated that adding condensed silica fume and high-range water-reducing admixture to a concrete mixture greatly increases compressive strength, which, in turn, increases abrasion resistance. The abrasion resistance of silica-fume concrete containing relatively soft limestone is similar to that of a high-strength conventional concrete mixture containing a very hard chert aggregate [13]. Apparently, for the high-strength silica-fume concrete, the hardened cement paste assumes a greater role in resisting abrasion and as such the aggregate quality becomes correspondingly less important. These very high-strength concretes appear to offer an economical solution to abrasion

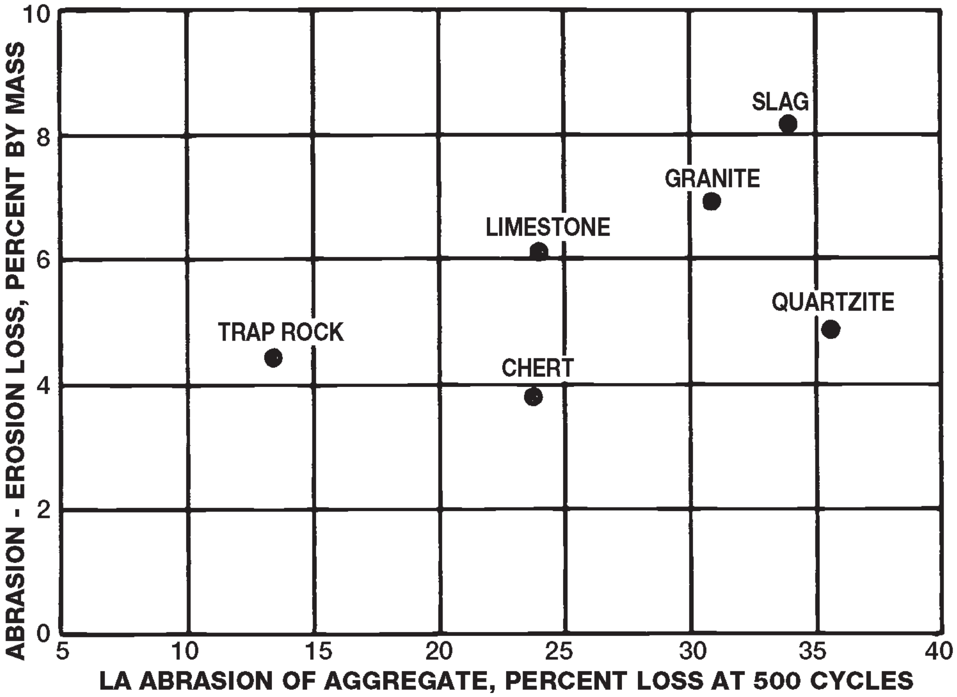

Fig. 1—Relationship between resistance of aggregate to abrasion and concrete abrasion loss.

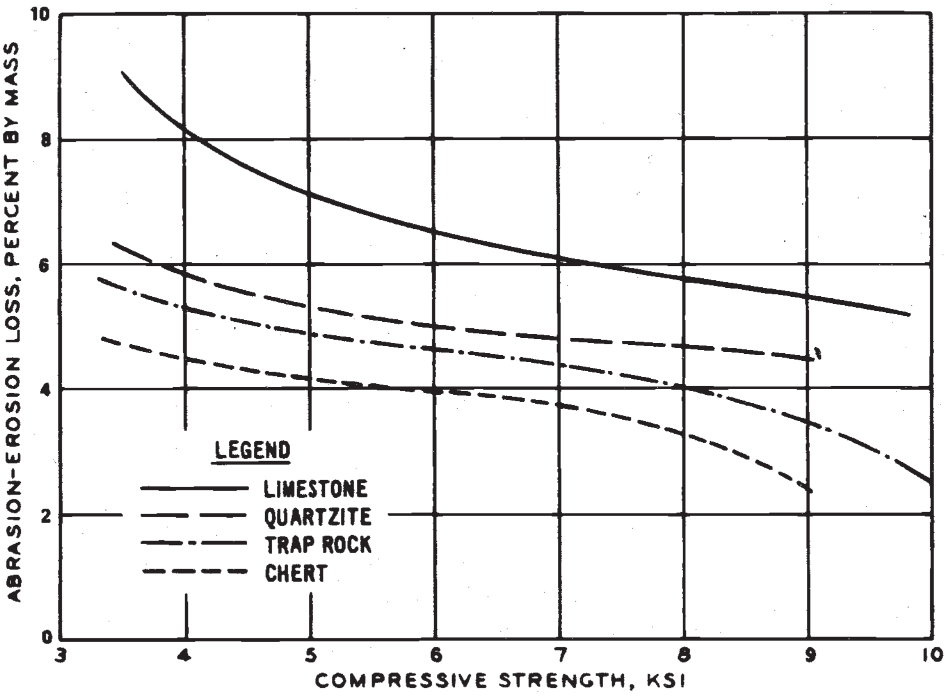

Fig. 2—Relationship between abrasion resistance and compressive strength.

problems, particularly in those areas where locally available aggregate otherwise might not be acceptable.

Tests by the U.S. Army Corps of Engineers indicated that the abrasion resistance of concrete containing fly ash is comparable to that of concrete without fly ash, as long as the concretes contain the same type of aggregate and have comparable compressive strength at the time of tests.

Shrinkage compensating concrete, when properly proportioned and cured, has an abrasion resistance from 30 to 40 % higher than portland cement concrete of comparable mix proportions [14]. Other studies with shrinkage compensating cement have indicated similar results: Nagataki and Yoneyama [15] and Klieger and Greening [16].

While the addition of steel fibers in the concrete mixture would be expected to increase the impact resistance of concrete, fiber-reinforced concrete is less resistant to abrasion than conventional concrete of the same aggregate type and water-cement ratio [17]. The abrasion losses of fiber-reinforced concrete as determined by ASTM C 1138 were consistently higher than those of the conventional concrete over wide ranges of water-cement ratio and compressive strength. This is attributed primarily to the fact that fiber-reinforced concrete generally has less coarse aggregate per unit volume of concrete than comparable conventional concrete.

Polymer-impregnated concrete for highway bridges was investigated by Fowler of the University of Texas [18]. Concrete of 34.5- to 44.8-MPa (5000- to 6500-psi) compressive strength, non-air-entrained, treated with polymer showed considerable increase in abrasion resistance. Experimental studies by Liu [6], Krukar and Cook [19], and Dikeou [20] also demonstrated remarkable improvements in concrete properties, particularly in strength and abrasion resistance due to impregnation and in-place polymerization of monomers in overlays and in mature concrete.

The relative abrasion resistance of four different types of polymer concretes (that is, polymer-impregnated concrete, polymer portland cement concrete, methyl methacrylate polymer concrete, and vinyl ester polymer concrete), which all contained limestone aggregates, was investigated by Liu [21]. Among these polymer concretes tested, the vinyl ester polymer concrete ranked first in abrasion resistance, followed by methyl methacrylate polymer concrete, polymer-impregnated concrete, and polymer portland cement concrete.

Finishing Procedures

Abrasion resistance of concrete is affected by the finishing procedures used. Tests by Kettle and Sadegzadeh [22] indicated that the abrasion resistance of concrete subjected to power finishing is significantly higher than that subjected to hand finishing. This is attributed to surface compaction and to reduction of the w/c of the surface matrix.

Finishing techniques, including wood float, magnesium float, steel trowel, and hard-steel trowel finishes, are also compared with abrasion resistance of concrete in a study by Fentress [23]. The wood float tends to tear the surface and to displace the aggregate. The magnesium float, despite ease of finishing, causes a rough-textured surface and a lowering in abrasion resistance. Both the steel trowel and hard-steel trowel produce smooth surfaces, closing any existing imperfections and providing excellent resistance to abrasion.

The key to durable, cleanable concrete slabs can be achieved by proper finishing procedures and in a reduction in the w/c [24]. Combining these demands in one application, Ytterberg demonstrated that a deferred topping finish from which surface water is removed by a vibratory absorption process yields highest abrasion resistance [24]. The technique requires that after the water of workability has been removed, the surface is blade-floated, and upon stiffening, troweled.

Abrasion tests carried out by Kettle and Sadegzadeh [22], the U.S. Bureau of Reclamation [25], and the U.S. Army Corps of Engineers [21] confirmed that vacuum-treated concrete surfaces are considerably more resistant to abrasion than surfaces with either nontreated lean or richer concrete. The improvement in abrasion resistance of vacuum-treated concrete is due principally to the reduction of water content in the concrete mixture. This treatment is most effective on concrete with a high w/c.

Concrete wearing surfaces can be improved by the use of cast-on hardeners or "dry shakes" consisting of cement/hard-aggregate mixtures. Pockets of bleed water should be removed prior to the application of the dry shake. After the dry shake is applied to the concrete, it should be given enough time to absorb water prior to floating. Once the dry shake has absorbed water, it is power-floated and then steel troweled into the surface without the use of additional water to produce a hard, dense topping layer. Testing by the Portland Cement Association in 1970 demonstrated that abrasion resistance is significantly improved using cast-on surface hardeners with hard, steel-trowel, finish as compared to ordinary concrete without surface hardeners [26].

Curing

Efficient curing increases the abrasion resistance. A correlation of curing time and abrasion resistance reported by Sawyer [27] involved a series of tests comprising a wide range of cement contents, w/c, and incremental curing. From this study, it is apparent that marked improvement in abrasion resistance can be expected with extended curing time, especially for surfaces composed of leaner concrete.

A laboratory program conducted by the California Division of Highways [28] considered the effect on abrasion of such variables as slump, finishing, curing, and surface treatments using linseed oil. Test data indicated, among these variables, that the greatest abrasion losses encountered were associated with less-than-adequate curing procedures.

For concrete floor and slab construction using Type I portland cement, at least five days of curing at concrete temperatures of 21°C (70°F) or higher and a minimum of seven days at temperatures of 10–21°C (50–70°F) are required to assure adequate abrasion resistance [3].

Surface Treatment

Certain chemicals (magnesium and zinc fluorosilicates, sodium silicate, gums, and waxes) serve to prolong the life of older floors and are considered an emergency measure for treatment of deficiencies in relatively pervious and soft surfaces that wear and dust rapidly [29]. Treatment with fluorosilicates densifies and hardens surfaces and improves abrasion resistance of old, worn floors, but the improvement by the two types of fluorosilicates is not of equal magnitude [30]. While the magnesium fluorosilicate is more resistant to rubbing action simulated best by a revolving-disk-type abrasion machine, the zinc fluorosilicate shows greater resistance to impact-type wear as imposed by the dressing-wheel-type machine.

For poorly cured or porous concrete surfaces, beneficial effects from treatment with linseed oil were demonstrated by an increase in abrasion resistance of as much as 30 % [31].

Paints and coatings used to seal concrete surfaces and to protect the concrete from the attack of the environment or chemicals [32] possess only limited abrasion resistance and any test simulation to evaluate their effectiveness is difficult. However, some coatings, among them vinyl and heavy rubber, if properly bonded remain fairly resilient and effective in protecting concrete from abrasive actions. Several types of surface coatings including polyurethane, epoxy-resin mortar, furan-resin mortar, acrylic mortar, and iron-aggregate toppings have exhibited good abrasion resistance in laboratory tests [21]. However, problems in field application of surface coatings have been reported. These have been due primarily to improper surface preparation or thermal incompatibility between coatings and concrete. More recently, formulations have been developed that have coefficients of thermal expansion more similar to that of the concrete substrate [4].

Abrasion Test Methods

The techniques and test methods that have been employed for over a century to evaluate the abrasion resistance of concrete have attempted with varied success to reproduce the typical forces detrimental to concrete surfaces. Preceding any standardization of laboratory methods or of testing machines, reliance was placed initially on the use of rattlers that provide abrasive action based on the tumbling of steel balls impacting a test surface [33]. Numerous modified versions of the rattler principle continued to be the dominant test procedure along with the revolving metal disk pressed against a small concrete specimen [34]. The drill-press-type abrasion machine [35] in modified form still is used by highway departments and enjoys some popularity because of its simple design.

Currently, there are four standard ASTM test methods for evaluating the resistance of concrete subjected to various types of abrasive actions. The background, significance, and applicabilities of these ASTM test methods are presented here.

ASTM C 418

ASTM Test Method for Abrasion Resistance of Concrete by Sandblasting (C 418) dates back to 1958 and is based on the principle of producing abrasion by sandblasting. This procedure simulates the action of waterborne abrasives and abrasives under traffic on concrete surface. It performs a cutting action that tends to abrade more severely the less resistant components of the concrete. Adjustments in the pressure used and the type of abrasive permit a variation in the severity of abrasion that may be used to simulate other types of abrasion.

This test method was modified by ASTM Subcommittee C09.03.13 on Methods of Testing Concrete for Resistance to Abrasion from the original Ruemelin blast cabinet equipped with an injector-type blast gun with high-velocity air jet (Fig. 3). The control over such variables as gradation of the silica sand, air pressure, rate of feed of the abrasive, distance of the nozzle from the surface, and the area of the shielded surface is critical. The 1958 standard specified carborundum or silica sand. This procedure was revised in 1964 calling for the explicit use of silica sand that allows for closer control of the abrasive action. The 1976 revision specifies the use of oil-base modeling clay of known specific gravity for the filling of voids in the abraded concrete surface. This permits accurate measurement of abrasion by volume displacement and replaces the old weight-loss determination. Rushing [36] compared the 1958 and 1964 procedures of ASTM C 418 in a laboratory study and found the later revision permits a more accurate determination of abrasion loss. The current revision now allows for a ceramic nozzle in addition to cold rolled steel and hardened tool steel.

ASTM C 779

For almost two decades, ASTM C 418 remained the only specified standard for testing concrete resistance to abrasion. From

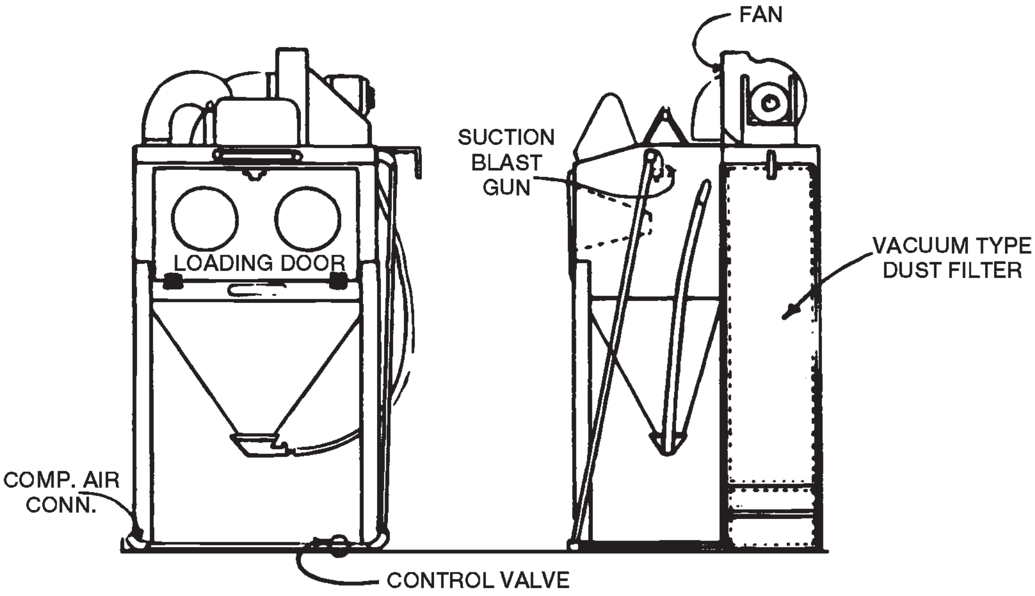

Fig. 3—Sand blast cabinet.

1964 through 1971, ASTM Subcommittee C09.03.13 initiated a number of major test programs conducted by independent laboratories. The results of these comparative programs established such parameters as severity and reproducibility, leading to the selection of three abrasion machines and their inclusion in one ASTM standard. ASTM Test Method for Abrasion Resistance of Horizontal Concrete Surfaces (C 779) covers three testing procedures: (a) the revolving-disk machine, (b) the dressing-wheel machine, and (c) the ball-bearing machine. All three machines are portable and adaptable for laboratory and in-place field abrasion testing.

The revolving-disk machine (Fig. 4) introduces frictional forces by rubbing and grinding. Sliding and scuffing is accomplished by rotating steel disks in conjunction with abrasive grit. The revolving-disk machine was redesigned by Master Builders Company, Cleveland, Ohio, and was essentially

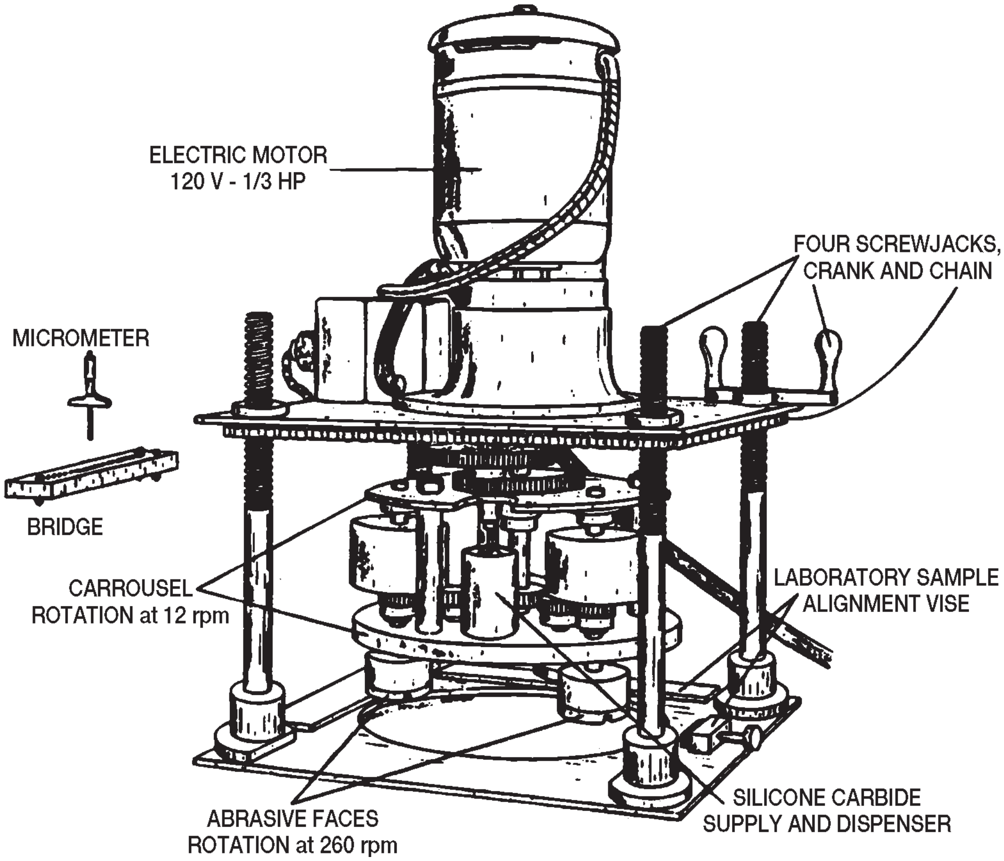

Fig. 4—Revolving-disk abrasion test machine.

patterned after a machine developed by the National Institute of Standards and Technology. Among the three standard methods, the revolving-disk machine provides the most reproducible results. A supply of No. 60 silicon carbide abrasive is fed to the disks at a rate of 4–6 g/min. A test period of 30 min generally produces significant wear on most concrete surfaces, but it is recommended to extend the test period to 60 min if information on the longtime abrasion resistance is desired. Davis and Troxell [37] reported that the depth of wear for periods of 30–60 min is about the same for ordinary slabs as for heavy-duty concrete surfaces. A plane surface resulted in either case regardless of the variation in the quality of the hardened cement paste or the toughness of the aggregate that became exposed as the depth of wear increased. The abrasive mode of this procedure best simulates wear by light-to-moderate foot traffic and light-to-medium tire-wheeled traffic or moving of light steel racks, etc.

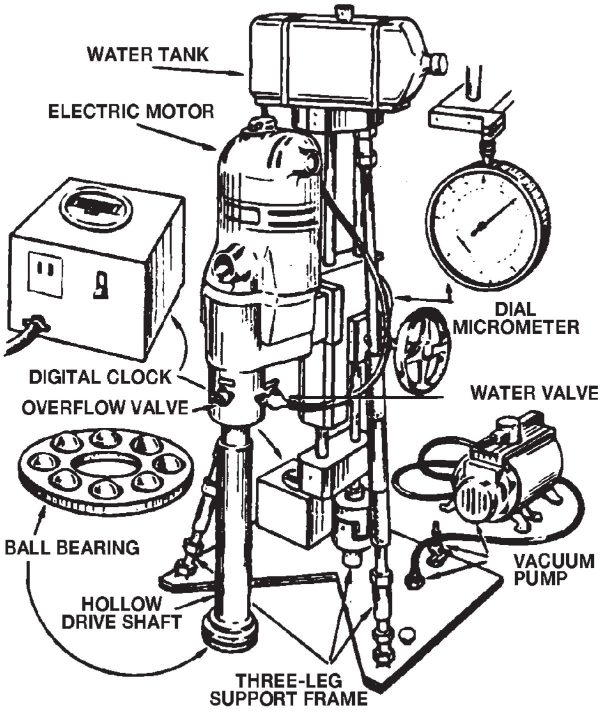

Fig. 6—Ball-bearing abrasion test machine.

In contrast to the disks, the dressing-wheel machine (Fig. 5) imparts high concentrated compressive forces with high impact stresses. The dressing-wheel machine is similar to the revolving-disk machine in appearance, except for three sets of seven dressing wheels mounted on horizontal shafts that take the place of the three rotating steel disks. However, no abrasive material is employed with this machine. Initial and intermediate measurements of the test path are taken with a depth micrometer. Tracking of the dressing wheel normally leaves a grooved path with the test surface being irregular and fairly rough as harder aggregate particles stand out from the softer aggregate particles and mortar that are abraded more quickly. On ordinary-finished concrete slabs, the dressing wheels produce a depth of wear more than double that obtained with the revolving-disk machine for the same test duration. Yet, approximately equal depths of wear are obtained from both machines for the same test period when hard troweled finished floors are tested. The coefficient of variation established by the Berkeley study [37] for slabs abraded with the dressing-wheel machine is several times as great as that for the revolving-disk machine. These results are essentially concurrent with a parallel study conducted in the Portland Cement Association laboratories [26]. Abrasion of concrete induced by the dressing-wheel machine closely simulates the rolling, pounding, and cutting action of steel wheels or the effect of studded tires.

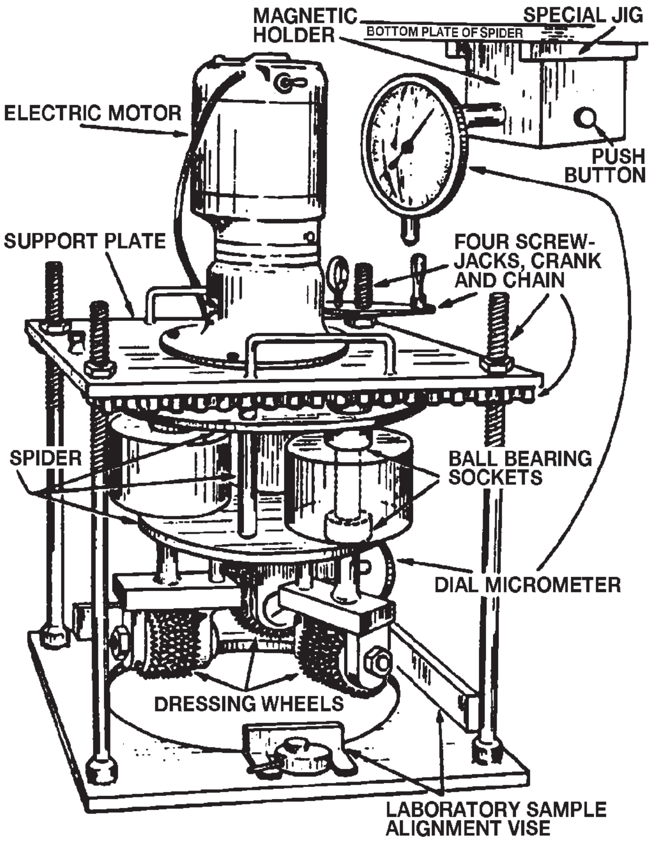

Fig. 5—Dressing-wheel abrasion test machine.

Repeated dynamic loading through strong impact, compressive forces, and high-speed rolling constitutes the abrasive action of the ball-bearing machine (Fig. 6). The ball-bearing machine operates on the principle of a series of eight ball bearings rotating under load at a speed of 1000 rpm on a wet concrete test surface. Water is used to flush out loose particles from the test path, bringing the ball bearing in contact with sand and stone particles still bonded to the concrete surface, thus providing impact as well as sliding friction. During the test, abrasion readings are taken every 50 s with a dial micrometer mounted directly to the supporting shaft allowing readings "on the fly." Readings are continued for a total of 1200 s or until a maximum depth of 3.0 mm (0.12 in.) is reached. In its abrasive severity, the ball-bearing machine exceeds both the revolving disk and the dressing wheel, also producing the highest coefficient of variation among the three procedures (Table 1). The effects of the ball-bearing machine on concrete surfaces indicate abrasive action becomes progressively more severe as the test continues, due to soft and hard spots causing the core barrel to bounce to high angular speed. This test procedure has merit in that the ball bearings are similar to rolling wheels and are more typical of actual loadings by steel-wheel traffic to which a concrete floor may be exposed.

Experience with testing of abrasion by ASTM C 779 demonstrates relatively good reproducibility over a fairly wide range of concrete surface types and conditions when the abrasive action is moderate, for instance, through attrition. More severe abrasion due to percussion, impact, cutting, or rolling creates erratic action and is less conducive to reproducibility. In general, loss in test accuracy may develop due to the following factors:

- Rapid wear of dressing wheels, revolving disks, and steel balls and failure to replace them regularly as specified;
- Failure to remove dust and loose-abraded material regularly from the test surface;
- Improper selection of a representative test area or specimen;
- Differences in age levels of concrete at which test results are compared;
- Arbitrary increase in the length of test or flow of sand; and
- Insufficient number of wear readings.

Excluding these variables, the within-laboratory precision shown in Table 1 indicates the lowest coefficient of variation is obtained with the revolving-disk method, increasing in order with dressing wheels and ball bearings.

ASTM C 944

ASTM Test Method for Abrasion Resistance of Concrete or Mortar Surfaces by the Rotating-Cutter Method (C 944) gives an indication of the relative abrasion resistance of mortar and concrete based on testing of cored or fabricated specimens. This test method has been successfully used in the quality control of highway and concrete bridges subject to traffic. This method is primarily intended for use on the top ends of 152-mm (6-in.) diameter concrete cores, mortar specimens, or other samples of concrete of insufficient test area to permit the conduct of tests by ASTM C 418 and C 779. This test method produces a much more rapid abrasive effect than the other test methods. The test apparatus is a fairly simple piece of equipment consisting of a rotating cutter and a drill press or similar device with a chuck capable of holding and rotating the abrading cutter at a speed of 200 rpm. Figure 7 shows a rotating-cutter drill press. The difficulty in maintaining a constant load on the abrading cutter when using the lever, gear, and spring system of a drill press has been eliminated by placing a constant load of 98 N (22 lbf) directly upon the spindle that turns the cutter. General practice is to clean the surface occasionally during the test by blowing the dust off the test specimen. The reproducibility of test results has been poor, with the single-operator coefficient of variation of more than 20 %.

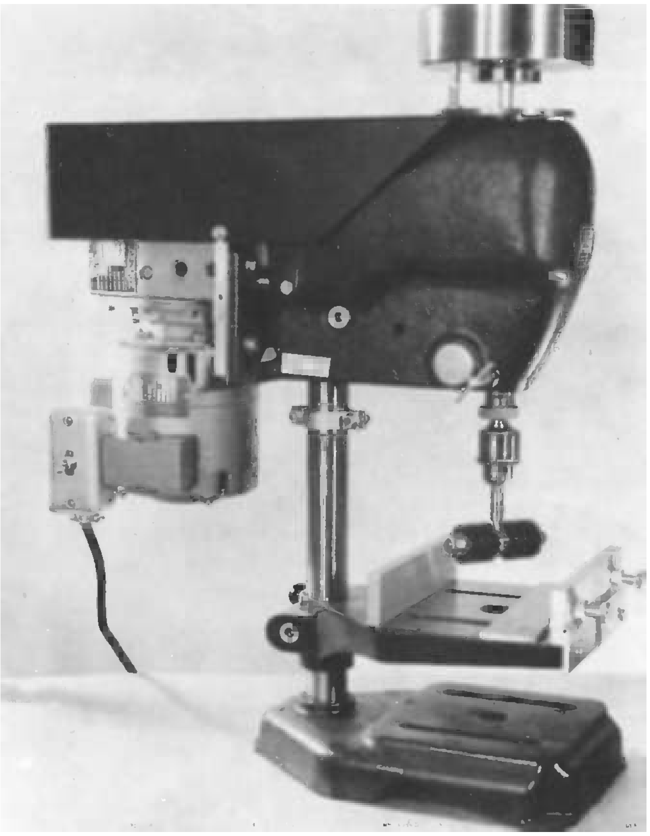

Fig. 7—Rotating-cutter drill press.

TABLE 1—Within-Laboratory Precision for Single Operator, ASTM C 779

Procedure	Coefficient of Variation, percent of mean[a]	Acceptable Range of Two Results, percent of mean[a]
A. Revolving disk	5.51	15.6
B. Dressing wheel	11.69	33.1
C. Ball bearing	17.74	50.2

[a] These numbers represent respectively the 1S % and D2S % limits as described in ASTM Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials (C 670), in the section on Alternative Form of the Precision Statement.

ASTM C 1138

ASTM Test Method for Abrasion Resistance of Concrete (Underwater Method) (C 1138) was originally developed by Liu [21] in 1980 for evaluating the resistance of concrete surfaces subjected to the abrasive action of waterborne particles on hydraulic structures such as stilling basins and outlet works. ASTM adopted this test procedure with some minor modifications in 1989. The apparatus consists of essentially a drill press, an agitation paddle, a cylindrical steel container that houses a disk-shaped concrete specimen, and 70 steel grinding balls of various sizes. The detailed cross-sectional view of the test apparatus is shown in Fig. 8. The water in the container is circulated by the immersed agitation paddle that is powered by the drill press rotating at 1200 rpm. The circulating water, in turn, moves the abrasive charges (steel grinding balls) on the surface on the concrete specimen, producing the abrasion effects. Testing, totaling 24 h, generally produces significant abrasion in most concrete surfaces. The standard test consists of six 12-h test periods for a total of 72 h. Additional testing time may be required for concrete that is highly resistant to abrasion. This test method can duplicate

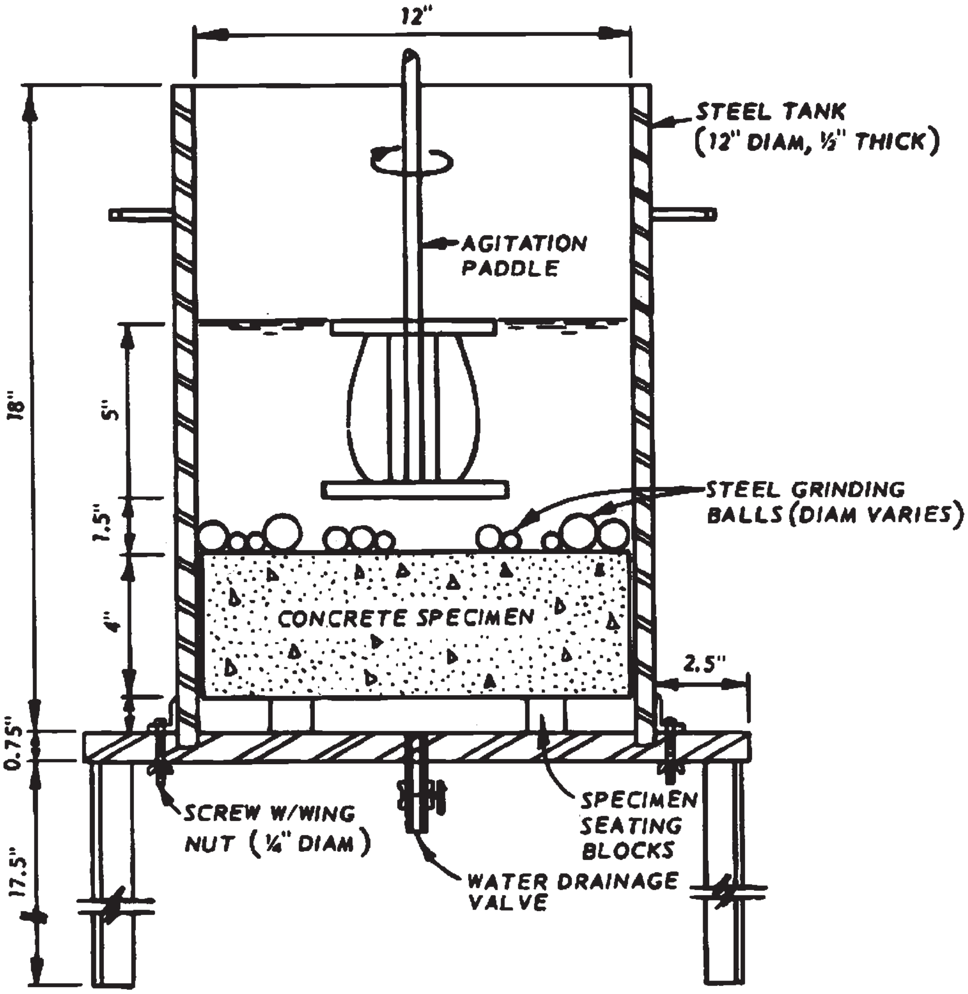

Fig. 8—Test apparatus, ASTM C 1138.

well the abrasive action of waterborne particles in the stilling basins. This method is not, however, intended to provide a quantitative measurement of length of service that may be expected from a specific concrete.

The abrasion loss, as determined by the original Corps of Engineers' test method [21], was expressed as a percentage of the original mass of the specimen. ASTM Subcommittee C09.03.13 revised the method by calculating the volume loss, or the average depth of wear, at the end of the test time increment to eliminate differences in results resulting from the allowable variations in specimen size. The single laboratory coefficient of variation made from a single batch of concrete is 14 %.

Application of Test Methods

As discussed in the previous section, ASTM C 418, C 779, C 944, and C 1138 offer six distinct procedures that simulate various degrees of severity and types of abrasive or erosive forces. Table 2 serves as a general guide for possible applications of the six standard procedures for various categories of abrasion. This tabulation attempts to correlate the severity of in-place wear with the abrasive action particular to each of the test methods. However, it is necessary to bear in mind that there is overlap in the type of abrasion imparted by these test applications and that a specific service condition may be reproduced by more than one procedure.

TABLE 2—Application of Test Procedures

Type of Abrasion	ASTM C 418	ASTM C 779 A B C	ASTM C 944	ASTM C 1138
Foot traffic or light-to-medium tire-wheeled traffic, etc.		X	X	
Forklift, heavy tire-wheeled traffic, automobile with chains, heavy steel-wheeled traffic, or studded tires, etc.		X X	X	
Abrasive erosion of waterborne particles on hydraulic structures	X			X

Conclusion

Concrete materials are susceptible to deterioration due to the abrasive action of environmental and man-made factors. In addition, intrinsic conditions related to properties of concrete and construction techniques contribute to the reduction in abrasion resistance. Proper selection of concrete-making materials, mixture proportion, curing, and finishing procedures are the basic requirements applying to any concrete subjected to abrasion from vehicular traffic or water-borne particles. Effects of variables on reduced abrasion resistance of concrete have proved to be cumulative, which may explain why only certain portions of a concrete surface may fail in resisting abrasion while the remainder gives satisfactory performance.

There are four ASTM test methods, that is, ASTM C 418, C 779, C 944, and C 1138, for evaluating the resistance of concrete subjected to various types of abrasive actions. These test methods serve to (1) evaluate, predict, or accept the quality of concrete surfaces; (2) evaluate specific effects of variables such as concrete-making materials, curing, finishing procedures, surface hardeners, or coating materials; (3) compare various types of concrete surfaces under simulated abrasion conditions; and (4) verify products or systems to meet specifications.

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19

Elastic Properties, Creep, and Relaxation

Jason Weiss[1]

Preface

A CHAPTER ON ELASTIC PROPERTIES OF CONCRETE first appeared in *ASTM STP 169* (1956) authored by L. W. Teller of the U.S. Bureau of Public Roads (now FHWA). Robert E. Philleo authored the chapters appearing in *ASTM STP 169A* (1966) and *ASTM STP 169B* (1978) under the title "Elastic Properties and Creep." The chapter on elastic properties was reprinted in *ASTM STP 169C* as it appeared in *ASTM STP 169B*. This version is based largely on these earlier chapters; however, this chapter has been modified to include information on nondestructive testing, elastic modulus inferred from other test methods, and early age creep and relaxation measurements.

Importance of Elastic Properties and Creep

Engineers need to be able to compute deflections of structures, to compute stresses from observed strains, to proportion sections, and to determine the quantity of steel required in reinforced concrete members. In each of these calculations engineers need material properties that can relate stress and strain. These properties are commonly referred to as the elastic properties. Strictly speaking, the stress-strain response of concrete is nonlinear and inelastic; however, it is frequently assumed that for low load levels (stresses less than 50 % of the strength and strains less than 1000 $\mu\varepsilon$ in compression and 100 $\mu\varepsilon$ in tension) the relationship between stress and strain can be described using a linear relationship. In this linear relationship the elastic modulus describes the ratio of the change in stress and change in strain. While the elastic properties can be used to describe the initial deformation under loading, concrete can exhibit increased deformations over time due to the presence of a sustained load. Creep describes the slow, progressive deformation of a material under a sustained loading. Relaxation describes the slow reduction in stress over time due to a system displacement. This chapter will review some of the basic elastic properties, compare these properties with other construction materials, illustrate why concrete may or may not be elastic, discuss common test methods for obtaining elastic properties and the stress strain response, discuss creep and relaxation, and discuss potential applications and future needs.

Background Information About Elastic Constants and Properties

When a load is applied to a material body it deforms. For many materials the body will return to its original dimensions after the load is removed. This, in its simplest form, describes a material's elasticity and the concept can be simply illustrated by considering a spring. When the spring is pushed or pulled it changes length and when sthe load is removed it returns to its original position (Fig. 1a). Hooke proposed this concept in 1678 by stating that the deformation of a body (δ) was linearly proportional to the force that is applied (F). Over time this proportionality constant (K) was termed as a spring constant that can be written mathematically as

$$F = K\delta$$

where

F = the applied force or load
K = the spring constant
δ = the deformation

In actuality, the spring coefficient can be thought of as a "stiffness" of the material/structure that is dependent on the given geometry of body that is loaded as well as the properties of the material. To overcome this limitation the force can be written in terms of the stress (i.e., $\sigma = \frac{F}{A}$, where A is the cross-sectional area) and the deformation can be written as a strain (i.e., $\varepsilon = \frac{\delta}{L}$, where L is the original specimen length) which gives rise to the more familiar form of Hooke's Law for uniaxial loading where stress is related to strain through a proportionality constant which is referred to as the elastic modulus or Young's Modulus (Fig. 1b). The elastic modulus is essentially only a function of the material and as such it is independent of specimen size and geometry, thereby making it a material property.

$$\sigma = E\varepsilon$$

where

σ = the applied stress
E = the elastic modulus
ε = the strain

When stress is applied in a given direction, there are changes in the dimension of the perpendicular directions. The magnitudes of the lateral strains are different for different materials.

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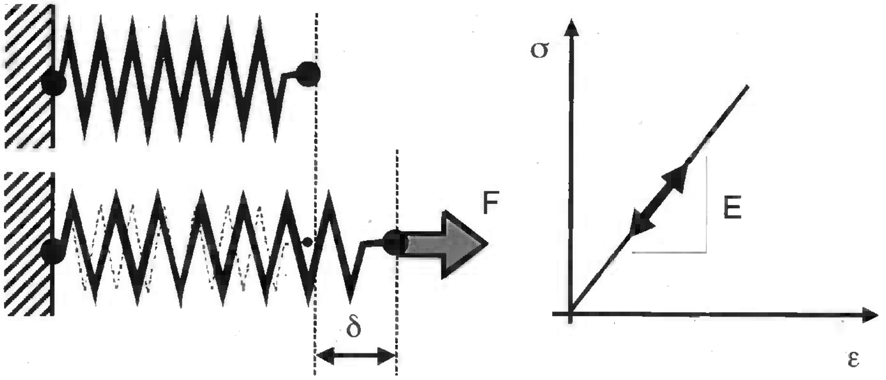


Fig. 1—a) Conceptual illustration of the relationship between force and deformation using a stiffness constant (K), and b) the linear relationship between stress and strain using the elastic modulus (E).

Thus, two parameters are required to describe the elastic behavior of a material. The parameters may take many forms, but the two most commonly used are elastic modulus and Poisson's ratio. Poisson's ratio is defined in ASTM Standard Terminology Relating to Methods of Mechanical Testing (E 6) as "the absolute value of the ratio of transverse strain to the corresponding axial strain resulting from uniformly distributed axial stress below the proportional limit of the material." The transverse strains are opposite in direction to the axial strains and can be described using the following relationship. A new material property (Poissons Ratio, μ) is introduced in the following equation to relate the axial and lateral strains:

$$\varepsilon_{Lateral} = -\mu\varepsilon_{Axial} = -\mu\,\frac{\sigma_{Axial}}{E}$$

where

σ_{Axial} = the applied stress in the axial direction
μ = Poisson's Ratio
ε_{Axial} = the strain in the axial direction
E = the elastic modulus, and
$\varepsilon_{Lateral}$ = the strain in the lateral direction.

Table 1 shows typical values for the elastic modulus and Possion's ratio for mature concrete and other commonly used construction materials. It can be seen that the elastic modulus for concrete is lower than that of most metals while it is slightly higher than that of wood. Poisson's ratio falls between 0 and 0.5 for nearly all materials. It can be seen that Poisson's ratio for concrete is lower than that of most metals and it is consistent with many ceramic materials.

It is important to note at this time that these two elastic properties (i.e., E and μ) are generally used to describe the response of many materials since a more general relationship can be written to account for cases of multi-axial loading. When an element is subjected to simultaneous normal stresses in each of three axial directions (*x, y, z*), the resulting strain components can be obtained from the following equations where the subscripts refer to actions in a specific direction.

$$\varepsilon_\chi = \frac{1}{E}\left[\sigma_\chi - \mu\left(\sigma_y + \sigma_z\right)\right]$$

$$\varepsilon_y = \frac{1}{E}\left[\sigma_y - \mu\left(\sigma_x + \sigma_z\right)\right]$$

$$\varepsilon_z = \frac{1}{E}\left[\sigma_\zeta - \mu\left(\sigma_x + \sigma_y\right)\right]$$

It is also important to note that once two elastic properties are known (i.e., in this case E and μ) any other elastic property can be determined. For example, since the elastic modulus and Poisson's ratio are known they can be used to calculate the shear modulus (G). The shear modulus, also called the modulus of rigidity or torsional modulus, is the ratio of shear stress to shear strain. Shear stress is defined in ASTM E 6 as "the stress or component of stress acting tangential to a plane," and shear strain is defined as "the tangent of the angular change between two lines originally perpendicular to each other." It can be

TABLE 1—Elastic Modulus and Poisson's Ratio of Commonly Used Construction Materials

	Elastic Modulus		Poisson's Ratio
Material	x10^6 psi	GPa	~
Steel, Grade A36	30	207	0.3
Iron	9.6 to 25	66 to 169	0.26 to 0.31
Aluminum	10 to 10.5	69 to 72.5	0.33
Copper	14 to 22	97 to 150	0.30 to 0.35
Concrete	3 to 6	21 to 42	0.18
Wood, Parallel to Grain	1.6 to 2.0	11 to 13.8	~
Wood, Perpindicular to Grain	0.08 to 0.10	0.55 to 0.69	~

shown that for an elastic material the following relationship exists between Young's modulus of elasticity in shear, and Poisson's ratio:

$$\mu = \frac{E}{2G} - 1$$

where

μ = Poisson's ratio
E = Young's modulus of elasticity
G = modulus of elasticity in shear

In addition to measuring elastic properties by applying a mechanical load and measuring the deformation it is important to note that the elastic properties can describe the natural frequency of vibration. The natural frequency of vibration in an elastic body is proportional to the square root of either the elastic modulus or the shear modulus, depending on the mode of vibration. In addition, the velocity with which a compression wave travels through an elastic body is proportional to the square root of the elastic modulus.

Also it should be noted that the deformation exhibited by many materials depends on numerous factors including the magnitude of the load, the rate at which it is applied, and the elapsed time after the load application that the observation is made. This response is generally known as rheological behavior. While instantaneous effects are referred to as the elastic response, time-dependent deformations are commonly referred to as creep or relaxation.

Elastic Properties of Concrete

Several standard tests exist to determine the mechanical response of concrete. Compressive testing is typically determined in accordance with ASTM Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens (C 39) to determine the peak strength and ASTM Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression (C 469) to determine the static elastic modulus in compression. No standard test currently exists to assess direct tensile strength; however, the flexural strength can be determined using ASTM Standard Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78) and ASTM Standard Test Method for Flexural Strength of Concrete (Using Simple Beam with Center-Point Loading) (C 293). Additionally, ASTM Standard Test Method for Flexural Toughness and First-Crack Strength of Fiber-Reinforced Concrete (Using Beam with Third-Point Loading) (C 1018) is commonly used to test the flexural toughness and first crack strength of fiber-reinforced concrete, though it would also be possible to use this test to determine the elastic modulus.

The elastic modulus is one of the most commonly used parameters to describe a material even though many materials do not exhibit a truly linear stress-strain relationship. Two additional terms are generally used to describe the limits of elastic behavior: (1) proportional limit and (2) elastic limit. The proportional limit is defined in ASTM E 6 as "the greatest stress which a material is capable of sustaining without any deviation from proportionality of stress to strain (Hooke's law)." The elastic limit is "the greatest stress which a material is capable of sustaining without any permanent strain remaining upon complete release of the stress."

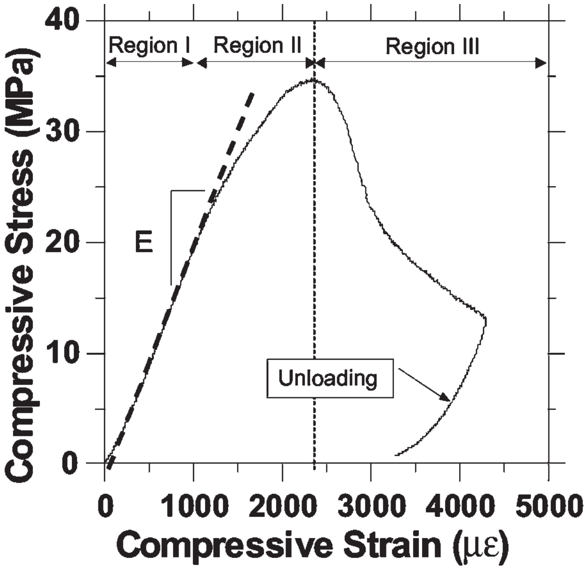


Fig. 2—A stress-strain response of a normal strength concrete in compression.

A typical compressive stress-strain response of concrete is illustrated in Fig. 2 [1]. While a line has been superimposed on the stress-strain diagram for low stress and strain values it quickly becomes apparent that the manner in which its modulus of elasticity is defined is somewhat arbitrary. It can be seen that in Region I of this diagram the relationship between the stress and strain is relatively linear. This can be seen to occur at stress levels below approximately 50 % of the peak strength and at strains lower than approximately 1000 $\mu\varepsilon$. It can be seen that at higher stresses and strains (Region II) the response becomes nonlinear due to the development of cracks between the aggregates and cracks in the paste. After the stress reaches a maximum value, the stress strain curve is observed to begin to soften due to the opening of cracks in Region III [2].

It should be noted that concrete has neither a definite proportional limit nor an elastic limit. As a result, various forms of the modulus are illustrated on the stress-strain curve in Fig. 3. ASTM E 6 defines several different moduli as follows:

1. Initial Tangent Modulus–The slope of the stress-strain curve at the origin.
2. Tangent Modulus–The slope of the stress-strain curve at any specified stress or strain.
3. Secant Modulus–The slope of the secant drawn from the origin to any specified point on the stress-strain curve.
4. Chord Modulus–The slope of the chord drawn between any two specified points on the stress-strain curve.

Modulus of Elasticity in Compression

Since structural concrete is designed principally for compressive stresses, by far the greatest amount of work on the elastic properties of concrete has been done on concrete in compression. The only ASTM test method for the static modulus of elasticity of concrete, ASTM C 469, is a compressive test method. It stipulates a chord modulus between two points on the stress strain curve defined as follows: the lower point corresponds to a strain of 50 millionths (i.e., 50 $\mu\varepsilon$) and the upper point corresponds to a stress equal to 40 % of the strength of concrete at the time of loading. The lower point is near the origin but far enough removed from the origin to be free of possible irregularities in strain readings caused by seating of the testing machine platens and strain measuring devices. The upper

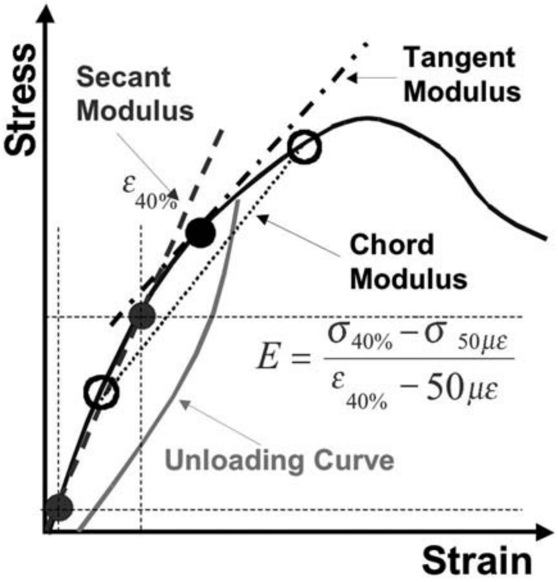


Fig. 3—Various forms of static modulus of elasticity.

point is taken near the upper end of the linear behavior and near the maximum working stress that is assumed in most designs. Thus, the determined modulus is approximately the average modulus of elasticity in compression throughout the working stress range.

The 150 by 300-mm (6 by 12-in.) cylinder is the specimen size, a commonly used specimen geometry for the determination of the modulus of elasticity in compression; however, it should be noted that 100 by 200-mm (4 by 8-in.) cylinders may be common for concretes with smaller aggregates. In order to compensate for the effect of eccentric loading or nonuniform response by the specimen, strains should be measured along the axis of the specimen or along two or more gage lines uniformly spaced around the cylinder. The selection of the gage length is important. It must be large in comparison with the maximum aggregate size so that local strain discontinuities do not unduly influence the results, and it must be large enough to span an adequate sample of the material. It must not, however, encroach on the ends of the specimen. This limitation is established because restraint occurs where the specimen is in contact with the steel platens of the testing machine. As a result the strains near the ends of the specimen may differ somewhat from strains elsewhere in the specimen. ASTM C 469 specifies that the gage length shall be not less than three times the maximum size of aggregate nor more than two thirds the height of the specimen. Half the specimen height is said to be the preferred gage length.

A convenient device for measuring the strains is a compressometer, such as the one illustrated in Fig. 4. The upper yoke is rigidly attached to the specimen, whereas the lower yoke is free to rotate as the specimen shortens. The pivot rod and dial gage are arranged so that twice the average shortening of the specimen is read on the dial. This type of device was used in the first comprehensive investigation of modulus of elasticity by Walker [4], and it is cited in ASTM C 469 as an acceptable device. It should be noted, however, that other procedures may exist [5,6]. Because the test is intended to measure only time-dependent strains, it is important that the specimen be loaded expeditiously and without interruption. For this purpose, an automatic stress-strain recorder is helpful but not essential. Figure 4 illustrates the use of a linear variable differential transformer (LVDT) displacement transducer in which the deformation, instead of being observed on a dial gage, is indicated and recorded.

Although the standard test method is not concerned with the behavior of concrete at stresses above 40 % of the strength, the shape of the stress-strain curve at high stresses is of significance in determining the ultimate load-carrying capacity of a concrete member [7–9]. When tested under load control, concrete cylinders fail suddenly, shortly after the maximum load has been attained.

Several different approaches have been used to assess the complete stress strain response of concrete. Hognestad et al. [9] utilized U-shaped specimens where the central portion of the specimen was loaded eccentrically to back calculate the stress-strain response. Shah et al. [10] tested steel in parallel with a concrete cylinder and subtracted the linear response of the steel to obtain the complete stress-strain response of con-

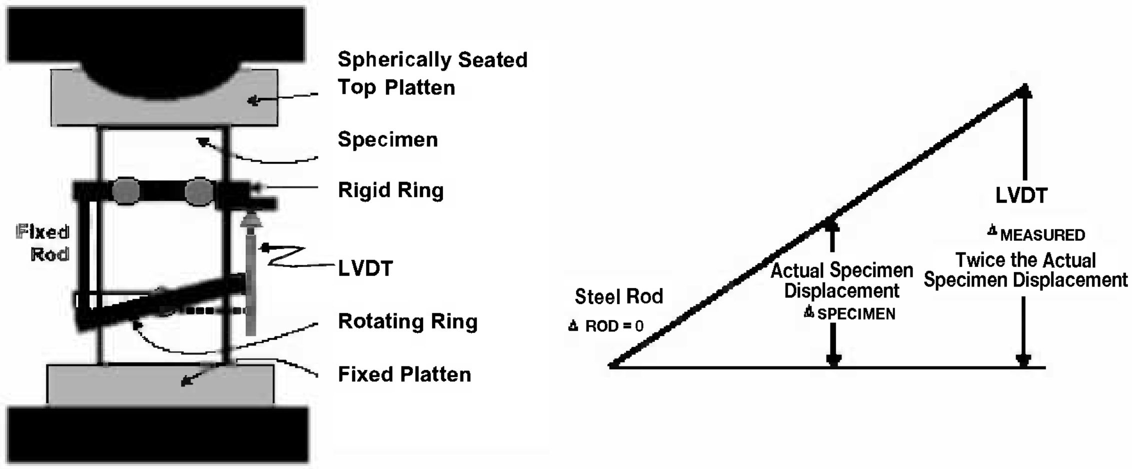


Fig. 4—Compressometer testing details: (a) compressometer testing apparatus, and (b) geometric relation for calculating strain [3].

crete. While the testing of two materials in parallel provides a simple approach for obtaining the stress strain response, it can be subject to errors in the later portions of the post-peak stress-strain response since the large contribution of the steel is being subtracted from the large composite response. To overcome these limitations a closed-loop testing control method was developed in which the expansion of the cylinder in the circumferential deformation was controlled at a constant rate [11]. This approach takes advantage of the fact that as damage develops in the specimen (at high stress levels and in the post-peak region) the circumferential (lateral) deformation increases much more rapidly than the lateral deformation. The circumferential control was used to illustrate the stress-strain curves of concrete of various strengths (Fig. 5) [12]. It can be observed that as the strength of the concrete increases the pre-peak behavior is linear up to a higher level of stress. The higher strength materials show a steeper response in the post peak region that corresponds with a more brittle material. It is important to understand how the end conditions of the cylinder influence the results. For example, capping compounds may demonstrate a disproportionate deformation and friction at the plattens may result in a confining effect. One common method that is used to overcome end confinement or excessive deformation is grinding the specimen ends and using friction reducing substances or brush plattens [13]. Figure 6 can also be used to show the influence of testing specimens of different length [14–16]. In the pre-peak region the stress-strain response is relatively similar irrespective of specimen length. After the peak is reached, however, the larger specimens demonstrate a more brittle response. This occurs since the zone of damage appears to be constant irrespective of specimen size. Newer testing procedures are being developed in which closed-loop testing enables the stress strain diagram to be measured on specimens of different size [15].

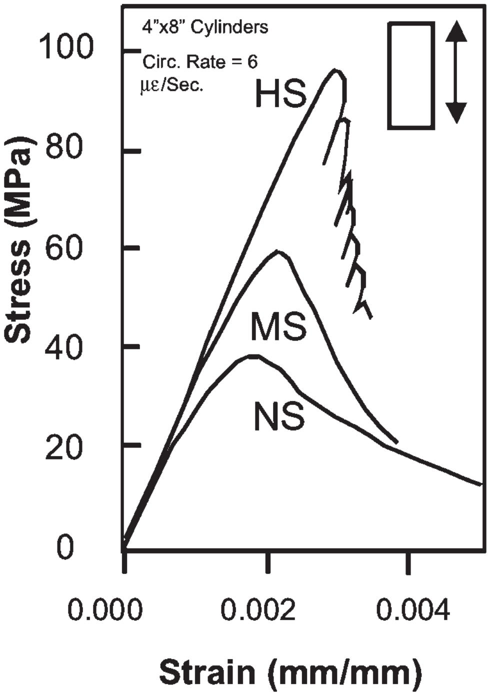


Fig. 5—Typical stress versus axial strain plots for a normal strength, medium strength, and high strength concrete.

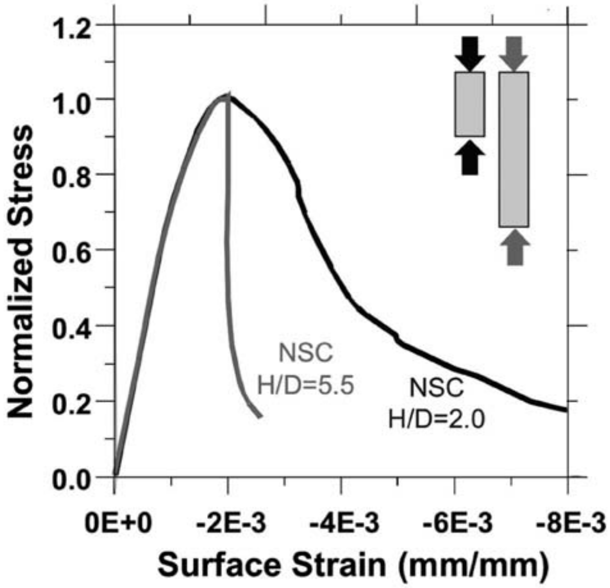


Fig. 6—Influence of specimen size on the measured compressive stress-strain response.

Modulus of Elasticity in Tension and Flexure

Substantially less work has been done to determine the elastic modulus when concrete is tested in tension [17–19]. The test is complicated by the problems associated with gripping the specimens in tension, low strains at failure, and the need to avoid eccentricity. Several different approaches have been advocated over the last three to four decades mainly aimed at developing procedures for improving the stress distributions at the specimen ends for different grips and removing difficulties associated with eccentricity [20,21]. Much of the research has not focused specifically at only assessing the elastic modulus, but rather much of the work has focused on assessing the softening response that occurs when cracking begins to develop [22–24].

Since a principal use of concrete is in flexural members, several investigators have determined the elastic modulus on specimens loaded as beams. An obvious approach is to measure deflections caused by known loads and to calculate the modulus of elasticity from well-known beam deflection formulas. It should be noted, however, that the span-to-depth ratios of concrete beams normally used for such tests are so large that shear deflection comprises a significant part of the total deflection. In applying shear corrections, certain other corrections must be made to take care of discontinuities in the shear deflection curves at load points. For center-point loading, Seewald [25] gives the following deflection formula:

$$\delta = \frac{Pl^3}{48EI}\left[1 + (2.4 + 1.5\mu)\left(\frac{h}{l}\right)^2 - 0.84\left(\frac{h}{l}\right)^3\right]$$

While the deflection for third-point loading can be computed using the following expression from ASTM C 1018.

$$\delta = \frac{23Pl^3}{1296EI}\left[1 + \frac{216h^2(1 + \mu)}{115l^2}\right]$$

where

δ = maximum deflection
P = applied central load
l = distance between supports

E = modulus of elasticity
I = moment of inertia of the section with respect to the centroidal section
μ = Poisson's ratio
h = depth of the beam

The portion of the expression outside the brackets is the simple beam formula without considering the effects of shear. It should be noted that the deflections used in determining the elastic modulus should be carefully measured on the flexural specimens. Researchers have illustrated that substantial errors can occur if deformations are not measured correctly. This is illustrated in Fig. 7 in which the deflection of the machine (i.e., stroke or ram deflection) is compared with the deflection measured directly on the specimen [26]. It can be seen that the measured machine deflection can be much higher than the deflections measured on the specimen. This can be primarily attributed to crushing of the specimen at the load points and deflections of the testing fixtures and machine itself. Deflection is commonly measured at the center point of the beam using a "Japanese Yoke," which is a frame that is attached to the neutral axis of the beams directly over the supports. The frame is designed so that rotations are permitted at one end while rotation and translation is permitted at the other end. The deflection frame provides a reference location which does not move during the test and to which the measurement device is attached so that it can react off of a smaller element that is attached to the beam.

A large number of results have been reported in the literature. The range of results has been from 7 to 40 GPa (1 to 6 × 10^6 psi) [27]. It should be noted that the elastic modulus in tension or flexure does not appear to be substantially different from the elastic modulus in compression at low stress levels.

It is also interesting to note that Olken and Rostasy [28] presented relationships for the development of various mechanical properties (Fig. 8 shows this relationship as a function of the degree of hydration). It can be seen that the elastic modulus develops at a much faster rate than either the tensile or compressive strength. While all of the reasons for the differences in the rate are not completely understood [29], it appears that this may be related to the composite nature of concrete and the fact that the aggregate, interfacial transition zone, and paste influence each of these properties differently. Barde et al. [30] recently demonstrated that differences in flexural strength and elastic modulus development may be due to the influence of aggregate fracturing in strength related properties while this does not occur for modulus measurements due to the low level of stress that is applied.

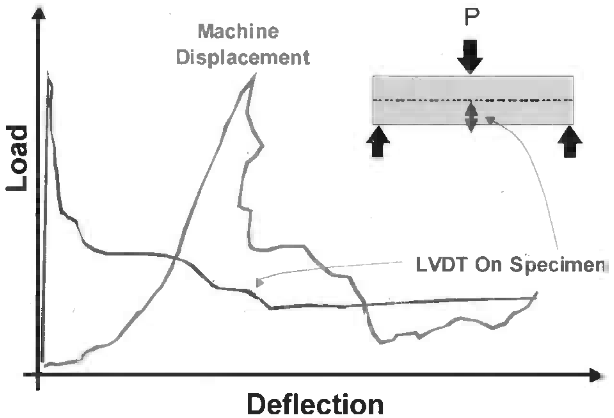


Fig. 7—Conceptual illustration of the importance of measuring deflections on the specimen.

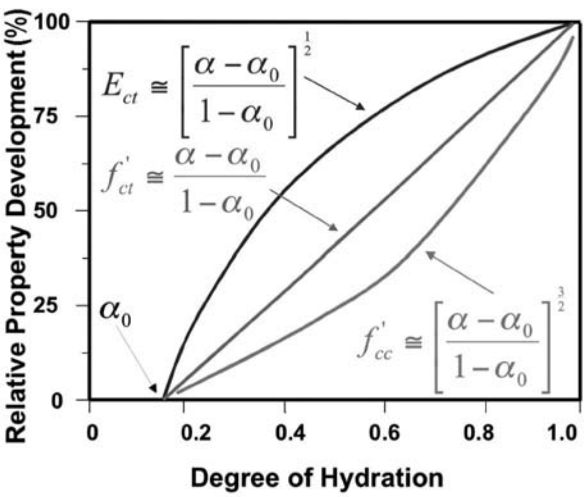


Fig. 8—Rate of material property development as a function of degree of hydration.

Elastic Modulus from Ultrasonic Measurements

In addition to measuring elastic properties by applying a mechanical load, elastic properties can be determined nondestructively. Of all the nondestructive methods, ultrasonic methods offer a distinct advantage in that they can be conducted with a relatively low cost and without causing any new damage. The main premise of the ultransonic pulse velocity test is related to the concept that the velocity with which a compression wave travels through an elastic body is proportional to the square root of the elastic modulus. ASTM Standard Test Method for Pulse Velocity Through Concrete (C 597) can be used for the determination of the compressional wave speeds. A schematic illustration of the testing equipment is illustrated in Fig. 9. This testing procedure relies on the development of compression wave pulses that are generated by exciting a piezo-electric crystal inside the transmitting transducer with a high voltage pulse. The transmitting transducer is held in contact with one end of a specimen (usually a coupling agent is applied between the specimen and the transducer). The second transducer is held on the opposite side and used to record the time that it takes this wave to reach the second transducer. Using information obtained from the test, the wave speed (or pulse velocity) can be determined using the following equation:

$$V = \frac{L}{\Delta t}$$

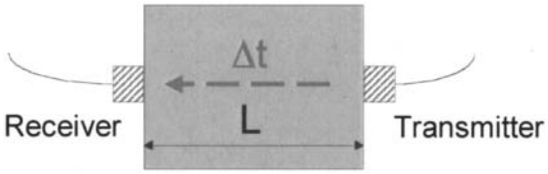


Fig. 9—An illustration of the ultrasonic pulse velocity measurement procedure, ASTM C 597.

where

$V =$ the compressional wave speed (i.e., the pulse velocity)
$L =$ the distance between the transducers
$\Delta t =$ is the transit time (i.e., the time it takes the wave to travel between the transducers)

The elastic modulus can be determined using the velocity of the compression wave through concrete if a value for Poisson's ratio is assumed (typically 0.22–0.24). The elastic modulus can be determined using the pulse velocity from the following expression:

$$E = V^2 \frac{[\rho(1+\mu)(1-2\mu)]}{(1-\mu)}$$

where

$\rho =$ density
$V =$ the pulse velocity
$\mu =$ the dynamic Poisson's ratio
$E =$ the dynamic modulus of elasticity

Typical values of the ultrasonic wave speed can range from 3500 to 5500 m/s depending on the strength of the concrete or age at which the concrete is tested. To improve the accuracy of the results the wave speed of a known material is typically measured to enable the testing apparatus to be properly calibrated before a test. The modulus of elasticity determined from the ultrasonic test (typically referred to as the dynamic modulus) can be up to 25 % higher than the static modulus. This occurs for two reasons. First, the ultrasonic test is conducted at low stress levels and as such the test results more closely resemble an initial tangent modulus (Fig. 3). Second, the elastic modulus is dependent on the rate at which load is applied. Loads applied at a higher rate result in a higher elastic modulus. It should be noted that the elastic modulus in saturated concretes may be 5 % higher than that in dry concrete [31]. Further information on the method for measuring pulse velocity can be found in ASTM C 597 or in the committee report from ACI 228.1R [32]. It should be noted that alternative ASTM test methods can be used for measuring the compressional wave speed (P-wave) in concrete such as ASTM Standard Test Method for Measuring the P-Wave Speed and the Thickness of Concrete Plates Using the Impact-Echo Method (C 1383). Results from this test can provide an alternative method to measure the wave speed which can be used in the preceeding equation to estimate the elastic properties.

An alternative nondestructive test is based on the concept that the natural frequency of vibration of an elastic body is proportional to the square root of either the elastic modulus or the shear modulus, depending on the mode of vibration. ASTM Standard Test Method for Fundamental Transverse, Longitudinal, and Torsional Resonant Frequencies of Concrete Specimens (C 215) was developed for determining the fundamental transverse, longitudinal, or torsional resonant frequencies of concrete specimens. This test is generally conducted either by forcing the specimen into resonant vibration using an electromechanical driving unit or using a small impactor to generate a vibration that is recorded by an accelerometer. This test method is commonly used in the lab for assessing freeze-thaw damage in prisms. Further details on this testing procedure and its use for determining elastic properties are available in ASTM C 215.

Poisson's Ratio

Static determinations of Poisson's ratio are made by adding a third yoke and second dial gage to a compressometer so that a magnified transverse strain may be measured, or by mounting strain gages on the surface of a specimen perpendicular to the direction of loading. The same considerations apply to gage length for lateral strain measurement as for longitudinal strain measurement. Procedures for determination of Poisson's ratio are included in ASTM C 469. Poisson's ratio is also commonly computed from results of the elastic modulus and shear modulus determined dynamically. The static value at stresses below 40 % of the ultimate strength is essentially constant; for most concretes the values fall between 0.15 and 0.20. The dynamic values are usually in the vicinity of 0.20–0.25.

It should be noted, however, that at high stresses or under conditions of rapidly alternating loads, the measured value of Poisson's ratio can change dramatically. When the applied stress is below 50 % of the peak strength there is a decrease in volume of the body as a compressive load is applied. However, at higher loads cracking develops which results in an increase in the volume of concrete and an increase in Poisons ratio [33].

Property Specification and Estimation of Elastic Properties

Frequently designers do not specify elastic modulus but rather they rely on approximations using other properties (namely compressive strength) to estimate these properties for their design. It should be noted however that in some structures where deflections need to be minimized aggregates may be restricted to those that can economically produce low elastic deformation and low creep. Other structures may specify the use composite sections (of concrete and steel or concrete and fiber reinforced composites) to increase the stiffness of the overall structure.

Although code specifications are primarily associated with concrete strength, information on the elastic modulus is required for many aspects of civil engineering design. The ACI Building Code [34] permits the modulus of elasticity to be taken for normal weight concrete as

$$E = 57\,000\sqrt{f'_c}\ (\text{in psi}) = 473\sqrt{f'_c}\ (\text{in MPa})$$

where

$E =$ modulus of elasticity
$f'_c =$ specified comprehensive strength

For concrete with a hardened unit weight between 90 and 155 lb/ft^3 (1440 to 2885 kg/m^3) the modulus can be taken as

$$E = 33w_c^{1.5}\sqrt{f'_c}\ (\text{in psi and lb/ft}^3)$$
$$= 0.043w_c^{1.5}\sqrt{f'_c}\ (\text{in MPa and kg/m}^3)$$

where

$E =$ modulus of elasticity;
$w_c =$ unit weight; and
$f'_c =$ specified comprehensive strength.

It should be noted that while this equation is useful, the elastic modulus is highly sensitive to the modulus of the aggregate and as such the measured modulus may be expected to vary by approximately ±20 % of the computed value. It has been also been noted by ACI 363 that the aforementioned expressions overestimate the elastic modulus for higher strength concretes. As such, it has been suggested that the following empirical relationship can be used for concretes with compressive strengths between 3000 psi (21 MPa) and 12 000 psi (83 MPa) [35]:

$$E = 23w_c^{1.5}\sqrt{f_c'} + 1\times10^6\left(\frac{w_c^{1.5}}{145}\right)\text{(in psi and lb/ft}^3\text{)}$$

$$E = 0.030w_c^{1.5}\sqrt{f_c'} + 6895\left(\frac{w_c^{1.5}}{2325}\right)\text{(in MPa and kg/m}^3\text{)}$$

where

E = modulus of elasticity
w_c = unit weight
f_c' = specified comprehensive strength

Poisson's ratio is also commonly assumed to be 0.18–0.20 for static measurements while values are usually assumed to be 0.20–0.22 for dynamic measurements or rapid loading conditions.

Rheologic Properties: Creep and Relaxation

Creep is defined in ASTM E 6 as "the time-dependent increase in strain in a solid resulting from force." Nearly all materials undergo creep under some conditions of loading. Unlike other materials, however, the creep of concrete is unique since it is observed under normal service conditions at all stress levels. Furthermore, creep of concrete is approximately a linear function of stress up to 50 % of its strength (on mature concrete in compression) and it appears to increase at higher stresses presumably due to the cumulative effects of creep and microcracking.

The creep of concrete appears to have been first described in the United States in 1907 by Hatt [36]. Since that time over 1000 papers have been written on various aspects of creep. It should be noted, however, that the interest in creep has been high at various times over the last century due to various applications. In the 1930s the rise in dam construction was driving research in creep, and this gave way over the next 20 years to an improved understanding of creep for prestressed beams, plates and shells, and long-span structures. In the 1970s interest in creep once again increased due to applications in nuclear structures. In the 1980s concerns with creep in higher strength concretes emerged since these materials were being used in offshore oil structures and high-rise buildings. In the 1990s interest in creep once again increased due to problems associated with early age cracking and the desire to understand how stress relaxation influences the behavior of concrete at very early ages.

First it should be noted that creep is a property of the paste. The cement paste exhibits creep due to its porous structure with a large internal surface area (nearly 500 m^2/cm^3) that is sensitive to water movements. It appears reasonable to conclude that the movement of water in the paste structure is responsible in large part for creep in concrete elements. In fact, Mullen and Dolch [37] found no creep when pastes were oven dried. The fact that creep is associated primarily with the cement paste adds a particular complexity to the problem of trying to describe creep that does not appear in many other materials. This complexity is called aging which generally refers to the fact that cement pastes continue hydration which means that the pore structure and elastic properties are changing over time or with age. This frequently results in problems, however, when information is desired at early ages since the cement is hydrating relatively rapidly. Although creep is a paste property it is important to note that this does not mean that the aggregates play no role. On the contrary, aggregates (especially stiff aggregates) substantially reduce the creep of a material. In some structures, where deflections need to be minimized, aggregates may be restricted to those that can economically produce low elastic deformation and low creep.

At this point it should also be noted that the names applied to the rheological response (often, as done in this document, creep is generally used to describe all aspects of the rheological response) of concrete are frequently less than precise. Strictly speaking, creep describes the deformation that may occur under a constant stress. Creep is illustrated in Fig. 10a. It can be seen that initially the specimen is unloaded (a). At some time (t_o) the specimen is loaded with a stress (σ_o) and the specimen exhibits an initial elastic deformation. Over time this - deformation increases (c) due to the effect of creep. If the load

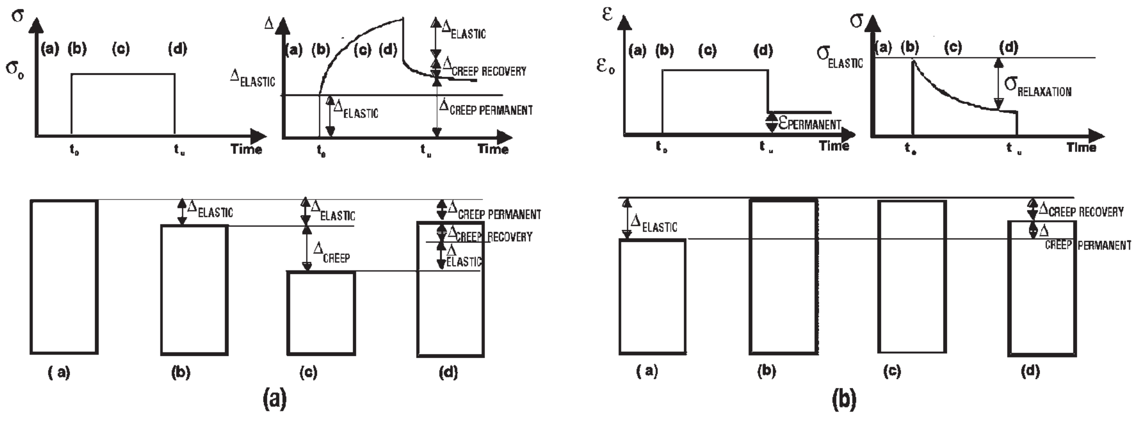


Fig. 10—A conceptual illustration: a) creep, and b) relaxation.

is removed at some time (d) the specimen unloads elastically and continues to unload over some time (creep recovery) though it should be noted that only two thirds of the original creep deformation is recovered. On the other hand stress relaxation describes the reduction in stress that occurs when a specimen is deformed and this deformation is maintained constant. While stress relaxation is related to creep because both occur due to the movement of water in the microstructure under an applied stress, stress relaxation is illustrated in Fig. 10b. An initial specimen can be considered (a) that is deformed elastically at time (t_o) to a strain of (ε_o). This deformation causes the specimen to develop an initial elastic stress (b); however, over time if this deformation is maintained constant the stress will decrease (c) due to relaxation. If the applied deformation is then released (d) the stress in the specimen will be reduced to zero while some permanent deformation may remain in the material.

Creep and relaxation properties are not frequently defined in specifications. Rather designers often use very approximate calculation procedures or apply larger safety factors to account for them. The reason for the approximate nature of many of the calculations may be traced to the fact that the composition and size of the cement and composition and size of the aggregate play such a large role in determining the amount of creep that can be expected. Further, these materials vary from location to location making it very difficult to "predict the effects of creep" with a high level of accuracy without having testing information on the local materials. Finally creep tests are frequently labor intensive, require a conditioned space to perform the tests, and take a substantial amount of time to perform.

Measurement of Creep in Compression

The age at which creep tests begin and the stress level to which specimens are loaded are usually dependent on how the data will be used. A test procedure has been standardized in ASTM Test Method for Creep of Concrete in Compression (C 512). The method stipulates loading moist-cured specimens at an age of 28 days to a stress not exceeding 40 % of the strength of the concrete at the time of loading, although provision is made for other storage conditions or other ages of loading. The stress is restricted to the range throughout which creep has been found to be proportional to stress. Limitations on gage lengths similar to those in the test for modulus of elasticity apply. The method is intended to compare the creep potential of various concretes. Testing at a single age of loading is satisfactory for this purpose. It is required in the test method that the stress remain constant throughout the one-year duration of the test within close tolerances. The load may be applied by a controlled hydraulic system or by springs, provided in the latter case the load is measured and adjusted frequently.

ASTM C 512 requires companion unloaded specimens. Length changes of these specimens are measured and subtracted from the length changes of the loaded specimens to determine creep due to load. This correction is intended to eliminate the effects of shrinkage and other autogenous volume change. While this correction is qualitatively correct and yields usable results, most modern theories deny the independence of shrinkage and creep and thus indicate that the two effects are not additive as assumed in the test. It is now common to label creep which occurs in the absence of drying "basic creep" and to label the additional deformation not accounted for by shrinkage "drying creep" [38]. Thus, the total shortening at any time may be considered the sum of elastic strain, basic creep, drying creep, and shrinkage.

Effect of Specimen Size

It has been demonstrated [39,40] that creep of sealed specimens is independent of specimen size. This observation plus the observation concerning mass concrete in the preceding paragraph indicate that the techniques and specimens of ASTM C 512 are applicable to all types of concrete sealed to prevent loss of moisture. For unsealed specimens exposed to a drying atmosphere, it is evident that there must be a size effect associated with the moisture gradients within the specimen.

It should be noted that the creep of a structure may be only a fraction of that in a test specimen. Hansen and Mattock [41], in an investigation of both size and shape of specimens, found that shrinkage and creep were dependent only on the ratio of surface to volume. Information of this sort may make it possible to apply correction factors to the data obtained from ASTM C 512 to determine the creep in any size and shape of structure.

Measurement of Tensile Creep or Relaxation

Early-age cracking sensitivity of concrete recently has been a topic receiving much attention. Toward this end early-age creep and relaxation properties have been heavily investigated. Although no standardized testing procedures have been developed, the testing procedures fall into a few distinct categories. The first category of tests consists of a uniaxial tensile creep test that relies on the application of load through a dead weight. Umehara et al. [42] and Bissonette and Pigeon [43] have conducted uniaxial tensile creep tests where a specimen is loaded using a dead-weight that is attached to a lever arm. The second category of tests consists of a pressurized cylindrical specimen that applies a constant pressure on the inner surface of a hollow cylinder [3,44]. The third category of tests consists of using an electric or hydraulic mechanical testing device to apply either a constant load or constant displacement to a single specimen or series of specimens [45–47]. The final category of testing devices consists of horizontal testing frames that use a closed-loop control to rapidly adjust the force on a specimen to maintain a specified displacement. The closed loop test provides the total stress history of a specimen and is quite useful [48–53]. It should be noted that in each of these tests the entire testing frame or specimen is generally placed in a controlled environment.

Property Specification and Estimation of Creep and Relaxation Parameters

As previously mentioned, the time under loading influences the corresponding deformation of the concrete. The ratio of long-term strain to immediate strain can be as high as 3.0. The amount of creep exhibited is generally proportional to the stress level (at least to 50 % of the peak strength), to the age at loading with materials loaded at an earlier age showing more creep, to the duration of loading with more creep in materials under a longer duration of loading, and to the strength of the material being tested with higher strength materials showing less creep.

A simple method for computing the effects of creep at various times under loading was defined by ACI-209R-92 using a creep coefficient. This creep coefficient (C_{CU}) can be thought of as simply the ratio of the long-term (ultimate) strain, which includes both elastic and creep effects (ε_{CU}), to the initial elastic strain (ε_{CI}):

$$C_{CT}(t) = \frac{t^{0.6}}{d + t^{0.6}} C_{CU}$$

where

C_{CT} = coefficient at any time
t = time in days
d = is a constant (typically assumed to be 10) in days
C_{CU} = is the long-term on creep coefficient (typically assumed to be 2.35)

Typical values for C_{CU} range from 1.3 to 4.15 but the recommended value is 2.35. It should be noted that ACI-209 provides an approach to correct C_{CU} to account for moist or steam curing, duration of moist curing, relative humidity, member size, and surface to volume ratio.

Over the last three decades several models have been proposed to overcome the shortcomings of the creep coefficient approach to predict the response of concrete under sustained loads. These models are commonly referred to as the Gardner-Lockman Model (GL2000) [54], the Bazant Models (BP or B3) [55,56], or the CEB model (CEB-90) [57]. It should be noted that there is no universally accepted model for creep and even the most accurate models are commonly believed to be accurate to only approximately ±35 %. Due to space limitations the reader is referred to the original documents or summary documents for further information on predictive creep models and their application [58–60].

Significance and Use of Elastic Properties, Creep, and Relaxation

Deflection of Compressive and Flexural Members

Concrete members undergo deflection upon application of load and continue to deflect with the passage of time. This may be of interest for reinforced beams, girders, slabs, or columns. It is not uncommon for a reinforced concrete flexural member eventually to reach a deflection three times as great as its initial deflection, while a precise prediction of these deflections is possible only if the elastic and creep properties are known. Relatively little creep testing is directed to predicting deflections of specific structures; however, predictive equations and approximations are frequently used.

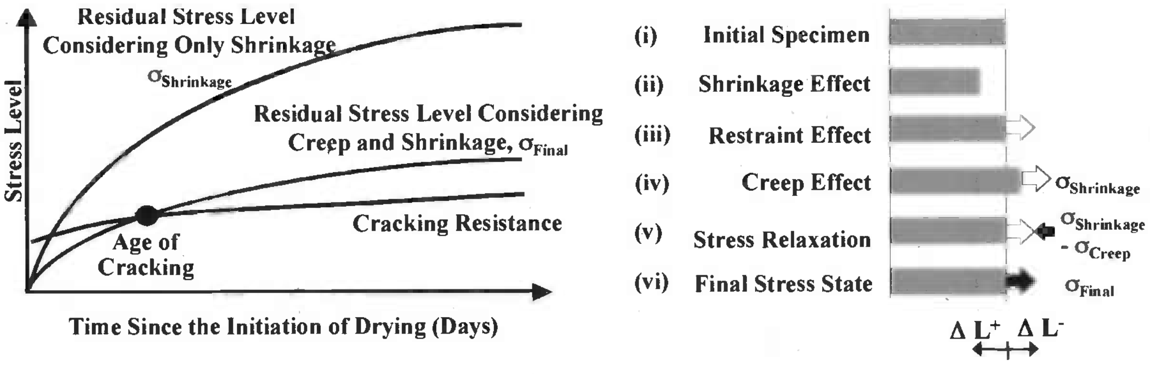
Loss of Prestressing Force

In contrast to the lack of precision needed for deflection measurements, an accurate knowledge of the early-age rheological properties of concrete is valuable to the prestressed concrete industry. After the prestressing force is applied, there is a loss of prestressing force resulting from creep (and shrinkage) of the concrete and relaxation of the steel. Since the initial prestressing force is limited by the strength of the steel and the load-carrying capacity of the member is limited by the residual prestressing force, a knowledge of the factors governing loss of prestressing force has important economic implications.

Residual Stress Calculations

Recently it has become increasingly common to see concrete structures developing cracks due to thermal, drying, or autogenous shrinkage. Although numerous factors influence whether a concrete will crack [61], it can be simply stated that cracking will occur if the residual stresses that develop exceed the tensile strength of the material. Figure 11a illustrates how one can compare the time dependent strength development with the time dependent residual stresses that develop [62]. As a first point of analysis, it can be argued that if the residual stress development exceeds the strength of the specimen the concrete can be expected to crack. This is illustrated in Fig. 11a as the point at which these two lines intersect. Similarly, it follows that if the strength of the concrete is always greater than the developed stresses, no cracking will occur.

The residual stress that develops in concrete as a result of restraint may sometimes be difficult to quantify. Shrinkage strains can be converted to stresses with knowledge of the elastic and creep (relaxation) behavior of concrete. This residual stress cannot be computed directly by multiplying the free shrinkage by the elastic modulus (i.e., Hooke's Law) since stress relaxation (creep) can substantially reduce the stress by 30–70 %. This reduction can be described by Fig. 11b in which a specimen of original length (i) is exposed to shrinkage. If the specimen were unrestrained, the applied shrinkage would cause the specimen to undergo a change in length (shrinkage) of ΔL+ (ii). To maintain the condition of perfect restraint (i.e.,



(a) (b)

Fig. 11—An illustration of the restrained shrinkage cracking problem: a) residual stress development, and b) a schematic description of stress development.

no length change) a fictitious load can be envisioned to be applied (iii). However, it should be noted that if the specimen were free to displace under this fictitious loading the length of the specimen would increase (due to creep) by an amount ΔL-(iv). Again, to maintain perfect restraint (i.e., no length change) an opposing fictitious stress is applied (v) resulting in an overall reduction in shrinkage stress (vi). This illustrates that creep (relaxation) can play a very significant role in determining the magnitude of stresses that develop at early ages. As a result substantial research has been conducted over the last decade to better determine how stresses develop at early ages. For further information on early age stress development the reader is referred to RILEM TC-181 [63]. It should also be noted that a simple test method was recently added as an ASTM standard (C 1581) to assess the behavior of restrained concrete elements. This test consists of casting an annulus of concrete around a steel ring. As the concrete dries (or experiences autogenous shrinkage) it attempts to shrink; however, this shrinkage is restricted by the restraining steel. This places the concrete in tension and compresses the steel. While the test is primarily used to determine if sufficient tensile stresses develop in the concrete ring to cause cracking, additional research has described how this test method could be used to determine the residual stresses and effect of creep [64–66].

Ongoing Efforts and Future Needs

Interest in more accurately assessing rheological properties of hardened concrete is increasing due to concerns related to early age cracking, prestressed concrete, behavior of thin elements, and behavior of high-strength low-water-to-cement ratio concretes. The following list provides an outline of ongoing efforts in the area of rheological properties as well as future needs:

- Standardized testing practices exist for determining the elastic modulus; however, standardized tests do not exist for determining the complete stress-strain response for concrete. Other organizations have assembled substantial data for assessing the stress-strain response of concrete. In the absence of ASTM standards on the subject, it is suggested that readers review the practices advocated by the RILEM SSC (strain softening in concrete) committee [67].
- Significant advancements are taking place in attempting to describe the behavior of concrete at early ages. This frequently includes computation of deformations and stresses which require elastic properties at early ages when they may be changing dramatically due to hydration. Further research is needed to define how elastic properties develop over time (i.e., different maturities). This includes both rate of development as well as "time-zero" and it is suggested that research and standardized procedures be developed to define how these properties should be defined [68,69]. Further, it is suggested that engineers begin to discuss how calorimetric measurements of property development can be compared to ultrasonic, electrical, and mechanical measurements which may measure fundamentally different aspects of the system.
- Interesting work is being performed in the area of the increased use of nondestructive testing procedures to assess the elastic properties of concrete, especially the age-dependent elastic properties. Research linking ultrasonic, acoustic, and electrical measurements with rheological properties should be further developed and validated over a wide range of material properties.
- Standardized practices are needed for conducting early age creep and relaxation tests. As these tests are developed and standardized it is recommended that experimentalists consider recommendations of model developers that will enable this data to be utilized in future model developments [70].
- It currently appears that predictive models may be needed that are capable of meeting the needs of two distinct audiences. The first audience desires a model that includes a description of material behavior from first scientific principles. It would be anticipated that these models would be incorporated by the computer modeling community since complicated calculations can be performed in the models. The second audience for these models are users that may want to be able to perform approximate calculations very quickly using hand calculations.
- It is recommended that future standard test procedures consider the addition of an appendix that would define standard reporting procedures for reporting data from the test and the development of a data bank for elastic modulus, creep, early-age creep, and relaxation tests that is similar to that RILEM Shrinkage and Compliance Data Bank.

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20

Petrographic Examination

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Preface

ALMOST 50 YEARS HAVE PASSED SINCE THE PUBLICAtion of *ASTM STP 169A,* in which this chapter was first published. Katherine Mather, who wrote the first two versions of this chapter in *ASTM STP 169A* and *169B,* quoted, in her closing, from St. Paul, "Things which are seen"–concrete and mortar–"were not made of things which do appear." But then, St. Paul was not a petrographer. St. Paul is not with us–neither is Katherine, who died in 1991. Much of this paper is still her "quote" on petrography, but with changes and additions that update the subject.

Introduction

Almost 50 years have passed since the inception of *ASTM STP 169A*, and 27 years have passed since the updated *ASTM STP 169B* was published. During these times, there was little reference material available on petrography of concrete. Today, there are literature references to petrographic examinations available in which the focus is on petrography as a tool used to provide information about concrete instead of petrography that is incidental to the main subject of a paper. Both approaches, however, are needed because doing petrography solely for the sake of providing information can be an exercise in futility, while doing petrography for the purpose of providing links connecting "activity" within concrete to its behavior and performance is indeed a fulfillment of the science.

Significant advances in petrography and petrographic methods have been published related to concrete. Once considered by many to principally revolve around light optical microscopes, the science has greatly expanded to include new types of instrumentation and techniques that cover subjects from light optical microscopy to specimen preparation, "wet" chemical analyses to infrared spectroscopy, X-ray diffractometry and spectroscopy, scanning electron microscopy with attendant elemental analysis, differential scanning calorimetry, and other analytical tools.

In the history of petrography of concrete as we know it today, at least from published papers, was the work of Johnson who described and related microscopical observations of the composition and texture of deteriorated concrete to its performance. He further applied what he saw toward a philosophy of what makes inferior concrete inferior, and said, "even with the very best of materials, only concrete of inferior strength is commonly produced." There were trailblazers before Johnson, but his work was, perhaps, the first widespread enough so that it truly reached out to engineers. His work was published in a six-part series from January through March 1915 [1].

Subsequent and more recent informative documents include Refs 2–15. The International Cement Microscopy Association (ICMA) was founded in 1978. That organization, whose main emphasis originally was on portland cement, has expanded and now includes work on many aspects of concrete. It also provides publications of interest to concrete petrographers and others. About three years ago the Society of Concrete Petrographers (SCP) was founded and now provides a home base to concrete petrographers.

In June 1989, the first symposium specifically directed toward presenting information on petrography of concrete and concrete aggregates was sponsored by ASTM. The papers presented at that symposium resulted in *ASTM STP 1061* [16]. In 1999, ACI sponsored a symposium on determining the water-cement ratio and durability aspects of concrete [17] that included a number of papers on petrographic methods. A book devoted entirely to details of concrete petrography and correlations to field concrete performance, and aptly titled *Concrete Petrography* [18], was published in 1998. ASTM Practice for Petrographic Examination of Hardened Concrete (C 856) has been modified throughout the years and now provides for more stringent qualification requirements for petrographers doing petrographic examinations and more detailed information on a number of aspects of petrographic examinations. The scanning electron microscope (SEM) and its ancillary energy dispersive spectroscopical (EDS) method for elemental analysis is used so frequently for petrographic work that ASTM Committee C09.65 on petrography has been working to standardize that approach.

The closing remarks of Katherine Mather, who authored the initial versions on petrographic examination of concrete in *ASTM STP 169A* and *ASTM STP 169B*, foretold the potential advances in the science of petrography. Today, the imagination extends even further, and it is gratifying that her visions are in use and being extended.

Petrography

Within the realm of petrography, which includes the use of a broad variety of analytical and physical methods, is the scientific description of the composition and texture of materials, including the systematic classification of rocks. It also in-

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[1] Petrographer, The Erlin Company, Latrobe, PA 15650.

cludes almost anything that can be said about a concrete, from its mineralogy to its strength and volume stability.

Petrographic analysis of concrete–a man-made rock–is its examination by analytical techniques that will identify procedures and the sequence of its production, its composition and internal structure, and allow its classification as to type, original and existing conditions, and future serviceability.

The physical and chemical properties of concrete, especially immediately and shortly after it is made, are a physical wonder. Within a very short time, measurable in terms of days, it becomes hard and strong and usually endures for long periods. Its strength originates within itself by complex chemical reactions. It is re-created "rock" akin to the rock conglomerate that Mother Nature has made.

Like rocks and minerals, concrete is a "mirror" with a memory. Petrographic examinations allow us to interpret the concrete's past as it really was–to identify, beyond all of the obscurities, essential facts about its manufacture and performance. Its makeup and past performance, in light of research and practical experiences, allow projections of its future serviceability.

Petrography is used frequently to assist in forensic evaluations, where it is vital for supplying factual information. That information can relate to mixture proportions that include coarse and fine aggregates, portland cement and other cementitious materials, water-cement ratios, air contents, mixing, placing, bleeding, cement hydration, finishing, curing, cracking, scaling, spalling, low strength, excessive wear, blistering, delamination, adverse chemical and physical reactions resulting from internal and external sources, and other features. To be effective, the petrographer must have a good understanding of each concrete-making material, concrete manufacture, and the influence of environmental exposure on its stability and performance.

This chapter provides insight into the use of petrographic examinations for evaluating hardened concrete, noting problems inherent in its applications, outlining what it involves, describing information it can produce, and showing how this information can be applied.

General questions that petrography tries to answer are: (a) does the concrete conform to specification requirements? (b) how will it behave in use? (c) why did it behave the way it did? and (d) what can be anticipated in the future? The most useful method for developing practical information from which to answer these questions is to study the concrete in the laboratory and correlate what is found to its field performance.

Petrographic examination of hardened concrete is among the subjects in this volume because it provides direct observational information on what is in concrete and provides another way of establishing the relationship of concrete composition to concrete performance.

Responsibilities

A petrographic examination usually begins with discussions between someone who requests the examination and a petrographer. Usually the discussions include a summary of the situation that prompted the need for the examinations. The person requesting the examinations may not be familiar with techniques petrographers use or petrographic approaches available. His reliance is on the background and experience of the petrographer for obtaining the information necessary to answer the question that prompted his need for the petrographic study.

The petrographer should not expect his petrographic results to be taken on faith. Standard procedures should usually be used, or modifications of standard procedures, as may be appropriate–the rationality of the techniques that are used should be demonstrable. Useful background information that may help the petrographer direct his work and evaluate the petrographic data may be from plans, specifications, materials tests, inspectors, contractors, concrete producers, suppliers of the concrete-making components, engineers, architects, and most anyone involved in the concrete. The petrographer should examine concrete with all available means commensurate with the needs and economic restrictions imposed by his client.

In a recent article, Paster [19] comments that the water-cement ratio (w/c) cannot be determined using petrographic methods. However, in ACI SP 191 [17] and in dozens of other papers, a number of methods for doing so are discussed. That comment brings up the competence of the now relatively large number of concrete petrographers and their limitations in examining and interpreting what they find in concrete and their comfort level in estimating water-cement ratios. That comfort level does indeed vary between petrographers, depending upon their education, experience, and training. Because one petrographer has a low comfort level of evaluation does not mean all petrographers are at that same low level, and it does not speak highly of anyone that apparently does not have background information upon which to make that kind of statement. A petrographer's responsibilities include not only petrographic examinations, reports of the petrographic data, and interpretations of the data, but also data that are qualified. ASTM C 856 establishes minimum requirements for concrete petrographers. Recently, The Society of Concrete Petrographers[2] was formed as a means of getting together hands-on concrete petrographers. The Society recognizes the weaknesses of some petrographers in areas of their work–one of the goals of the Society is to provide a meeting ground for petrographers to discuss issues of mutual interest so that they can better perform and interpret their work.

Methods—Standardization and Description

ASTM C 856 was adopted in 1977. It turned out to be more complicated than was expected because there are different purposes for examining concrete. The concrete also may represent exposures to different environments for different periods of time, specimens from laboratory test programs, and samples from newly cast laboratory and field structures. ASTM C 856 does the following: (1) serves those who request a petrographic examination and want more understanding of what they are getting and why; (2) provides information to petrographers conducting petrographic examinations; and (3) serves as a reminder to petrographers of things they may have forgotten or neglected.

ASTM Guide for Petrographic Examination of Aggregates for Concrete (C 295) was first published in 1952. ASTM Practice for Examination and Sampling of Hardened Concrete in Constructions (C 823) was adopted in 1975. It gives guidance on steps to be taken before laboratory examinations, and pre-

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[2] Derek Cong, Secretary (dcong@wje.com); Bernard Erlin, President (be5023@aol.com).

liminary investigations are outlined and include: (1) the desirability of assembling reports and legal documents concerning the construction, and (2) the usefulness of interviews with contractors and others connected with the construction and with the owners, occupants, and users of the construction. Procedures for detailed investigations of the concrete in-place are described. Sampling hardened concrete is discussed along with the preparation of appropriate sampling plans and selection of the number and size of samples. Information needed to accompany samples is described. ASTM C 823 was prepared to be useful to petrographers, engineers, and others who have reasons for a need to examine concrete.

ASTM Quantitative Determination of Phases in Portland Cement Clinker by Microscopical Point-Count Procedure (C 1356) provides a standard systematic procedure used to identify the volumetric properties of portland cement clinker phases. It also provides information that assists in an evaluation and interpretation of the manufacturing history of clinker. In 1976, Transportation Research Circular No. 176 [20] was published. It provides help in (1) identifying alite and belite residues in paste, and (2) examining paste by X-ray diffraction, differential thermal analysis, and infrared absorption spectroscopy. Another publication for help in petrographic examination of hardened concrete is Highway Research Board Special Report No. 127 [21]. Other publications of interest are in volumes of the Fifth International Symposium on the Chemistry of Cement [22] and those of the Sixth International Symposium on the Chemistry of Cement [23]. A stimulating publication originating in 1971 [24] covers a wide field of cement and concrete topics and has the advantage of being truly international because the editorial board and contributing authors come from various parts of the world where cement and concrete research is carried out.

Today, sophisticated analytical instrumentation is bringing about a breakthrough by orders of magnitude in our ability to decipher and interpret (and sometimes misinterpret) the composition and performance of concrete. These methods can bring with them the danger of losing touch with the primary purposes of an examination due to great differences in scale. Those differences include concrete elements with dimensions in hundreds of metres (kilometres in the case of pavements); core samples with dimensions in hundreds of millimetres that can be examined with great advantage using low-power stereomicroscopes; thin sections about 800 mm^2 by 15- to 30-μm thick and immersion preparations even smaller examined using petrographic microscopes; X-ray diffraction samples that may be milligrams of material hand-picked under the stereomicroscope or a few grams of material concentrated by handpicking paste or aggregate from carefully broken concrete surfaces; scanning electron microscope specimens that may be 200 mm^3 down to micro-picked specimens—and ancillary elemental analysis using EDX methods of material volumes equivalent to one-trillionth of a cubic inch (1.64×10^{-11} cm^3); single crystals of a few angstroms examined using microprobes; and nanometer-sized material examined using transmission electron microscopes. However, few cases will be found in which all of these will be needed.

The sequence of steps involved in a petrographic examination can include: (1) observing the concrete construction; (2) evaluating samples using the naked eye and low-power stereomicroscopy; followed by (3) the selection of microscopical methods that allow greater insight into the makeup of the concrete; and (4) the selection of additional methods that will provide ancillary information needed to properly evaluate the problem that prompted the studies. The transition from the macro to the micro scale and the information from each unit of effort provides separate and complimentary data that can be used in final analyses and interpretations of the data.

Care is needed to ensure that proper sampling is done for each part of the study. Field samples may include examples of different outwardly appearing concretes, with cracks, scales, spalls, efflorescence, and anything else that may have contributed to or be part of a problem. Specimens for the different analyses should be taken to represent inherent parts of the concrete needed to understand its original makeup and subsequent physical or chemical alteration, or items that may have contributed to its current condition. The number of samples needed is dependent upon the specific problem—and should be representative of the various features of the concrete. The sampling should include unaltered concrete for comparison.

Sorting out major from minor causes or secondary effects of deterioration requires knowledge of many aspects of concrete, from individual concrete components to its manufacture to the things that affect cement hydration, and effects of the chemical and physical environment on concrete performance.

Analytical techniques offer a means for understanding internal chemical reactions in detail so that it is possible to characterize cement hydration and hydration products. Thus, the chemical reactions of normal hydration or of abnormal deterioration and alteration to concrete can be detected with reasonable clarity so that detailed information is available to decipher a concrete's history. The relative roles of chemical attack and physical attack that dictate concrete performance are usually decipherable. The basic microscopical techniques used for petrographic examinations of hardened concrete can be augmented, but not replaced, by other techniques.

Purpose and Approach

Purpose

The purpose of a petrographic examination is initially to understand the original concrete makeup, which includes: (1) aggregates—their grading, composition, and distribution, and any physical or chemical alteration; (2) portland cement, supplementary cementing materials, pozzolans, and their hydration and other reactions; (3) air-void systems including the size and distribution of air voids; and (4) textural characteristics resulting from its placement, consolidation, and finishing. Once that understanding is in hand, the appropriateness of each item and alteration to the original concrete can be identified. The question of why the concrete malperformed can then be identified, if suitable, and appropriate analytical techniques can be used.

Approach

The concrete problem must usually be defined in order to provide direction for the petrographic examinations. Sometimes the problem is purposely not defined because of a desire to not prejudice the petrographer in his examination and interpretation of the analytical data or to test his skillfulness. The latter is unneeded because his prowess ultimately becomes known when his report is finalized. A good petrographic examination is the one that provides enough information to resolve the problem. A better petrographic examination is one that resolves the problem with maximum economy in minimum time. In both cases, the end result is what is important. The good, or better examination, varies depending on the problem and particularly the skill and adeptness of the petrographer. The petrographer's skills include deftness in obtaining data and in interpreting the data.

Texture and Composition

Concrete is a very heterogeneous material because it is made up of a variety of coarse and fine materials in terms of its basic components, coarse and fine aggregates, paste, and air. The processes of batching, mixing, transportation, discharging, placing, consolidating, the differential settlement of components (among the results is bleeding), finishing, and curing are independent factors that control its final makeup and texture. Texture is the way in which concrete components are finally allied and aligned. On a macro scale, honeycombing, lift lines, and stratification of components are examples of textural features that are usually undesirable. What is most desirable is a uniform distribution of concrete components. On a micro scale, texture can be reflected by the uniformity of distribution of, for example, calcium hydroxide, a main hydration component of cement hydration that can be uniformly or nonuniformly distributed throughout the paste, at aggregate-paste interfaces, or preferably located along the bottom side of aggregate particles.

Petrographic examinations are useful for identifying textural and compositional characteristics, cracks, and crack patterns. Some examples are: low strength resulting because the physical structure (texture) is inadequate can be caused by poor mixing or inadequate consolidation; cement hydration may have been curtailed because of a number of factors; contaminants may be present; air-void systems may be inadequate to provide resistance against damage by cyclic freezing; poor finishing may have created incipient delaminations in the surface region; there may have been undesirable chemical reactions of the aggregates; and aggressive chemicals may have been introduced from the environment.

Each concrete and each part of a concrete is unique in terms of composition, texture, history, and exposure. Deciphering the texture and composition of concrete is where petrography shines.

Observations

Petrography is the science of observation, whether on a macro or micro scale. In a sense, everyone is a petrographer on a macro scale. The petrographic effort becomes valuable on a micro scale, which requires patience, skill, and knowledge—not only about microscopes, as most people recognize, but also about ancillary techniques to which attention is directed based upon microscopical evaluations.

The cupboard of a petrographic laboratory, from an instrumentation viewpoint, must include: (1) a series of light-optical instruments that includes a low-power stereomicroscope, petrographic microscope, and may also include a metallurgical microscope; (2) specimen preparation equipment that includes saws, grinding and lapping equipment; and (3) accessory equipment and supplies needed to make each of the preceding effective (see ASTM C 856). Supplementary equipment may include a scanning electron microscope and an ancillary elemental probe. A variety of analytical instrumental methods that include X-ray diffractometry, differential scanning calorimetry, differential thermal analysis, infrared spectroscopy, a variety of instruments for elemental analysis, and wet chemical methods may also be needed. The petrographer should be familiar with specimen preparation, the kind of information each of these can provide, and limitations of their use.

Age of Concrete Under Study

The age of the concrete may be important for judging the significance of petrographic observations. For example, calcium sulfoaluminate (ettringite) found in many voids as far as 130 mm (5 in.) from the outer surfaces of a concrete pavement of unknown age and of high flexural and compressive strength may be of importance relative to projected service. That observation is of particular importance when in other 15-year-old, similarly exposed field concretes from the region calcium sulfoaluminate is not abundant. If the concrete of unknown age is in fact five or seven years old and it differs conspicuously, the difference probably justifies some concern about its future performance; if it is 15 years old, it is peculiar, but the peculiarity is probably of less practical importance.

Recently, Neville [25] reviewed dating the age of cracks using depths of carbonation as signposts. Although relative ages could be identified, specific times of cracking could not unless the cracks occurred very early (e.g., plastic shrinkage cracks).

Sources of Concrete

Knowing the concrete background and its geographic location is sometimes of importance because aggregates, cements, and environmental conditions vary both locally and regionally. For example, regional geology—as it determines quantity and uniformity of aggregates—may be of importance. However, today, with longer-range shipment of concrete-making materials, changes to cement manufacture, use of supplemental cementitious materials, and due to material shortages, there is a broader overlap of different materials so that new problems may arise in areas previously thought immune to some problems. For example, whereas low alkali cements may have once been common to a regional area because of local cement production, increased shipment of higher-alkali cements into an area can change the previous "immunity" of some siliceous aggregates to alkali-silica reactivity, or significantly affect the efficacy of air-entraining and other admixtures.

Reconstruction of History of Field Concrete

To progress from consideration of simple petrographic examination to the petrographic examination of concrete that has aged and perhaps deteriorated in service introduces two important new unknowns—time and the precise environment of the concrete. The effects of the passage of relatively short periods of time on the constituents present in several cement pastes of known w/c stored under laboratory conditions have been investigated. However, anomalies remain in the results, even though compositions of pastes and nature of the environments were known and controlled far more thoroughly than the composition and environment of any field concrete. Today, where there is extensive use of fly ash, silica fume, and ground granulated blast-furnace slag, there are even more anomalies.

Composition

Petrographic examinations provide the most direct and more information about concrete than any other technique because it can be used for identifying its physical and chemical makeup, and its proportions and factors leading to an understanding of its behavior. Things that can be deciphered include: (1) the types of cementitious materials present, the products that have formed as a consequence of their chemical reactions, and estimates of amounts present; (2) aggregates, their rock and mineralogical types, grading, uniformity of distribution, and information about their chemical and physical stability or instability; (3) estimates of water-cementitious materials ratios and their uniformity throughout a concrete; (4) air-void characteristics, whether non-air-entrained or air-entrained, distribution of the voids and parameters of air-void systems; and (4) features related to consolidation, finishing, curing, premature carbonation, surface dusting, aggregate coatings, mineral surface hardeners, depth of carbonation, and cracks.

One of the many items of interest and concern about concrete is the water-cementitious materials ratio. Petrographers have been using a variety of techniques for estimating the w/c and water-cementitious materials ratio (w/cm) of hardened concrete. All of them are subjective and rely upon the petrographer and his or her experience and expertise in interpreting the data, and they are generally accepted by the concrete industry.

There are four general methods among petrographers that are popular today: (1) a water-droplet absorption technique [26]; (2) scratch hardness [26] and indentation hardness [27] techniques; (3) a combination of 12 or so microscopical and physical observations of the paste technique [28,29]; and (4) methods where thin sections of concrete are impregnated with dyed epoxy and are compared to thin reference sections made from concrete having known w/c's. The latter includes two techniques in which the dye is either blue [26] or fluorescent [29]. Sometimes combinations of methods are used.

Unfortunately, there are few specific details in the literature of how to translate what is observed to assessing the w/c using these methods, except for the fluorescence method. That method originated in Europe and has been evolved by a handful of people over which there has been controversy about its claimed accuracy of ±0.02 when estimated using a manual technique, and an accuracy of ±0.01 when estimated using a semi-automatic technique. There is nothing in the literature about the accuracy of the scratch hardness, water droplet absorption, and blue tone methods. The w/c estimate using the combined optical and physical paste properties technique is reported to cover a ±0.02 range.

The degree of interpretation of data from each method is based upon the comfort of the petrographer in extending his or her expertise to provide that estimate. That comfort zone is from no interpretation (no comfort) to ±0.01 and ±0.02 (a relatively high degree of comfort) to ±0.03 to ±0.06 (a range of intermediate comfort levels) to 0.10 (a low comfort level)–and some petrographers even decline to provide an estimate. The deftness, skill, and experience of the petrographer will usually dictate his or her comfort zone and, hence, the degree of interpretation of the items used to estimate the w/c. However, sometimes confounding that interpretation is the existing concrete condition, depending, for example, on the varieties of chemical and physical alteration that the concrete has undergone.

The acceptability of the petrographic estimates depends on: (1) the competency of the petrographer; (2) the validity of the techniques involved; (3) the ability of the petrographer to qualify the techniques used for the estimates; (4) the purpose to which the data will be used; and (5) the needs of those who either want to accept the estimates or debunk the estimates–for whatever reason.

ASTM Practice for Use of the Terms Precision and Bias in ASTM Test Methods (E 177) says, in so many words, that from a statistical viewpoint it is virtually impossible to have an actual, true reference to base accuracy on. As a result, in ASTM test documents where numerical values are obtained, there is a mandate that precision and bias statements, suitably qualified, be used to express accuracy.

Here is an example of how deceptive the w/c expressed as an average or mean value of a number of estimates can be. Jakobsen et al. [29], in a paper on the fluorescence method for estimating the w/c, used round robin tests to provide information on the accuracy of the method. Their "E" Round Robin Test Series has a mean w/c value of 0.47 versus the mix design w/c of 0.44, a difference of +0.03. That doesn't sound too bad. However, the six w/c values reported are from 0.40 to 0.55 (a difference of 0.15), which means that the variation from the mean is from −0.07 to +0.08. Although average or mean values may look good, the range of values is hidden. The reported standard deviation of 0.52 for those values is more realistic for expressing the variance of values of a method than the mean or average.

ASTM Committee C09.65 on petrography of hardened concrete is currently assessing different methods petrographers use for estimating the w/c of hardened concrete. The methods and techniques used for estimating the elusive w/c and w/cm of hardened concrete may never be developed to the satisfaction of all, but perhaps when that assessment is completed and some verification studies are done, the distance to the estimate may well be narrowed.

Environment

Singularly, the environment to which concrete is exposed has the greatest influence on its performance. If the petrographic examination is of laboratory-made concrete from an environmentally controlled exposure, many key factors controlling its performance and consequent alteration can be more easily deciphered than if the examination were of field concrete, in which many variables are unknown. Usually, for samples of field concrete, there is a decrease of available background information compared to examinations of new concrete or laboratory test specimens of concrete.

Sometimes an answer to the specific questions posed is not interpretable from the data initially uncovered even though the petrographer has recovered evidence not accessible by other approaches. Yet, the results of a petrographic study can be used to eliminate certain factors or direct attention to others. Complicating the interpretation is the fact that the deteriorated field concrete may have performed abnormally because of more than one cause.

Field concrete examined by a petrographer is concrete that has worried a responsible person enough to put forth the effort and expense of sampling and testing. Usually concrete sent for petrographic examination is "controversial" concrete. In practice, this usually means that sampling was from the poor concrete, and the results are frequently extrapolated to the entire area of concrete in a project. Unless samples representing all field and other conditions and exposures are included, there is a built-in bias in the sampling process–good concrete is rarely controversial.

Although, for example, it can be deduced from petrographic examinations of old outdoor-exposed concrete that the w/c was high or low, the cement content was low, medium, or high, fly ash or other supplementary cementing materials were or were not present, general quality of workmanship was good or inadequate, finishing was improper, etc., the alkali content of the cementitious materials cannot usually be reconstructed although it may have helped direct the performance of the concrete.

Finally, deteriorated field concrete usually shows superimposed traces of several processes, with at least one in an advanced stage. The most advanced process may conceal evidence of others that had a more important effect.

Evaluations of data from laboratory test exposure samples make the interpretative process easier because the materials and mixture design are known compared to natural exposures where many factors are unknown and environmental conditions are variable. Consequently, laboratory-induced procedures often result in different diagnostic "symptoms" than evidenced by field-exposed concrete. For example, samples of field concrete, when examined using light optical microscopy, are frequently found to contain secondary calcium carbonate near outer surfaces, along old cracks, and sometimes in the interior. That calcium carbonate, when examined by optical methods, is generally found to be calcite, rarely aragonite, and almost never vaterite (the birefringent spherulitic form of calcite containing interstitial water). Vaterite, however, was found by optical methods to be common in mortar bars that had been tested according to ASTM Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method) (C 227) and had been found in concrete specimens tested for resistance to freezing and thawing according to ASTM Test Method for Resistance of Concrete to Rapid Freezing and Thawing (C 666).

The use of X-ray diffraction to examine cement paste concentrates from field concrete has revealed that vaterite (not easily identified using optical methods) is frequently a major constituent of secondary calcium carbonate [30], especially on samples from seawater exposures or from other wet environments. Vaterite is known to persist for several months in laboratory specimens stored at room conditions; however, conversion of aragonite and the poorly crystallized vaterite to well-crystallized calcite in pastes and mortars has been clarified based upon work in Ref 31 that identified naturally occurring vaterite [32].

Concrete exposed to accelerated freezing and thawing in water using Procedure A of ASTM C 666 can produce a characteristic loss of surface mortar that is unlike the condition of specimens exposed, for example, on the mean-tide rack at Eastport, Maine [33]. Field concrete that is not air-entrained and has deteriorated because of natural freezing and thawing usually develops sets of sub-parallel cracks normal to the direction of the freezing front (because of directional freezing) that may not be reproduced in accelerated freezing and thawing in water.

Field concrete that has deteriorated because of alkali-silica reactions usually has much more advanced and conspicuous internal symptoms of this reaction than are found in mortar bars of expansive combinations examined after they are tested according to ASTM C 227. On the other hand, some field concretes regarded as undeteriorated have shown a range of evidence of alkali-silica reaction.

Alkali-carbonate and alkali-silica reactions sometimes exist concurrently in varying degrees of development in some concretes, and inconspicuous degrees of reaction may be the only recognizable peculiarities in cases of unsatisfactory service with possibly expensive consequences. Alkali-carbonate and alkali-silica reactions are described in other papers in this publication.

Because of varying specimen orientation and variable exposure conditions, features symptomatic of specific types of distress may not always be similar.

Petrographic Assistance in Other Types of Examinations

The identification of compositional properties of portland cement-based products analyzed using other methods and techniques is sometimes essential for proper interpretation of the analytical data. For example, ASTM Standard Test Method for Analysis of Hardened Masonry Mortar (C 1324) (also applicable to portland cement-based plaster and stucco) has an initial detailed section that deals exclusively with petrography and that supplements the second section on chemical analysis and allows suitable interpretation of the chemical data because these materials are made using portland cement and other calcareous and siliceous materials that skew the chemical data. ASTM Standard Test Method for Portland-Cement Content of Hardened Hydraulic-Cement Concrete (C 1084) provides for petrographic examinations as a means of understanding chemical influences of constituent materials, other than portland cement, on the analysis. These materials include aggregates, pozzolans, supplementary cementing materials, and mineral admixtures. Petrographic analysis of post-test specimens, such as specimens from a variety of ASTM tests involving alkali-silica or alkali-carbonate reactivity, is mentioned in those test standards as a means of providing supplemental assessment of damage.

Conclusion

The kinds of information about hardened concrete that petrographic examinations reveal can be used to identify a great variety of data about its physical and chemical makeup. That information, in turn, can be used to identify unsound aspects of portland cement that have led to bulk concrete expansion [34,35]; improprieties of aggregates that can cause low concrete strength [36]; stray naturally occurring organic chemicals in aggregates that can cause variable concrete air contents [8]; the causes of concrete failure to set; rapid slump loss; poor proportioning and batching; factors resulting in large concrete shrinkage; early and late concrete instability due to a variety of causes; conformance to specifications; malperformance due to chemical reactions of aggregate; surface distress due to improper finishing, curing, and inadequate air entrainment; and poor performance due to composition, manufacture, and exposure to aggressive environments for which it is not designed.

Before the petrographic trail was blazed and during its early adventuresome period, the factors that led to good or poor concrete performance were unclear; even today, with new and exciting methods and techniques being constantly developed, we still have some ignorance concerning these factors. However, there is no need to view our analytical level of understanding as insignificant.

It makes no difference if the concrete is a 10 ton cherry pie 14 ft (4.2 m) in diameter and 2 ft (0.6 m) deep constructed in Charlevoix, Michigan to commemorate the birthday of George Washington, or a dam, nuclear reactor facility, bridge, foundation, wall, pavement, sidewalk, concrete electrical insulator, the liner of a bank safe, or coffin vault located anywhere in the world; the basic constituents are similar at any age. Petrographic methods are ideal for evaluating how concrete has been put together and changes that have occurred to it since it was installed, and provides an understanding of its behavior and performance. It is invaluable in assisting engineers and others in understanding why it failed to function as expected and, less often, why it functioned as expected.

Petrography of concrete is flourishing. Like in other sciences, the development of new methods and techniques of examination, more sophisticated equipment, more qualified petrographers, more laboratories available to complete work, and better understanding of the physical and chemical makeup of concrete now make possible better interpretations of the causes of concrete performance and malperformance.

Along with improvements in analytical methods and equipment comes a new problem—the development of petrographers who are able to obtain the "right" analytical data and also have the background to interpret the data. There is a need for hands-on appreciation of mixtures of portland cements; blended cements; mineral and chemical admixtures; water; varieties of coarse and fine aggregates; methods of mixing, placing, and consolidation; the physics and chemistry of what happens to and within plastic and hardened concrete; and the effects of time and environmental exposure on performance. Too frequently, the analyst becomes a specialist who operates in a narrow walkway of self- or organization-imposed semi-isolationism.

In this day of specialization, the techniques and particularly the analytical equipment are very expensive. The cost of getting petrographic information dramatically increases as the size and scale of what is analyzed progressively decreases. However, there is still a lot of information obtainable at moderate and justifiable costs.

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21

Volume Change

Fred Goodwin[1]

Preface

IN THE PREPARATION OF THIS CHAPTER, THE contents of the 4th edition of this series, *ASTM STP 169C*, were drawn upon. The author acknowledges the author of the volume change chapter in the 4th edition, P. Kumar Mehta [1], as well as those in the previous editions. In *ASTM STP 169* and *ASTM STP 169A*, the chapter on volume changes in hardened concrete was authored by George W. Washa [2,3]. In *ASTM STP 169B,* the chapter was authored by James L. Sawyer [4]. In the interest of consistency, the essential format as well as many of the fundamental aspects of the earlier publications are retained in this paper. The current edition will review and update the topics as addressed by the previous authors, introduce new technology that has been developed, and include up-to-date references.

Introduction

Concrete is subject to several types of volume changes during its service life. The magnitude of volume change is generally stated in linear rather than volumetric units, since test data are generally evaluated in these terms. In engineering practice, the volume change is generally expressed as "strain," which is defined as the change in length per unit length and therefore is a dimensionless quantity. The term "microstrain" is frequently used to quantify the magnitude of shrinkage. One microstrain is 1 millionth of a unit length change per unit length change (i.e., 1 millionth of a metre per metre or 1×10^{-6} inch per inch).

Freshly mixed concrete remains plastic for a relatively short period of time. During this period, volume changes can occur due to cement hydration, water evaporation, thermal change, absorption, and bleeding. These types of volume changes, commonly called "plastic shrinkage," are discussed elsewhere in this book.

The instantaneous strain on loading that is fully reversible and also is proportional to the applied stress is called "elastic strain." On sustained loading, especially when the applied stress level is 50 % or more of the ultimate strength of concrete, the material shows nonlinear stress-strain behavior. The gradual increase in strain with time under a constant load, which is not completely reversible on unloading, is termed "creep strain" or, more commonly, "creep." The strains resulting from concrete's response to applied stress are discussed elsewhere in this publication and therefore are not covered by this chapter.

There are other types of volume change that occur as a result of concrete's response to environmental effects such as wetting, drying, heating, cooling, freezing, and thawing. Thermal volume changes are discussed in the chapter by Tatro, and moisture effects are covered in the chapter by Hearn et al. Due to the relatively low tensile strength of concrete, nonuniform restrained expansions or contractions can easily exceed the cohesive tensile strain capacity of the concrete resulting in the formation of cracks.

Ingress of sulfate ions from the environment or an excess of sulfate intermixed within the concrete causes an expansive reaction known as sulfate attack resulting in the formation of ettringite (calcium sulfo-aluminate). This expansive mechanism is discussed in the chapters by Forster and Meininger.

Concrete's reaction with carbon dioxide from the environment (carbonation) lowers the pH (thereby lowering the corrosion threshold of imbedded reinforcing steel) as well as causes shrinkage due to chemical reaction with the cement hydration product. Carbonation that occurs during the first few hours following concrete placement causes a soft, dusty surface on the area that is exposed to the carbon dioxide [5]. Corrosion of imbedded steel, while not a volume change of the concrete constituents, still results in cracking and spalling of the concrete due to the expansive nature of the formation of steel oxidation products within the rigid concrete matrix.

Uncombined crystalline calcium oxide (CaO) and magnesium oxide (MgO) present in the cement or intermixed within the cement cause another expansive mechanism as the hydroxides of these minerals form during hydration.

The reaction of alkalis (sodium and potassium) with certain types of aggregates as described in ASTM C 295 and other references produces expansive reaction products causing cracking and potential disintegration of the concrete. This topic is covered in more detail in the chapters on petrographic examination by Erlin and Wong, respectively.

Volume changes that are not covered elsewhere include carbonation shrinkage, drying shrinkage, and expansion due to hydration of free CaO and MgO in hardened concrete. Also discussed in this chapter is the autogenous shrinkage associated with cement hydration, which can occur in sealed and unloaded specimens. The last section of this chapter contains a brief description of volume change in expansive cements that are used in the production of shrinkage-compensating concrete.

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Under conditions of restraint, the volume changes involving shrinkage commonly produce cracking through the generated tensile stress from shrinkage exceeding the tensile strain capacity of the concrete. Significant work is underway to relate shrinkage, creep, tensile strength, and modulus of elasticity; however, standards are only now beginning to be developed [6,7]. Concrete typically is under sufficient restraint due to connecting members, foundations, subgrade friction, steel reinforcement, etc. to exceed the tensile strain capacity. While cracking may not be serious enough to jeopardize the structural integrity of an element, it is still undesirable for a variety of reasons.

Cracks create a plane of weakness within the formerly homogeneous structure. Continuing movement of cracks frequently causes problems with coatings, toppings, or overlays installed as barriers to protect the underlying concrete. Cracks in structures subjected to traffic can rapidly deteriorate the adjacent concrete due to point loading of the crack edges as well as D-cracking in freezing environments. Cracks also look bad and are the most frequent subject of complaints about concrete, particularly architectural concrete. More importantly, large external cracks, interlinking with internal voids and micro cracks that are always present in concrete, make it possible for water, harmful chemicals, and gases to penetrate with relative ease into the interior of the concrete mass. This phenomenon is likely the most common cause of numerous concrete durability problems with field structures.

Unrestrained, expansive volume changes also cause cracking and are therefore harmful. Frequently, restrained controlled volume changes are used to shrinkage-compensate concrete and mortars in an attempt to produce expansion at rates and magnitudes approximately equal to the shrinkage. Many of these mechanisms rely on attempts to control otherwise deleterious reactions such as ettringite formation, uncombined calcium oxide, and metal oxidation. If conditions exist in which insufficient restraint or inhomogeneous uncontrolled expansion occurs, internal fractures can develop within the concrete causing failure.

Autogenous Volume Changes

Autogenous volume changes are associated with cement hydration alone and do not include environmental effects due to variation in moisture and temperature. Autogenous deformation combines the mechanisms of chemical shrinkage and self-desiccation [8]. The autogeneous volume change with ordinary portland cement concrete is usually small, that is, less than 0.010 % or 100-microstrain expansion or shrinkage. The magnitude is dependent on the overall effect of two opposing phenomena: (*a*) the increase in the disjoining pressure in poorly crystalline C-S-H[2] and ettringite due to water adsorption and (*b*) the reduction in the disjoining pressure due to removal of adsorbed water by desiccation. According to Washa [2], in most instances the initial expansions obtained during the first few months do not exceed 0.003 %, while the ultimate shrinkage obtained after several years usually does not exceed 0.010 %. Differences between volume and length change measurements of autogenous deformation have been reported by Barcelo et al. [9].

Chemical shrinkage results from the chemical reaction of the cement with water, which produces hydration products of less absolute volume than the volumes of the reactants, resulting in the densification of the solid phases during the hydration process [8]. Chemical shrinkage has been determined by dilatometry and by the change in weight of a hydrating sample suspended in water [10]. The factors affecting chemical shrinkage of hydrating cement systems include the mineralogical composition of the cement, the particle size distribution of the cement [11], the kinetics of the hydration reaction, and the use of supplemental cementing materials and other admixtures [12,13]. The magnitude of chemical volume change in concrete for a given consistency increases as the fineness of cement and the cement content are increased. Ultimate contraction appears to be somewhat greater for high C-S-H forming cements (namely, ASTM Type IV cement) than for normal portland cement (Type I). The addition of pozzolans such as silica fume and metakaolin, which increase the amount of CSH formation, can therefore also increase the amount of autogenous shrinkage. Chemical shrinkage is the dominant factor in autogenous shrinkage during the time prior to setting or the formation of a self-supporting skeleton of hydration products. Self-desiccation becomes dominant after setting [14,15].

Occasionally, a period of swelling has been observed beginning a few hours after initial mixing and lasting up to 20 h. This appears to be due to the formation of ettringite as some correlation with C3A, C4AF, and gypsum levels has been found [16–18].

Self-desiccation results from the consumption of the internal water due to the hydration of the cement. The formation of a rigid structure of decreasing permeability as hydration progresses decreases the migration of water to the hydrating regions near the cement particles. Air fills the pore structure of the concrete resulting in a decrease of internal humidity within the system. Hydration can cease to occur when the relative humidity of the pore structure falls below 75–80 % [19,20]. The development of high-strength systems with very low water-cement ratios has brought the phenomenon of autogenous shrinkage to the attention of researchers again [21,22]. Using cement pastes made with a 0.23 or 0.30 water-cement ratio, a high early-strength portland cement (430 m^2/kg, Blaine), and a superplasticizer, Tazawa and Miyazawa [23] reported that the autogenous shrinkage of sealed specimens at an age of 70 days was of the order of 1000 microstrain. This autogenous shrinkage value increased to almost double when the cement was replaced with 10 or 20 % condensed silica fume by weight.

Carbonation Shrinkage

Carbonation shrinkage occurs as a result of chemical interaction between atmospheric carbon dioxide (CO_2) and hydration products of cement. Carbonation shrinkage is probably caused by dissolving crystals of $Ca(OH)_2$ while under compressive stress imposed by drying shrinkage and the subsequent deposition of $CaCO_3$ in spaces free from stress [24]. Hydration products near the pore dissolve continuously to furnish Ca^{++} ions to the liquid phase from which the ions migrate to the $CaCO_3$ nucleating site, where the crystal growth takes place.

Since it takes place concurrently with drying shrinkage, most reported data do not distinguish between the two and designate both as drying shrinkage. As the cement hydrate is carbonated, the mass of the sample increases. Drying shrinkage is

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[2] Cement chemistry abbreviations are used: C = CaO, S = SiO_2, A = Al_2O_3, F = Fe_2O_3, S = SO_3, and H = H_2O.

accompanied by a loss of water resulting in a mass loss. This inverse relationship can prove misleading when the specimen mass is used to determine the equilibrium point of drying [25].

The rate of carbonation is dependent on several factors such as porosity of concrete, size of the member, relative humidity, temperature, CO_2 concentration, time of exposure, method of curing, and the sequence of drying and carbonation. Carbonation proceeds slowly and usually produces little shrinkage at relative humidities below 25 % or near saturation. At low humidities, there is insufficient water in the pores for the CO_2 to form carbonic acid and react with the alkaline cement hydrates. As the pore humidity approaches saturation, the diffusion rate of CO_2 decreases. With permeable concrete, the magnitude of carbonation shrinkage may approach the magnitude of drying shrinkage in a CO_2-rich environment at 50–65 % relative humidity [26]. Concrete that has been subjected earlier to carbonation shrinkage will still shrink or swell with changes in relative humidity; however, the magnitude of these volume changes will be smaller than before carbonation.

Carbonation reactions, such as the one shown here, tend to release moisture

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$$

All of the constituents of hydrated portland cement, namely, C-S-H, CH, ettringite, and monosulfate hydrate, are eventually subject to carbonation. The porosity of the cement paste and the specimen size are controlling factors in carbonation because they determine the diffusion rates of CO_2 ingress and the egress of moisture released by CO_2 reacting. While autoclaved blocks are relatively free from carbonation, concrete blocks cured in steam at atmospheric pressure show maximum carbonation shrinkage near 50 % relative humidity [26]. Also, it is reported that precarbonation improves the volume stability of blocks cured in steam at atmospheric pressure.

Ying-yu and Qui-dong [27] investigated the mechanism of carbonation of cement mortars and the dependence of carbonation on pore size. Since the gaseous-phase carbonation process is a chemical reaction controlled by diffusion, the authors suggest "prediction of the carbonation coefficient" by the equation

$$a_1(2C_1/kP)^{1/2}$$

where C_1 is the partial pressure of CO_2, k is a constant, and P is porosity. The authors found that pores with radii above 32 nm increased the carbonation coefficient greatly.

Factors influencing the depth of carbonation in different concrete types were discussed in several studies. Schubert [28] reported the carbonation behavior of mortars and concretes containing different types of cements and fly ashes. The author found an inverse relationship, $(1/\sqrt{fc})$, between the carbonation rate and the 28-day compressive strength. Cements with unusually low lime contents (that is, 70 % slag cement) showed increases in carbonation rates. Carbonation depths of mortar specimens moist-cured for 28 days were much less than the 7-day moist-cured specimens. For fly ash concretes with seven days of standard moist curing, Ho and Lewis [29] found that carbonation depth depended mainly on the water-cement, not the water-binder, ratio regardless of the mix constituents used in their study.

Dhir et al. [30] studied the effect of 28-day concrete strength on depth of carbonation in 20 years, using an accelerated test method. It was found that superplasticized concrete had a lower depth of carbonation than the corresponding normal concrete. Malhotra et al. [31] reported that a superplasticized, high-volume fly ash concrete containing 56 % fly ash by weight of the cementitious material, with 30 MPa compressive strength at 28 days, showed only 3 mm carbonation in one year and 7 mm in five years at 50 ± 5 % relative humidity. Paillere et al. [32] found that portland cements containing up to 29 % Class F fly ash or blast furnace slag showed similar behavior to carbonation as neat portland cements. At higher levels of cement replacement, the carbonation of concrete increased.

Oye and Justness [33] investigated the carbonation resistance of latex-modified cement mortars and reported that, contrary to a general improvement in other properties, the carbonation resistance of latex- and epoxy-modified mortars showed no significant improvement when compared to unmodified mortars. Aimin and Chandra [34] investigated the effect of acrylic polymer additions on the rate of carbonation of portland cement paste. Increasing the polymer content reduced the porosity and water absorption of the cement paste, but the rate of carbonation reached maximum at 10–15 % polymer loading. Sakuta et al. [35] found that the addition of amino alcohol and glycol ether derivatives was effective in controlling carbonation. Amino alcohol derivatives that are used for desulfurization and deoxidation possess the ability to absorb atmospheric CO_2. The use of a water-insoluble glycol ether derivative as an anti-foaming agent reduced the size of air bubbles in concrete thereby decreasing the permeability. Paillere et al. [32] investigated the effect of freezing and thawing (F-T) cycles on air-entrained concrete containing fly ash or slag. With increased F-T cycles, carbonation depths were increased. Whereas the presence of entrained air improved the resistance to freezing and thawing cycles, it did not help against the penetration of CO_2; Massazza and Oberti [36] reported that carbonation depths of pozzolanic cements were similar to those of portland cements provided that concretes having similar strengths were compared. The carbonation behavior of blended cements was inferior to portland cement only when the moist-curing period was too short. With prolonged moist curing, there was usually an improvement in carbonation resistance of blended cements.

Sakai et al. [37] compared the carbonation characteristics of a Type K expansive cement concrete with ordinary portland cement concrete from a 22-year-old building in Nigata Prefecture, Japan. Core samples with 58 MPa compressive strength and 0.57 water-to-cement ratio were found to have carbonation depths of about 12 mm. Mineralogical analysis of the carbonated material confirmed that C-S-H was decomposed to calcite and silica gel, whereas the ettringite phase in the expansive cement concrete decomposed to calcite, gypsum, and $Al(OH)_3$ gel. The authors observed that the decomposition rate of ettringite by carbonation was somewhat slower than that of C-S-H.

Drying Shrinkage

When plain, normal-weight, hardened concrete is dried from a saturated condition to a state of equilibrium with air at 50 % relative humidity, the shrinkage associated with moisture loss is in the range of 0.04–0.08 % (400 to 800 microstrain). The change in volume of drying concrete is not equal to the volume of water removed. The initial loss of water causes little or no shrinkage since the most easily evaporable water resides in large pores of the concrete. Further evaporation of adsorbed water is reversible upon resorbtion of additional moisture [38]. One source of irreversible drying shrinkage in concrete is the loss of water held in small capillary pores of the hydrated cement paste [39]. It has been suggested that the evaporation of capillary water causes a disjoining pressure when confined to narrow spaces between two solid surfaces. The removal of the capillary water reduces the disjoining pressure and brings about the shrinkage of hydrated cement paste on exposure to drying conditions. In regard to capillary water, it has been suggested that the water meniscus in small capillaries (5–50 nm) exerts hydrostatic tension, and removal of this water tends to induce a compressive stress on the walls of the capillary pores, thus contributing to the overall contraction of the system. The introduction of shrinkage-reducing admixtures has allowed a reduction in drying shrinkage of up to 50 %. Most of these materials reportedly work by changing the capillary water surface tension [40].

Among the more important factors influencing the drying shrinkage of concrete are the content of cement paste, the water-cement ratio, the degree of hydration, the elastic modulus of aggregate, the characteristics and amounts of admixtures used, the time and the relative humidity of exposure, the size and shape of the concrete mass, and the amount and distribution of internal reinforcement (such as steel and fibers [41]). Drying shrinkage of concrete and the factors influencing it are discussed in numerous reports, such as those authored by Ytterberg [42] and Meininger [43]. A brief discussion of these factors follows.

Concrete is made up of two constituents: cement paste and aggregate. The former shrinks and the latter restrains shrinkage. The effectiveness of aggregate to restrain the shrinkage of cement paste is related to the elastic modulus of the aggregate. From the data obtained by an experimental study of concrete mixtures with water-cement ratios of 0.35 or 0.50, Powers [44] developed the following expression showing how the drying shrinkage of concrete (S_c) is related to the drying shrinkage of cement paste (S_+), the volume fraction of the cement paste $(1 - g)$, and a constant (n) that is dependent on the elastic modulus of aggregate:

$$S_c = Sp(1 - g)^n$$

Thus, for a given aggregate and cement paste the drying shrinkage may be doubled when the aggregate volume fraction is decreased from 80 to 50 %. Similarly, long-term studies on the effect of aggregate type on drying shrinkage [45] showed that with a given concrete mixture the 23-year shrinkage was more than twice for the low-elastic-modulus aggregates than for the high-elastic-modulus aggregates. Concrete made with dirty sand or with unwashed aggregates containing silt and clay shrinks significantly more than concrete made with clean aggregates. To the extent that the water requirement for a given consistency of concrete is influenced by the maximum size, grading, shape, and surface texture of the aggregate, these factors also affect drying shrinkage.

The quality of the cement paste is primarily a function of the water content for a given cement content in concrete (water-cement ratio) and the degree of hydration, which in turn depends on the fineness and chemical composition of cement as well as the curing time, temperature, and humidity conditions. Since unhydrated cement particles can offer restraint to shrinkage of hydrated cement paste in the same manner as aggregate particles, the factors leading to a greater degree of hydration generally contribute to a higher drying shrinkage. Thus, finer cements usually show greater shrinkage values. Portland cements with high-C_3A and high-alkali contents tend to give high drying shrinkage, which can be controlled by using optimum gypsum content via ettringite formation and the associated shrinkage-compensating effect (see the last section of this chapter that deals with testing methods).

Admixtures that increase the water requirement of concrete for a given consistency (namely, diatomaceous earth or calcined clays) generally increase drying shrinkage but many water-reducing admixtures that reduce the water content do not reduce drying shrinkage. Accelerating admixtures such as calcium chloride and triethanolamine tend to increase drying shrinkage.

Atmospheric diffusion of the adsorbed water present within cement paste and the water held by capillary tension takes place over a long period of time and is accelerated by high environmental temperatures, high porosity, and low relative humidity. For a wide range of concrete mixtures only 20–25 % of the 20-year drying shrinkage was realized in two weeks, 50–60 % in three months, and about 75 % in one year. Almost twice as much drying shrinkage is obtained at 45 % relative humidity as compared to 80 % relative humidity exposure.

The size and shape of the concrete mass have a considerable effect on the rate and total amount of drying shrinkage. The rate and ultimate shrinkage of a large mass of concrete are smaller than the values for small-size concrete elements, although the shrinkage process continues over a longer period for the large mass [46]. In full-sized concrete members, orientation and edge effects complicate this relationship. There appears to be a linear correlation between the volume-to-surface ratio and the logarithm of shrinkage, as well as to the logarithm of time required for half of the ultimate shrinkage to be achieved [47]. In realistic terms, it seems that slower drying conditions yield lower ultimate shrinkage values, but theoretically the ultimate shrinkage may be independent of size [48,49]. Also, in massive concrete members differential drying conditions produce larger shrinkage at and near the surface. This gives rise to tensile stresses at and near the surface while compressive stresses are developed in the interior. Consequently, surface cracking may occur if the tensile stresses exceed the tensile strength of the material and the stress relaxation provided by creep. These cracks then increase the available surface area for evaporation of water, as well as cause redistribution of internal stresses.

Shrinkage of reinforced unrestrained structures produces tension in concrete and compression in steel. By increasing the amount of reinforcement, the shrinkage can be correspondingly reduced but it will increase the tensile stresses in concrete such that it is likely to crack when excessive reinforcement is used. Reinforced concrete elements with normal amounts of reinforcing steel may show drying shrinkages of the order of 0.02–0.03 %. An increase in steel reinforcement will cause cracks at a closer spacing and also reduce the crack widths. Fibers added to concrete also redistribute the stresses resulting in a similar reduction in crack width and closer spacing of the cracks [41].

According to Washa [2], drying shrinkage values for structural lightweight concrete may vary between 0.04–0.15 %. Higher shrinkage values generally result from concretes containing aggregates that have high rates of absorption, low modulus, a high proportion of fineness, and/or require high cement contents to obtain the specified strength. Moist-cured, cellular (foamed) products made with neat cement and weighing 10–20 lb/ft^3 (0.22–0.44 kg/m^3) may have drying shrinkage as high as 0.3–0.6 %. Autoclaved concrete products containing fine siliceous additives may weigh 40 lb/ft^3 (0.88 kg/m^3) and have drying shrinkage in the range of 0.02–0.1 %. Conversely, internal curing occurs when saturated lightweight aggregate is used, resulting in a decrease in shrinkage [50].

Silica fume is being used increasingly as a supplementary cementing material for concrete elements. Data from ACI 234R indicate that the drying shrinkage of silica fume concrete is generally comparable to that of a control concrete after 28 days of moist curing regardless of the water-to-cementitious materials ratio *(w/c* + m) [51]. The silica fume quantity and curing regimen prior to drying were found to be important factors. Concrete shrinkage is influenced little by silica fume content up to 10 % by mass of cement [52]. Early drying increases shrinkage for lean silica fume mixtures *(w/cm* greater than 0.60) and for high silica fume contents (greater than 10 % by mass of cement) because early drying inhibits the pozzolanic reaction. Fly ash and ground granulated blast furnace slag in the concrete mixture are reported to both increase drying shrinkage (probably due to the increased gel formation of the pozzolanic reaction products) as well as to have minimal effect [53–55]. Incorporation of silica fume leads to reduced pore sizes in the cement paste, thus increasing the surface tension in small capillary pores and therefore the autogenous shrinkage.

Expansion Due to Hydration of Free CaO and MgO

It has been often reported in the published literature that hydration of crystalline MgO (periclase) or CaO, when present in significant amounts in a portland cement, can cause expansion and cracking. The crystalline size, the thermal history of the material, and the solubility of the material within the other clinker constituents determine the reactivity of both CaO and MgO in cement. Very fine particles react rapidly after mixing with water and likely are consumed in the plastic state of the cement. The higher the calcining temperature of both CaO and MgO, the lower the reactivity, as the crystalline structure becomes denser. CaO and MgO are both chemically combined within the clinker phases. Low burning temperatures can leave unreacted calcium oxide present in the cement, as well as clinker composition containing calcium levels above the stochiometric requirement for combination with silica, alumina, and ferrite (Lime saturation factor $>$ 1) [56].

Laboratory tests on early portland cements showed that the cement pastes made with low-MgO cements containing over 3 % free (crystalline) CaO gave considerable expansion, which caused cracking of the unrestrained paste [57]. The phenomenon is virtually unknown in modern portland cements due to better manufacturing controls, a result of which is that the content of uncombined or crystalline CaO seldom exceeds 1 %. In addition to analytical verification of the free CaO content, physical verification using the ASTM C 151 [58] autoclave expansion test is now also conducted. Expansive additives for cement containing a large amount of crystalline CaO are being used in Japan to obtain controlled expansions under restraint in shrinkage-compensating concretes, which are described in the next section of this chapter.

In regard to crystalline MgO or periclase, some early portland cements, which were made at much lower temperatures than used today, contained large amounts of this compound. The expansion and cracking of cement pastes and concretes containing these cements were attributed to periclase. It is now accepted that the periclase formed in modern portland cement clinker at a kiln temperature of 1400–1500°C is either inert to moisture or too slow to hydrate under ambient temperature conditions. Only under autoclaving conditions, in accelerated laboratory tests such as ASTM Test Method for Autoclave Expansion of Portland Cement (C 151), is periclase known to hydrate and cause considerable expansion in unrestrained pastes (see the section on test methods and specification for additional discussion). Since portland cements meeting ASTM Specification for Portland Cement (C 150) [59] are required to comply with the maximum limits of expansion in the autoclave test, it is one of the chemical requirements of ASTM C 150 that the total MgO content of portland cement shall not exceed 6 %. Note that approximately 2–5 % MgO goes into the solid solution of portland cement clinker compounds (C_3S, C_2S, C_3A, and F_{ss}),[3] and the remainder shows up in the form of periclase.

Volume Changes in Expansive Cements

ASTM Specification for Expansive Hydraulic Cement (C 845) [60] covers hydraulic cements that expand during the early hardening period after setting. Large expansion occurring in an unrestrained cement paste can cause cracking. However, if the expansion is adequately restrained, its magnitude will be reduced but a self-stress will develop. When the magnitude of expansion is sufficiently small such that the prestress developed in concrete is on the order of 25–100 psi (0.2–0.7 MPa), the cement is known as shrinkage-compensating. This amount of expansion is generally enough to offset the effect of tensile stress due to subsequent drying shrinkage. During the last 20 years, the cements of this type have been commercially used for making crack-free industrial floors, airport taxiways, water-storage tanks, and post-tensioned concrete members for parking garages.

The formation of ettringite and hydration of crystalline CaO are two mechanisms known in concrete technology that are capable of causing disruptive expansion. Both phenomena have been harnessed to produce shrinkage-stress-compensating concretes. The expansive additive in U.S. practice is a modified portland-cement clinker that contains significant amounts of anhydrous calcium aluminosulfate ($4CaO{\cdot}3Al_2O_3{\cdot}SO_3$) and calcium sulfate in addition to the cementitious compounds of portland cement, $3CaO{\cdot}SiO_2$, $2CaO{\cdot}SiO_2$, and $4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$. The cement produced by blending this additive with normal portland cement is called Type K expansive cement.

In Fig. 1, a graphical representation is given of the concept showing how a Type K shrinkage-stress-compensating cement, when compared to normal portland cement, works to reduce the risk of drying shrinkage cracking [61]. Immediately following the start of hydration of Type K cement, it is observed that large amounts of ettringite start forming. After initial set, the concrete will bond to the reinforcing steel and any expansion will be restrained by the steel. Under these conditions, the steel will go into tension and concrete into

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[3] F_{ss} stands for the calcium aluminoferrite solid solution phase.

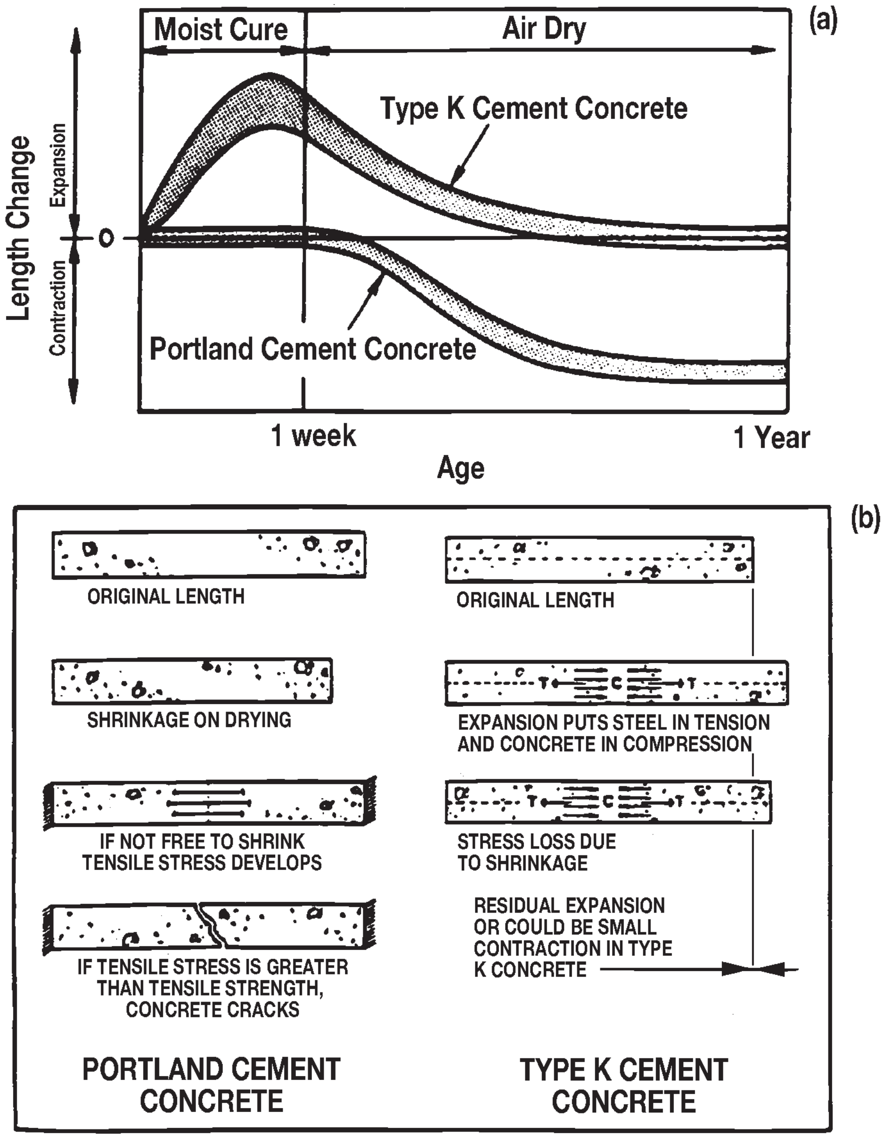

Fig. 1—(*a*) Comparison of length change characteristics between portland cement and Type K cement concretes, and (*b*) illustration showing why Type K cement concrete is resistant to cracking from drying shrinkage (Ref 38).

compression. At the end of the moist-curing period when the element is exposed to environmental drying conditions, it will shrink in the same manner as normal portland cement concrete. However, the shrinkage will first relieve the precompression in concrete before tensile stresses have a chance to develop. Thus by preventing the development of large tensile stresses, the expansive cements can become instrumental in reducing the risk of cracking in concrete from drying shrinkage. Due to their ability to produce reinforced concrete members that suffer little or no dimensional change during their service life, the shrinkage-compensating cements are sometimes called nonshrinking cements. This, however, is misleading because concretes made with expansive cements do show almost the same amount of drying shrinkage as normal portland cement concrete (Fig. 1*a*). With the former the overall dimensional change is negligible because of expansion during moist curing, which precedes the shrinkage on drying.

As with normal portland cement concrete, the rate and magnitude of drying shrinkage with Type K cement concrete is influenced by the aggregate content and type and water content. With increasing water-cement ratio, the expansion during this moist-curing period is reduced proportionately; therefore, the residual dimensional change after the drying shrinkage changes from positive to negative at high water-cement ratios. This effect is shown by test data from Polivka and Willson [62] in Fig. 2. With a water-cement ratio of 0.53 or less, the magnitude of initial expansion was large enough to ensure a residual expansion after two months of drying shrinkage. Since the magnitude of expansion and degree of precompression is reduced considerably with water-cement ratios above 0.6, it is recommended that water-cement ratios lower than 0.6 be used

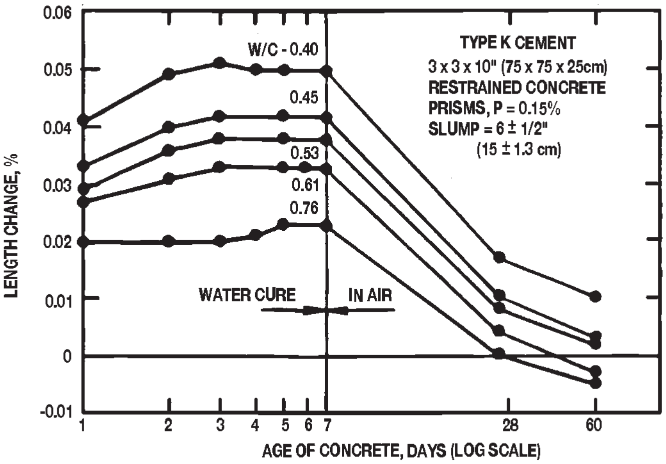

Fig. 2—Effect of w/c on restrained expansion and shrinkage of a Type K expansive cement concrete (Ref 39).

with Type K expansive cements, even when it is not needed from the standpoint of structural strength.

To develop adequate expansion and precompression in concrete, Kesler [63] found that good moist-curing conditions are absolutely essential (Fig. 3).

Test Methods of Determining Volume Changes

For evaluation of volume changes discussed in this chapter, ASTM offers several methods that are listed here:

1. ASTM Test Method for Autoclave Expansion of Portland Cement (C 151) [58]
2. ASTM Test Method for Length Change of Hardened Hydraulic Cement Mortar and Concrete (C 157) [64]

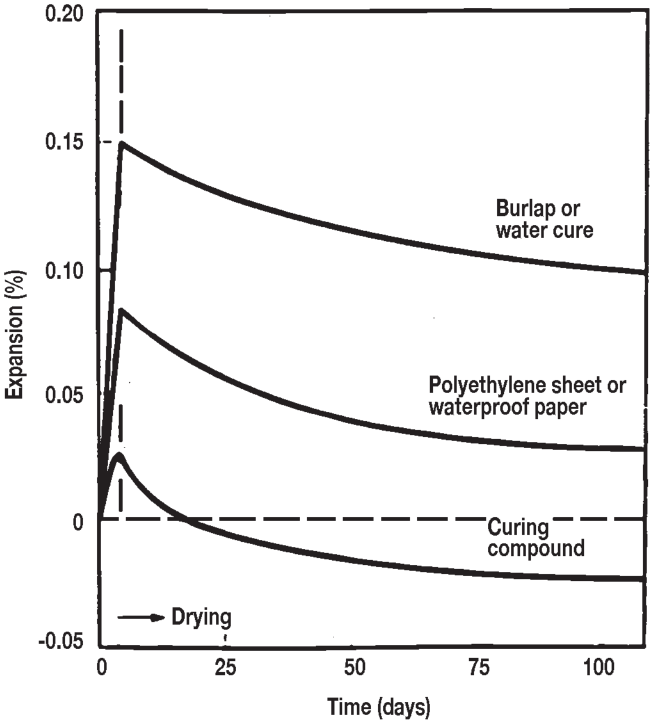

Fig. 3—Effect of curing conditions on restrained expansion of expansive cement concrete (Ref 40).

3. Standard Practice for Length Change of Drilled or Sawed Specimens of Hydraulic-Cement Mortar and Concrete (C 341) [65]
4. ASTM Test Method for Drying Shrinkage of Mortar Containing Portland Cement (C 596) [66]
5. ASTM Test Method for Restrained Expansion of Expansive Cement Mortar (C 806) [67]
6. Standard Test Method for Change in Height at Early Ages of Cylindrical Specimens of Cementitious Mixtures (C 827) [68]
7. Test Method for Restrained Expansion of Shrinkage-Compensating Concrete (C 878) [69]
8. ASTM Test Method for Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory (C 940) [70]
9. ASTM Test Method for Measuring Changes in Height of Cylindrical Specimens from Hydraulic-Cement Grout (C 1090) [71]
10. ASTM Test Method for Determining Age at Cracking and Induced Tensile Stress Characteristics of Mortar and Concrete under Restrained Shrinkage (C 1581) [72]

A brief description of these methods, including the scope and significance of each, follows.

Portland Cement

ASTM C 151 provides an index of potential delayed expansion caused by the hydration of CaO or MgO, or both. The method covers determination of the autoclave expansion of portland cement by testing neat cement paste specimens (25.4 by 25.4 by 285.8 mm) that are moist cured for 24 ± ½ h and then exposed to the action of steam under a pressure of 2 ± 0.07 MPa for 3 h. The rates at which pressure is increased in the beginning of the test and released at the end are specified. Linear expansion is measured by a micrometer comparator over an effective gage length of 254 mm. ASTM C 150 for portland cement permits a maximum expansion of 0.80 %.

The author of this chapter in STP 169C [1] (the previous edition) expressed his opinion that "The autoclave test for expansion is able to provide a quantitative measure of the expansion caused by delayed hydration of free CaO and crystalline MgO. The critics of the test say that the measured expansion has no significance because: (1) exaggerated expansion values are obtained by destroying the cohesive forces that would be present in normally hardened cement pastes; (2) the test conditions force the crystalline MgO present to hydrate and expand, whereas in normally cured commercial portland cements, within permissible chemical limits (i.e., maximum 6 % total MgO), the MgO present is either inert or hydrates at a rate that is too slow to be of any consequence; (3) the magnitudes of expansion in neat cement paste bars due to hydration of the free CaO normally present may be high (that is, high enough not to meet the requirements of the ASTM C 150 specification limit of maximum 0.80 % expansion), but in corresponding concretes it will not be high enough to cause significant expansion and cracking (due to the restraining effect of the aggregate); and (4) no correlation has ever been shown between the autoclave test specification limit and the soundness (cracking potential) of concrete." The fact remains that although the C 151 test may be overly severe, deleterious expansion problems caused by uncombined CaO and MgO are not issues with cements manufactured according to C 150 and C 1157 standards.

Another commonly used test serving this purpose is Le Chatelier's method. The Le Chatelier apparatus is a 30-mm, longitudinally split, cylindrical mold with indicator needles. At the end of a 24-h normal moist-curing period, the mold containing the cement paste is exposed to boiling water at atmospheric pressure for a period of 3 h. The specification for cement soundness according to the Le Chatelier test permits a maximum distance of 10 mm between the indicator points. Recently, the European Committee for Standardization has approved the use of the Le Chatelier method for testing cement soundness (EN 197) [73].

Portland Cement Mortar and Concrete

ASTM C 157 provides a method for potential volumetric expansion or contraction of mortar or concrete due to various causes other than applied stress or temperature change [64]. The method is particularly useful for comparative evaluation of potential unrestrained expansion or shrinkage in different hydraulic-cement mortar or concrete mixtures. The test utilizes triplicate 25.4 by 25.4 by 280-mm mortar prisms or 76.2 by 76.2 by 280-mm concrete prisms with 25.4-mm maximum size aggregate and 254-mm gage length in both cases. The specimens are kept in molds for 23.5 ± 0.5 h, or longer if necessary to prevent damage, then they are demolded, their initial measurements are taken, and they are moist-cured for 28 days. After curing, they are stored at 23.0 ± 1.7°C in water or in air at 50 ± 4 % relative humidity. The specimens in air storage have a clearance of at least 25 mm on all sides. Comparator readings after air storage are taken at 4, 7, 14, and 28 days, and after 8, 16, 32, and 64 weeks unless otherwise specified. Similarly, water storage readings are taken at the age of 8, 16, 32, and 64 weeks.

It may be pointed out that ASTM C 157 is intended for use in a standard laboratory environment. Much greater variability and, in some cases, higher drying shrinkage values result when specimens are cast in the field under temperature and humidity conditions that are different from the laboratory. Volume changes occurring prior to demolding at 24 h (or longer in the case of slow-hardening cements) are not captured during the test. The test allows different sized prisms to be used without compensation for the different surface-to-volume ratios and subsequent drying rates. A qualifying statement is imbedded within the test method that length change may be influenced by the size of the specimen, the demolding time of the specimen, and that specimens to be compared shall have the same dimensions and demolding time. Any specification limit based upon this method shall be applied to a specified specimen size and specimens that are to be directly compared with each other shall be subjected to the same conditions.

ASTM C 341 covers the determination of the length changes of drilled or sawed specimens of hydraulic-cement mortar and concrete due to causes other than externally applied forces and temperature changes [65]. It can be readily adapted, if desired, to studies of length change involving different schedules or environmental treatments than the standard procedures for sawed specimens of hydraulic-cement mortar and concrete due to various causes other than externally applied forces and temperature changes. Originally written as a standard test method, it was revised into a standard practice in 2003 due to the lack of applicable precision and bias data. The practice is essentially a modification of ASTM C 157 utilizing specimens that have either been cast for freeze-thaw testing or removed from field-applied concrete or mortar. A provision is made for application of gage studs to hardened specimens.

ASTM C 596 covers determination of the effect of portland cement on the drying shrinkage of a graded standard sand mortar subjected to stated conditions of temperature, relative humidity, and rate of evaporation in the environment [66]. In regard to significance and use of the method, it is stated that the drying shrinkage of mortar as determined by this method has a linear relationship to the drying shrinkage of concrete made with the same cement and exposed to the same drying conditions. Since drying shrinkage of concrete is greatly influenced by the aggregate content, aggregate stiffness, and water content, many researchers question the validity of extrapolating the data on mortar shrinkage to concrete shrinkage. For instance, Swayze [74] found it inconsistent to rely on the behavior of either neat pastes or rich mortars to predict the ultimate shrinkage of concrete, especially when the tests are concluded at early ages.

ASTM C 1581 covers the laboratory determination of the age at cracking and induced tensile stress characteristics of mortar or concrete specimens under restrained shrinkage. The procedure can be used to determine the effects of variations in the proportions and material properties of mortar or concrete on cracking due to both drying shrinkage and deformations caused by autogenous shrinkage and heat of hydration. This test method is not intended for expansive materials or concretes containing aggregates coarser than 3/4 in. (19 mm). During the test, a sample of freshly mixed mortar or concrete is compacted in a circular mold around an instrumented steel ring. The compressive strain developed in the steel ring caused by the restrained shrinkage of the mortar or concrete specimen is measured from the time of casting. Cracking of the test specimen is indicated by a sudden decrease in the steel ring strain. The age at cracking and the rate of tensile stress development in the test specimen are indicators of the material's resistance to cracking under restrained shrinkage. This test method is useful for determining the relative likelihood of early-age cracking of different cementitious mixtures and for aiding in the selection of cement-based materials that are less likely to crack under retrained shrinkage. Actual cracking tendency in service depends on many variables including type of structure, degree of restraint, rate of property development, construction and curing methods, and environmental conditions. This test method can be used to determine the relative effects of material variation on induced tensile stresses and cracking potential. For materials that have not cracked during the test, the rate of tensile stress development at the time the test is terminated provides a basis for comparison of the materials.

Expansive Cement Mortar and Concrete

ASTM C 827 affords a means for comparing the relative shrinkage or expansion of cementitious mixtures prior to final set [68]. It is particularly applicable to grouting, patching, and form-filling operations in which the objective is to completely fill a cavity or other defined space with a freshly mixed cementitious mixture that will continue to fill the same space at time of hardening. The test consists of filling a 100 to 300 mm high cylinder (depending upon aggregate size) with the freshly mixed cementitious material. A small ball selected to be 55 ± 5 % of the density of the mixture floats on the surface of the specimen and its image is magnified using a light source and lenses calibrated to a known magnification. As volume changes occur, the position of the ball moves relative to the height of the cylindrical specimen and the projected magnified image is read from a calibrated chart. Drops of oil may be used on the specimen surface to minimize the effects of bleed water evaporation.

The method affords a means for comparing the relative shrinkage or expansion of cementitious mixtures prior to setting. It would be appropriate to use this test method as a basis for prescribing mixtures having restricted or specified volume change before the mixture becomes hard. However, the specimen used in this test method is not completely unrestrained so that the measurements are primarily useful for comparative purposes rather than as absolute values and the degree of restraint to which the specimen is subjected varies with the viscosity and degree of hardening of the mixture.

ASTM C 806 covers the determination of length changes of expansive cement mortar while under restraint due to the development of internal forces resulting from hydration of the cement [67]. The test specimen is a 50 by 50 by 260-mm prism having a 250-mm gage length, which is restrained by using a standard restraining cage. The mix components of the mortar and the mixing procedure are specified. The specimens are removed from the molds at the age of 6 h and exposed to curing in lime-saturated water at 23.0 ± 1.7°C until an age of seven days. At the end of the water-curing period, the length change measurement is taken and percent expansion is calculated. ASTM Specification for Expansive Hydraulic Cement (C 845) requires the seven-day retrained expansion to range between 0.04–0.10 %.

ASTM C 878 covers the determination of the expansion of concrete made with shrinkage-compensating cement [69]. Since the potential for expansion under conditions of controlled restraint of concrete made with shrinkage-compensating cement cannot always be satisfactorily predicted from test mortars made in accordance with ASTM C 806, a need has been recognized for a test method in which concrete specimens are tested. ASTM C 878 can be adopted readily to studies of expansion involving differences in degree of restraint, cement composition, cement content, mixture proportions, or environmental treatments that vary from the standard procedures prescribed by this test method. The procedure calls for a standard restraining cage for a 76 by 76 by 254-mm concrete prism that is demolded at the age of 6 h, then water-cured until the age of seven days when the length change is measured.

ASTM C 940 determines the amount of expansion and accumulation of bleed water at the surface of freshly mixed hydraulic-cement grout used in the production of preplaced-aggregate (PA) concrete [70]. The method uses visual observation of the plastic volume change and bleeding in an 800 mL sample of grout confined in a 1000 mL graduated cylinder during the first 3 h after mixing.

ASTM C 1090 covers measurement of the changes in height of hydraulic-cement grout by the use of cast 3 by 6-in. cylinders [71]. The cylinders are covered by a restrained glass plate for the first 24 h after mixing and subsequently protected so that the tendency to change in height does not include evaporation so as to cause drying, uptake of moisture, carbonation, or exposure to temperatures outside the range of standard conditions. Measurements are performed using a depth micrometre to measure the distance between the grout surface and a reference micrometer bridge.

This test method is intended to provide a means of assessing the ability of a hydraulic-cement grout to retain a stable volume during the stipulated testing period of 28 days. If settlement shrinkage occurs, the grout will pull away from the glass plate during the first 24 h of the test. The test method is primarily intended for use in evaluating mixtures of materials intended to be used in producing “nonshrink grouts” for use either under machine bases or column bases, for bolt anchorages, and for similar applications. In these applications, the portion of the grout providing either the bearing support or bolt anchorage, or both, will set and harden under confinement.

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22

Thermal Properties

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Preface

IT IS AN HONOR TO REVIEW AND UPDATE THIS chapter on thermal properties, originally written in 1965 by L. J. Mitchell, a supervisory engineer with the Bureau of Reclamation, Denver, Colorado, and later updated by J. A. Rhodes, who retired in 1980 as chief of the Concrete Branch, Office Chief of Engineers. John Scanlon and James McDonald, both of whom retired from the Corps of Engineers, provided the last update.

Introduction

The thermal properties of hydraulic cement concrete, whether the concrete is massive or in thin sections, are the properties that are most ignored and the least understood by the general concrete engineering and construction industry. The thermal characteristics of concrete covered in this chapter are conductivity, expansion (or contraction), diffusivity, specific heat, and heat of hydration of cementitious materials. This last property is included because of its influence on the thermal and physical behavior of concrete.

One of the earliest and most comprehensive studies of the fundamental thermal properties of concrete was carried out by the U.S. Bureau of Reclamation in conjunction with the design and construction of Boulder (Hoover) Dam between 1930 and 1935. The investigations were stimulated by the realization that in massive structures heat generated by hydration of cement could be responsible for volumetric changes that would affect the integrity of a structure, and that this heat, under ordinary circumstances, could take as much as a century or more to dissipate. Results are given in the Boulder Canyon Reports [1].

As with most construction materials, there is a direct relationship between a change in temperature of concrete and its change in length or volume. This fact has been long recognized for such structures as highways, bridges, walls, and buildings. Only at very high and very low temperatures do the expansion characteristics vary from those under normal conditions. The favorable thermal insulation characteristic of normal-weight concrete, and the even more favorable properties of lightweight concretes have been used effectively in building construction and for other applications in which resistance to steady-state heat flow is needed. The unique ability of concrete to dampen annual and daily ambient temperature variations (unsteady state) and to store or release heat over significant time periods has been utilized by designers to control volume changes and consequent concrete cracking.

Thermal Conductivity

Definition and Units

Thermal conductivity, a measure of the ability of a material to conduct heat, is defined as the ratio of the rate of heat flow to the temperature gradient. In normal metric use, it can be considered to be the number of kilocalories passing between opposite faces of a 1-m cube per unit of time when the temperature difference is 1°C. An alternate set of dimensions is joules per second · square meter · degree Celsius per meter. By dimensional manipulation and substitution, the approved SI (Systéme International) units are obtained, watts per meter · Kelvin, thus keeping the unit of time at 1 s. In U.S. customary and British units, conductivity is frequently expressed in Btu per hour · square foot · degree Fahrenheit per inch. Values of conductivity in these units may be converted to the SI units by multiplying by 0.1441314. Typically, engineering disciplines frequently express temperature gradients, areas, and time in units of measure most useful to them, and users of conductivity values are cautioned to assure that compatibility and consistency exist, both within and between systems of units [2].

Parameters and Values

Three principal conditions (water content, density, and temperature) significantly influence the thermal conductivity of a specific concrete. The mineralogical character of the aggregates largely determines the thermal conductivity for normal-weight concrete, while with lightweight concretes air voids and moisture content mask the effect of aggregate type. Other factors of slight, or negligible, importance in their effect on conductivity are cement type and content, entrained air, water/cement (w/c), and age.

Neat cement pastes, with w/c ranging from 0.3 to 0.6 and ages from three days to one year [3], exhibit a fairly constant thermal conductivity value of 1.2 W/m · K (8.0 Btu · in./h · ft^2 · °F) at normal air temperatures and in a moist ambient condition. Thermal conductivity measured under reduced ambient moisture conditions has little meaning because the specimens suffer extensive cracking because of drying.

The amount of free water in concrete, regardless of density, has a major influence on the thermal conductivity. While water is a relatively poor conductor of heat as compared to rock, its

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TABLE 1—Thermal Conductivity of Water

Water Temperature		Conductivity	
°C	°F	W/m · K	Btu · in./h · ft^2 · °F
20	68	0.59	4.1
0	32	0.56	3.9
−18	0	2.3	16.0
−59	−75	2.6	18.0
−101	−150	3.3	23.0
−157	−250	5.2	36.0

thermal conductivity as shown in Table 1 is many times that of air, which it replaces in concrete [3]. Thermal conductivity of concrete varies directly with moisture content [4,5]. The effect of moisture on thermal conductivity values from oven-dry to a moist condition (not necessarily saturated) is given in Table 2.

For heavyweight, normal-weight, and structural lightweight concretes, the mineralogical characteristics of the aggregate markedly affect the conductivity of the concrete, as shown in Tables 3 and 4. Insulating lightweight concretes, those with densities less than 960 kg/m^3 (60 lb/ft^3), may have been aerated (foamed) or may contain a very lightweight porous aggregate. Thermal conductivities [6,7] given in Table 5 are for air-dry or low moisture contents.

Over a temperature range from room temperature to −157°C (−250°F), the thermal conductivity of oven-dry, normal-weight and lightweight concrete is essentially constant [3,8]. For moist normal-weight concrete, the conductivity at −157°C (−250°F) has been found to be about 50% greater than at normal temperatures [3]. At elevated temperatures, up to about 750°C (1380°F), conductivities of cement pastes, mortars, and concrete decrease in a consistently uniform manner. This decrease has been attributed to disruption of the intercrystalline bonds in the aggregate caused by excessive thermal expansion [9]. Conductivity values at about 400°C (7500°F) are given in Table 6 [5]. Above temperatures of 400°C (70°F), gradual disintegration of the fully hydrated cement paste occurs, resulting in further decreases in conductivity [9].

Test Methods

Values for the thermal conductivity of concrete are usually calculated from the diffusivity and specific heat because they are easier to measure. The Corps of Engineers [10] Method for Calculation of Thermal Conductivity of Concrete (CRD-C 44) is suitable for calculating the thermal conductivity of concrete from results of tests for diffusivity and specific heat. However, the conductivity can be determined directly with any of the steady-state or transient (nonsteady-state) test methods described in the following.

The test method for thermal conductivity developed for the Boulder Canyon Project [1] used 200-mm (8-in.) diameter cylinders subjected to steady heat flow conditions. Use of water as the heating and cooling mediums limited the results to a specific portion of the temperature range between the freezing and boiling points of water and to saturated or near-saturated concrete.

The discontinued ASTM Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus (C 177) covered the achievement and measurement of steady-state heat flux through flat-slab specimens. This test method was applicable to the measurement of a wide variety of specimens, ranging from opaque solids to porous or transparent materials, and a wide range of environmental conditions.

TABLE 2—Typical Variations in Thermal Conductivity with Moisture at Normal Temperatures

	Conductivity	
Moisture Condition	W/m · K	Btu · in./h · ft^2 · °F
	Limestone Concrete	
Moist	2.2	15.0
50% relative humidity	1.7	11.0
Dry	1.4	10.0
	Sandstone Concrete	
Moist	2.9	20.0
50% relative humidity	2.2	15.0
Dry	1.4	10.0
	Quartz Gravel Concrete	
Moist	3.3	23.0
50% relative humidity	2.7	19.0
Dry	2.3	16.0
	Expanded Shale Concrete	
Moist	0.85	5.9
50% relative humidity	0.79	5.5
Dry	0.62	4.3

TABLE 3—Effect of Aggregate Type on Conductivity of Dry Concrete at Normal Temperatures

	Dry Density		Conductivity	
Aggregate Type	lb/ft^3	kg/m^3	Btu · in./h · ft^2 · °F	W/m · K
Hematite	179	2870	18	2.6
Marble	143	2290	12	1.7
Sandstone	120	1920	10	1.4
Limestone	126	2020	10	1.4
Dolerite	136	2180	8.6	1.2
Barite	180	2880	8.5	1.2
Expanded shale	89	1430	4.3	0.62
Expanded slag	103	1650	3.2	0.46
Expanded slag	60	960	1.5	0.22

Another discontinued test method, ASTM Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus (C 518) was a comparative method of measurement since specimens of known thermal transmission properties must be used to calibrate the apparatus. The method was used at ambient conditions of 10 to 40°C (50 to 104°F) with flat slab thicknesses up to approximately 250 mm (10 in.).

The Corps of Engineers [10] Method of Test for Thermal Conductivity of Lightweight Insulating Concrete (CRD-C 45) measures conductivity directly under steady-state conditions. Nominal specimen thickness is 25 mm (1 in.), and temperature differential through the oven-dry specimen is from 32 to 60°C (90 to 140°F).

For experimental work, Campbell-Allen and Thorne [5] developed a hollow-cylinder, steady-state system for measuring conductivity at temperatures up to 200°C (392°F). The specimen, at a selected moisture content, was coated with mercury on all surfaces to retain moisture and electrically heated internally and cooled externally. Its acceptability or modifications are not known.

Lentz and Monfore, with the Portland Cement Association [8], developed a transient hot-wire method for determining thermal conductivity of concrete or rock. A thermocouple cast along the axis of a prism (or in the center of a split and lapped specimen) measures temperature response to a measured alternating current input. There are no moisture or density restrictions, and measurements are completed within a few minutes time.

TABLE 4—Effect of Aggregate Type on Conductivity of Moist Concrete at Normal Temperatures

	Moist Density		Conductivity	
Aggregate Type	lb/ft^3	kg/m^3	Btu · in./h · ft^2 · °F	W/m · K
Hematite	190	3040	28	4.1
Quartzite	150	2400	28	4.1
Quartzite	152	2440	24	3.5
Dolomite	156	2500	23	3.3
Quartzite	...	...	23	3.3
Limestone	153	2450	22	3.2
Quartzite	147	2350	21	3.1
Sandstone	133	2130	20	2.9
Sandstone	150	2400	20	2.9
Granite	151	2420	18	2.6
Limestone	151	2420	18	2.6
Marble	152	2440	15	2.2
Limestone	152	2440	15	2.2
Basalt	157	2520	14	2.0
Rhyolite	146	2340	14	2.0
Barite	190	3040	14	2.0
Dolerite	147	2350	14	2.0
Basalt	158	2350	13	1.9
Expanded shale	99	1590	5.9	0.85

TABLE 5—Thermal Conductivity of Insulating Concrete

Density		Thermal Conductivity	
kg/m³	lb/ft³	W/m · K	Btu · in./h · ft² · °F
		Aerated	
320	20	0.07	0.5
480	30	0.11	0.75
640	40	0.14	1.0
800	50	0.20	1.4
960	60	0.26	1.8
		Vermiculite	
400	25	0.10	0.72
		Expanded Clay	
825	52	0.17	1.2

Specific Heat

Definition and Units

The rigorous definition of specific heat is the ratio of the amount of heat required to raise a unit mass of the material 1° to the amount of heat required to raise the same mass of water 1°. In those systems of units in which the heat capacity of water is 1.0 (either Btu/lb · °F or cal/g · °C), the specific heat (and heat capacity) values are the same. In SI units, specific heat is expressed in joules/kilogram · K, which is obtained from cal/g · °C by multiplying by 4.1868×10^{3}.

Parameters and Values

The mineralogical differences among such aggregates as generally used have little effect on specific heat of the concrete, with values at normal temperatures ranging between 0.22 and 0.24 cal/g · °C (Btu/lb · °F). Increased water content, according to the Boulder Canyon studies [1], tended to increase the specific heat of the concrete. The actual values found were 0.22 cal/g · °C at 4 % of mixing water by weight of concrete and 0.24 cal/g · °C at 7 % mixing water by weight and higher.

Specific heat varies directly with concrete temperatures, as indicated in Table 7. These values represent mass concrete mixtures and several aggregate types [11]. Specific heats of most rock types tend to increase up to at least 400°C (750°F), and hydrated cement pastes up to 1000°C (1830°F). Investigations by Harmathy and Allen [12] indicate the specific heat of expanded aggregate lightweight concrete differs little from that of normal-weight concrete at ordinary temperatures and also increases up to at least 600°C (1110°F).

TABLE 6—Conductivity Values

	W/m · K	Btu · in./h · ft² · °F
Cement paste	0.56	3.9
1:3 mortar	0.75	5.2
Sandstone concrete	1.6	10.9
Ilmenite concrete	1.2	8.2

TABLE 7—Typical Specific Heats of Concrete

Temperature		Specific Heat	
°C	°F	J/kg · K	cal/g · °C
10	50	917	0.219
38	100	971	0.232
66	150	1038	0.248

Testing Methods

Some type of calorimeter apparatus is employed in all procedures to measure the specific heat (heat capacity) of hardened concrete, aggregates, cement, and cement pastes. The most suitable procedure is the Corps of Engineers [10] Method of Test for Specific Heat of Aggregates, Concrete, and Other Materials (CRD-C 124). In this procedure, approximately 1 kg (2 lb) of the material, with no particles larger than 25 mm (1 in.) in size, is tested. The ASTM Test Method for Specific Heat of Liquids and Solids (D 2766) is applicable to materials, such as cement and cement pastes, which would react with water. The test requires smaller samples (up to 100 g in weight) and provides for selection of nonreactive cooling media whose physical or chemical properties differ from those of water.

The Bureau of Reclamation uses a procedure described as Procedure No. 4907, Specific Heat of Aggregates, Concrete, and Other Materials, that can be found in the Bureau's Concrete Manual [13].

Thermal Diffusivity

Definition and Units

The diffusivity property is described as a measure of the facility with which temperature changes take place within a mass of material. Thermal diffusivity is defined numerically as thermal conductivity divided by the product of specific heat and density, or $\alpha = k/(c \cdot p)$. Thus, diffusivity results from the consolidation of three other properties that appear in differential equations that define heat flow and heat storage under unsteady-state conditions.

When calculating diffusivity from its parts, one must take care to assure that the dimensional units of the three constituents are compatible. These are shown in Table 8.

In approved SI base units, diffusivity values are usually small. The following formulas are used to convert British to SI units.

$$\text{ft}^2/\text{h} \times 2.58064 \times 10^{-5} = \text{m}^2/\text{s}$$

Parameters and Values

Those variables and conditions that influence thermal conductivity, specific heat, and density also affect thermal diffusivity. Both thermal conductivity and density are sensitive to moisture content of the concrete, and derived thermal diffusivity values should be based on conductivities and densities that

TABLE 8—Diffusivity Calculations

British system units	
Conductivity	Btu/h · ft^2 · °F per ft
Specific heat	Btu/lb · °F
Density	lb/ft^3
Thus α	ft^2/h
SI units	
Conductivity	W/m · K
Specific heat	J/kg · K
Density	kg/m^3
Thus α	m^2/s, where W = J/s

correspond to the condition of the concrete in service. Within the same system of units, higher diffusivity values are associated with concrete that heats or cools most easily.

Thermal diffusivity of concrete is determined largely by the mineralogical characteristics of the coarse aggregate. Since specific heat varies directly with temperature, diffusivity values for a specific concrete will decrease as the concrete temperature increases.

Typical diffusivity values in Table 9, taken from Refs 1, 11, and 12, show the general range that can be expected for normal-weight and structural lightweight concrete. The Bureau of Reclamation [1] reported diffusivities for neat cement pastes ranging from 0.0012 to 0.0016 m^2/h (0.013 to 0.017 ft^2/h) at and somewhat above normal room temperatures.

Tests Methods

The Corps of Engineers [10] Method of Test for Thermal Diffusivity of Concrete (CRD-C 36) outlines a procedure for determining the thermal diffusivity of partially saturated concrete. This method is directly applicable to a 152- by 305-mm (6- by 12-in.) cylinder; however, specimens of other sizes and shapes may be accommodated. A test specimen is heated in boiling water and then transferred to a bath of running cold water. The thermal diffusivity of the concrete is determined from the relationship between time and the temperature differential between the interior and the surface of the specimen as it cools.

The Corps of Engineers [10] Method of Test for Thermal Diffusivity of Mass Concrete (CRD-C 37) determines diffusivity with large moist concrete cube specimens. The 0.23-m^3 (8-ft^3) test specimen is heated by hot air until the specimen is in thermal equilibrium at 38°C (100°F); then a water spray cools the surfaces. The cooling procedure creates a moisture gradient from the surface of the specimen inward, and the concrete mass is considered to be "moist" rather than either "dry" or "saturated." While the results of both tests are applicable only to moderate temperature ranges, they are quite reliable.

TABLE 9—Typical Thermal Diffusivity Values

Type of Aggregate in Concrete	Thermal Diffusivity			
	m^2/h	ft^2/h	ft^2/day[a]	m^2/day
Quartz	0.0079	0.085	2.04	0.190
Quartzite	0.0061	0.065	1.56	0.146
Limestone	0.0055	0.059	1.42	0.132
Basalt	0.0025	0.027	0.65	0.060
Expanded shale	0.0015	0.016	0.38	0.036

[a] Convenient when computing heat flow in large structures.

Another standard procedure (USBR Procedure 4909) for determining thermal diffusivity can be found in Ref 13.

One practice, not necessarily always preferable, is to calculate thermal diffusivity from conductivity, specific heat, and density as determined from laboratory tests. Test methods for conductivity are essentially limited to an oven-dry condition, and diffusivity values are applicable to dry environments only.

Thermal Expansion

Definition and Units

As with most construction materials, concrete has a positive coefficient of thermal expansion, which can be defined as the change in linear dimension per unit length divided by the temperature change. While a general value of 10 millionths/°C (5.5 millionths/°F) has been widely used, change in length is a complex process reflecting principally materials, moisture, and temperature individually and together. The actual thermal expansion is the net result of two actions occurring simultaneously. The first is a normal expansion typical of anhydrous solids. Second, there is a hygrothermal expansion or contraction associated with the movement of internal moisture from capillaries or from gel pores.

Parameters and Values

Since aggregate comprises from 80 to 85 % of concrete, its thermal properties greatly influence the behavior of the concrete. Thermal expansion can vary widely among aggregates because of differences in mineralogical content. Quartzites and other siliceous aggregates exhibit high thermal expansion properties, and concrete containing such aggregates frequently show values up to $13 \times 10^{-6}/^\circ C$ ($7.2 \times 10^{-6}/^\circ F$) at normal temperatures. Some limestone aggregate concretes exhibit expansion values of $5.6 \times 10^{-6}/^\circ C$ ($3.1 \times 10^{-6}/^\circ F$) for comparable conditions, and other natural rock types usually have values between these two.

Cement paste occupies only about 15 to 20 % of the concrete volume, but its expansion coefficient ranges from 9 to $22 \times 10^{-6}/^\circ C$ (5 to$12 \times 10^{-6}/^\circ F$), which may be several times that of the aggregate itself. In addition, most of the capillary water and essentially all of the absorbed water is contained within the gel pore system, which makes the paste sensitive to water movements caused by temperature changes. Powers [14] showed one aspect of the significance of the moisture content of paste specimens, in which the expansion coefficients for oven-dry and saturated conditions were half the maximum expansion coefficient of $11 \times 10^{-6}/^\circ C$ ($6.1 \times 10^{-6}/^\circ F$) that occurred at 75 % relative humidity. A similar relationship was found for concrete, except that the dry and saturated condition values were from 65 to 80 % of the maximum value, which occurred at about 60 % relative humidity. Generally, the coefficient of expansion increases with decrease in the w/c.

Some sources report a slight increase in the coefficient of thermal expansion with age up to three months but a general decreasing trend thereafter. For concretes used in Ilha Solteira Dam in Brazil, the thermal expansion coefficient was found to increase significantly with age when the aggregate was all quartzite and less when only the fine aggregate was quartzite (Table 10).

TABLE 10—Effect of Age on Thermal Expansion[a] (Units Are Millionths/K) (Millionths/°F)

Approximate Age, days	Quartzite Concrete[b]	Basalt Concrete[c]	Units are millionths/K (°F) Quartzite Mortar[d]
4	12.9(7.14)	10.2(5.64)	12.2(6.75)
9	12.9(7.18)	10.2(5.67)	12.8(7.12)
17	13.2(7.34)	10.2(5.65)	14.0(7.75)
30	13.4(7.42)	10.7(5.96)	15.0(8.33)
48	13.5(7.51)	10.8(5.98)	15.6(8.69)
62	13.7(7.62)	10.6(5.89)	16.0(8.90)

[a] Results are from University of California (Berkeley) tests with Ilha Solteria (Brazil) materials.
[b] All natural quartzite aggregate.
[c] Basalt coarse aggregate and quartzite sand.
[d] Quartzite natural sand.

For a given concrete mixture, the magnitude of thermal expansion or contraction at temperatures from freezing to about 65°C (150°F) is the same for each unit temperature change, so the coefficient of thermal expansion is a constant figure. Below the freezing point of water, the length changes are smaller per unit of temperature, so the thermal expansion coefficient decreases. The slowing rate of contraction at low temperatures results in continued contraction of the concrete (and of the ice already formed) and an opposite expansion resulting from the formation of additional ice. Berwanger and Sarker [15] found the coefficient decreased from 7.7 to 5.1 millionths/°C (4.3 to 2.8 millionths/°F) for temperatures above and below 0°C (32°F), respectively, and Monfore and Lentz [16] showed a similar typical decrease from 9.4 to 6.8 millionths/°C(5.2 to 3.8 millionths/°F). There is some evidence that the coefficient continues to decrease with further decreases in temperatures.

As stated previously, the expansion of concrete exposed to increasing temperature is the net result of the inherent thermal expansion property of the aggregate and a complex hygrothermal volume change of the cement paste. Up to about 100°C (212°F), the paste has achieved its natural expansion, and at higher temperatures starts to shrink [17], continuing to do so up to about 500 or 600°C (930 or 1100°F). At this level, only the original dry ingredients remain. For calcareous aggregate concrete, Philleo [17] reported a thermal expansion coefficient of 8.5 millionths/°C (4.7 millionths/°F) below 260°C (500°F), and 22.5 millionths/°C (12.5 millionths/°F) above 425°C (800°F). For expanded shale aggregate concrete, the values were 5.0 millionths/°C (2.8 millionths/°F) and 8.8 millionths/°C (4.9 millionths/°F) for similar temperature levels.

Over a number of years, the Bureau of Reclamation and Corps of Engineers have conducted thermal expansion coefficient tests of specimens composed of concrete mixtures that have moderate to low cement factors and large-size aggregates. The aggregates, from the actual job sources, are frequently complex in terms of type and mineralogical content; and the expansion coefficients do not lend themselves to unique groupings by rock type. Some typical results are listed in Table 11. The values therein are applicable for an average temperature of about 38°C (100°F).

Test Methods

The significant effect that moisture and temperature have on the behavior of concrete and the wide variety of environmental conditions to which concrete is exposed have precluded development of a standard test method for general use. Physical measurement of specimen length change with

TABLE 11—Thermal Expansion Coefficients for Mass Concrete

Dam Name	Aggregate Type	Coefficient of Thermal Expansion millionths/K	Coefficient of Thermal Expansion millionths/°F
Hoover	limestone and granite	9.5	5.3
Hungry Horse	sandstone	11.2	6.2
Grand Coulee	basalt	7.9	4.4
Table Rock	limestone and chert	7.6	4.2
Greers Ferry	quartz	12.1	6.7
Dworshak	granite-gneiss	9.9	5.5
Libby	quartzite and argillite	11.0	6.1
Jupia (Brazil)	quartzite	13.6	7.5

the necessary accuracy is not a major obstacle, but conditioning of the specimen and control of the conditions during the test may be difficult. Many test procedures, some quite sophisticated, have been developed in conjunction with research studies [8,17].

The Corps of Engineers [10] Test Method for Coefficient of Linear Thermal Expansion for Concrete (CRD-C 39) determines the thermal coefficient of concrete by measuring length changes of a specimen alternately immersed in 5 and 40°C (40 and 140°F) water baths. A length comparator similar to that described in ASTM Specification for Apparatus for Use in Measurement of Length Change of Hardened Cement Paste, Mortar, and Concrete (C 490) is used to measure length changes. When laboratory molded specimens are used, strain meters embedded along the longitudinal axes of 152- by 405-mm (6- by 16-in.) cylinders are used in lieu of the length comparator. Because the thermal coefficient of concrete varies with moisture condition, being at a minimum when saturated or oven dry and at a maximum at about 70 % saturated, it is important to select the relevant moisture condition for the tests to be made.

The Bureau of Reclamation has a procedure (No. 4910) that can be found in Ref 13 for determining the linear thermal expansion for concrete.

Heat of Hydration

Definition and Units

When water is added to cement, the reaction is exothermic, and a considerable amount of heat is generated over an extended period of time. The heat liberated up to a specific time or age is measured in calories per unit mass of cement (cal/g) or kilojoules per kilogram (kj/kg). When the amount of this heat and the heat capacity of the paste, mortar, or concrete are known, the resulting temperature rise can be calculated, assuming no heat loss to the surroundings.

Parameters and Values

The hydration reactions of portland cement have been studied and reported by many authors for many years. All the compounds present in cement are anhydrous and, when in contact with water, react at various rates and at varying times. There is general agreement that the tricalcium aluminate (C_3A) is the largest single compound contributor to the evolved heat, followed by tricalcium silicate (C_3S) and tetracalcium aluminoferrite (C_4AF) with about equal contributions, and finally dicalcium silicate (C_2S). Approximate contributions of the four principal calculated compounds in 0.40 w/c cement pastes cured at 21°C (70°F) are given in Table 12. Although the data apparently indicate progressive increases in the total heats generated by each compound, values at intermediate ages for both C_3A and C_4AF deviate from this pattern by generating a higher proportion of heat after one year. Possible causes and significance of the regression characteristics are discussed in Ref 18. When water is first brought into contact with portland cement, there is a very rapid and very brief heat evolution with the evolution of heat reaching possibly a rate of 1 cal/g/min (4.2 joules/g/min). There then occurs a 1- or 2-h delay, followed by a gradually increasing hydration rate to about 6 or 8 h, and a slow decline in rate thereafter. Relative proportions of the four major compounds, other minor compounds, alkalies, and gypsum content cause variations in both early- and later-age hydration rates. The nature and interrelationships of the chemical reactions are complex, variable, and sometimes inconsistent or controversial (the reader is referred to Lea [6] or other comparable texts for a comprehensive presentation on the chemistry of cements).

TABLE 12—Typical Compound Contribution to Heat of Hydration [18]

Compound	Heat Evolved in cal/g		
	3 Day	1 Year	13 Year
C_3S	58	117	122
C_2S	12	54	59
C_3A	212	279	324
C_4AF	69	90	102

As with most chemical reactions, the rate of cement hydration increases with temperature. The accelerating effect of high curing temperatures is limited to early ages, and subsequently the hydration process slows to a rate less than that corresponding to normal placement and curing temperatures. Verbeck [19] suggests this slowing is the result of a dense hydration product created by high temperatures at early ages surrounding the cement grains, thereby substantially retarding subsequent hydration. This degradation in hydration is generally true for temperatures above 24°C (75°F) and becomes significant at 52°C (125°F) and above. At and below 0°C (32°F), a sharp drop in rate of hydration occurs, and between −10 and −15°C (14 and 5°F) hydration ceases.

The w/c in pastes has a significant influence on the amount of heat generated at ages of three days and greater. The differences over the range 0.4 to 0.8 w/c are greatest at intermediate ages but continue to be evident at up to six years. According to Verbeck [19], the hydration product steadily increases in volume, filling the capillary void space in the paste. If the capillary void space is small (low w/c), the available space will become filled completely with hydration products and hydration of the remaining cement will cease. Thus, a high w/c will tend to result in more complete hydration and in more heat developed. Data reported by Verbeck [19] show that an increase in w/c from 0.4 to 0.8 had only a slight effect on heat liberation for Type IV (low-heat) cements but produced an increase of 10.8 cal/g (4520 J/kg), or 14 %, for Type III (high-early) at three days of age. The increases in heat of hydration caused by an increase in w/c from 0.4 to 0.8 ranged from 11.4 cal/g (4770 J/kg) for Type II (moderate-heat) cement to 15.9 cal/g (6660 J/kg) for Type III at one year of age.

Prior to about 1935, the fineness of a cement was a major factor in the rate and amount of heat developed during hydration. The finer cements presented much more surface area to be wetted and, therefore, caused more rapid and complete hydration. More recently all cements are so finely ground that the moderate variation in fineness of different cements is no longer an important factor in cement hydration. However, high-early strength cements are finer than other types to the degree that their extra fineness works with their more active chemical compounds to produce earlier strength and more rapid release of heat.

Pozzolans are defined in ASTM Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete (C 618) as siliceous or siliceous and aluminous materials that in themselves possess little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties. Raw or calcined natural pozzolans (sometimes used to replace a portion of cement in mass concrete) vary widely in their composition but will reduce the total heat evolved by a value up to one half of the replacement percentage figure [20].

Fly ash, also classified as a pozzolan, may range in fineness before processing from below to well above that of portland cement. When used in concrete, a minimum fineness limit considerably above the portland cement value is imposed. The reaction between the siliceous glass in the fly ash and the lime in the cement is particularly sensitive to heat, and adiabatic curing of concrete containing a fly ash replacement will serve to increase substantially the chemical activity of the fly ash component.

In a study of portland blast-furnace slag cements, Klieger and Isberner [21] found no consistent difference in heat of hydration up to three days of age between Type I and Type IS cements, and, at later ages up to one year, the Type IS cements exhibited slightly to moderately lower heat generation characteristics.

The term chemical admixtures, as is used here, refers to materials added in relatively small amounts to mortar or concrete during mixing to modify some characteristic of the product. Generally, accelerating, retarding, and, to some extent, water-reducing admixtures will affect the rate of hydration of the cement, but the processes are not well delineated and will differ significantly with different cements and proprietary products. There is little if any evidence that air-entraining admixtures influence either the rate or the total amount of heat generated for a specific cement or cement blend.

Test Methods

The most widely used method for determining the heat of hydration of a hydraulic cement over long periods of time is by heat of solution, ASTM Test Method for Heat of Hydration of Hydraulic Cement (C 186). In this procedure, the heat of hydration of a hydraulic cement is determined by measuring the heat of solution of the dry cement and the heat of solution of a separate portion of the cement that has been partially hydrated for 7 and 28 days, the difference between these values being the heat of hydration for the respective hydrating period. The procedure requires several corrections and Lea [6] discusses a number of possible errors. Most significant to the user, other than laboratory techniques, is that the storage temperature is standardized at 23°C (73°F), which is usually not representative of the environment experienced in a concrete construction. A standard paste w/c of 0.40 is required, but this ratio will result in an understatement of the heat generated where higher ratios are encountered in construction. The procedure is not recommended for pozzolan blends nor for slag cements in which a portion of the test sample usually remains insoluble at the end of the test procedure.

The Carlson-Forbrich vane conduction calorimeter, developed prior to 1940, determined the amount of heat developed by a 0.40 w/c cement paste by measuring the rate of heat flow from the paste receptacle through cooling vanes into a water bath. Because of difficulties in controlling extraneous heat transfer, the test period usually was limited to three days. The method is sensitive to the initial starting temperature and is neither adiabatic nor isothermal.

Monfore and Ost [22] developed a refined calorimeter for measuring early rates of heat liberation that ideally is suited for laboratory research. In this calorimeter, the temperature rise of a sample of hydrating cement weighing up to 8 g (0.3 oz) can be held to less than 0.5°C (0.9°F), thus producing nearly isothermal conditions at any temperature level. The test usually is limited to a three-day duration, but the results include the immediate heat of hydration (0 to 1 h). The method is well suited to determining the effects of additions and admixtures to the cements, as well as properties of specific cement compounds or other similar materials.

The Corps of Engineers [10] Method of Test for Temperature Rise in Concrete (CRD-C 38) covers a procedure for determining the temperature rise in concrete under adiabatic conditions primarily due to heat liberated on hydration of cement. The test specimen is a 762- by 762-mm (30- by 30-in.) cylinder that is sealed, insulated, and maintained in a temperature environment corresponding to its own temperature history for a period of 28 days. The advantages of this method are: (a) cement-pozzolan blends may be used, (b) the aggregates proposed for use in the prototype structure may be incorporated in the test, and (c) the temperature rise approximates the nearly adiabatic conditions existing in the interior of a massive concrete structure. A minor difficulty is maintaining an adequate insulation condition at the later stages when cement hydration rate is very low.

The Bureau of Reclamation uses Procedure No. 4911 for determining temperature rise of concrete; this procedure can be found in Ref 13. In this procedure, temperature rise tests are conducted on 318-kg (700-lb) concrete specimens sealed in 546- by 546-mm (21 1/2- by 21 1/2-in.) cylindrical metal containers and placed in a calorimeter chamber. The temperature of the air within the chamber is maintained at the same temperature as the specimen by automatic control equipment, which keeps an electrical resistance thermometer exposed to the chamber air in balance with a resistance thermometer in a well extending to the center of the concrete specimen. As a result, any heat generated by the cement or pozzolan or both results in a temperature rise in the concrete and a corresponding rise in chamber temperature.

Significance of Thermal Properties

All concrete elements and structures are subject to volume change [24]. These volume changes may be due to heat generation and subsequent cooling, shrinkage, creep or stress relaxation, or other mechanisms. When these changes are restrained, either by internal or external forces, tensile, compressive, or flexural strains will develop. The magnitude of these strains is determined primarily by the coefficient of expansion and the amount of temperature change. Cracking occurs when the magnitude of these strains exceeds the tensile strain capacity of the concrete.

The design of concrete structures, regardless of size or purpose, must include provisions to accommodate restrained volume changes resulting from heat generated internally from the hydration of portland cement and other cementitious products and from heat added by environmental factors. Such designs often include considerations for the frequency and width of joints to allow movement. Where internal heat can be significant, measures are implemented to minimize the amount of heat generated, lower the initial temperatures, or lower the generated temperatures. The ultimate volume change can be conceptualized by the following simplified expression:

$$\Delta V = [\,(Ti + Tad - Tf) + Tenv\,] \times K \times CTE \qquad (1)$$

where

ΔV = volume change of the concrete, measured as linear length change

Tf = the final stable temperature of the concrete

Ti = the initial placing temperature of the concrete

Tad = the adiabatic temperature rise of the concrete

Tenv = the heat added or subtracted from the concrete due to environmental conditions

K = restraint coefficient(s) that factor the effect of structure geometry and foundation elasticity

CTE = the coefficient of thermal expansion

Heat Generation and Temperature Rise

The importance of heat generation and disposal of that heat has long been recognized by designers of large dams. This is also true for large foundation mats, bridge piers, and other relatively large placements. Upon final completion, these structures should be monolithic and in intimate contact with the foundation in order to achieve the design stress distribution and stability. Cracks that disrupt the stress pattern and decrease stability are caused principally by thermal tensile stress created by concrete volume changes associated with a temperature decline as heat is dissipated. While several design and construction practices are available to deal with temperature changes that tend to occur, restricting the amount of heat that causes the temperature rise is a fundamental and certain scheme for mitigating the thermal stress problem.

The heat resulting from the exothermic water-cement reaction over a given time interval is expressed in terms of temperature rise (Tad in Eq 1) when the specific heat value for the paste, mortar, or concrete mixture is known. While the range of values for specific heat of concrete is relatively narrow, the heat generation characteristics of the cementitious materials available for use in concrete are quite broad. When positive control over temperature rise is desired, upper limits on the heat of hydration of the cement may be imposed through ASTM Specification for Portland Cement (C 150) and ASTM Specification for Blended Hydraulic Cements (C 595). Specification limits for these cementitious materials and typical ranges for Types I and III not subject to specification restriction are indicated in Table 13, with values in calories per gram.

In lieu of established specification products, partial replacement of the cement in a mixture by natural or processed pozzolans or one of the several types of fly ashes will reduce significantly both the hydration rate and the total amount of heat generated. The use of ground granulated blast-furnace slag is becoming popular in certain regions to replace or supplement portland cement in mixtures to reduce heat from hydration. Aggregate adjustments and admixture usage are other means to attain required performance with less cementitious materials, and consequently less heat generated.

The practice of precooling concrete materials to lower the initial temperature of the concrete at the time of placing is resorted to frequently in massive concrete construction. This practice reduces the peak temperature that subsequently will be attained. A maximum placing temperature of 10°C (50°F), which is specified frequently for large concrete dams, is usually well below the ambient air temperature during the normal construction season. This low placing temperature reduces the rate of cement hydration initially, but during the period the concrete is at a temperature below air temperature, heat is gained from the surrounding air. However, this period of time is short as compared to the time the concrete is warmer than the surroundings.

Waugh and Rhodes [23] have listed experiences at several large mass concrete dams constructed by the Bureau of Reclamation, the Corps of Engineers, and the Tennessee Valley Authority. The favorable trend toward lower peak concrete temperatures (Ti + Tad in Eq 1) has allowed the use of larger monolith lengths without serious consequences. The advent of roller-compacted concrete in mass concrete construction has resulted in mixtures with significantly lower proportions of cementitious materials, higher pozzolan to cement ratios, and consequently lower temperature rise.

Pier footings and heavily reinforced raft foundation slabs up to 2 m (7 ft) thick and totaling 2000 m^3 (2616 yd^3) with cement contents of 400 kg/m^3 (675 lb/yd^3) have been successfully placed with no cracking detected [5]. Peak interior concrete temperatures about 50°C (90°F) above initial concrete placing temperature required exterior insulation to control gradients and cooling rates for prevention of thermal cracking.

Conventional concrete structures are routinely constructed using mixtures with cement contents in excess of 350 kg/m^3 (590 lb/yd^3). Peak temperatures in these concrete mixtures can be as much as 36°C (65°F) above the initial placing temperature depending on the member geometry and environmental conditions.

Heat Flow

The rate at which heat flows into, through, or out of a concrete structure is governed by the thermal conductivity of the concrete. The ease or difficulty with which the concrete undergoes temperature change, as a result of heat loss or gain, depends also on the heat capacity and is measured by the thermal diffusivity of the concrete.

Heat of hydration (Tad), discussed previously, is one source of heat for a concrete structure. Another common source is heat added or removed from the structure due to environmental conditions (Tenv in Eq 1). This is usually the result of high or low ambient air temperatures. Precipitation, humidity, wind, and solar gain are additional environmental factors that contribute to heat flow into or out of the structure. Cooling of concrete by embedded cooling pipes or water

TABLE 13—Specification Limits for Types I and II

	7 Days	28 Days
Type I (C 150)	63 to 88	83 to 109
Type II (C 150)	70	80
Type III (C 150)	84 to 95	101 to 107
Type IV (C 150)	60	70
Type IS (C 595)	70	80
Type IP (C 595)	70	80
Type P (C 595)	60	70

immersion and spraying are means to force an increased environmental heat flow change.

Concrete in massive structures is usually placed in horizontal lifts from 0.75 to 2.3 m (2.5 to 7.5 ft) in thickness; the shallower lifts facilitate the loss of heat. Control of thermal stresses by restricting lift thickness and placement frequency is especially effective for concrete that must be cast at a high initial temperature.

In exceptionally large concrete structures, principally arch and gravity dams, embedded pipe cooling is used frequently to remove much of the heat generated during hydration of the cement. Water is circulated through pipe coils placed at the bottom of each new lift of concrete. Spacing of the cooling pipes, initial temperature of the water, rate of water circulation, duration of the cooling operation, and capacity of the refrigeration-pumping plant are determined primarily from the thermal properties of the concrete, including heat generation characteristics of the cement.

Equally significant is the flow of heat that can lead to accelerated cooling of the mass or excessive cooling of the surface. Newly placed concrete must be maintained at temperature levels that will facilitate hydration and development of minimum strength requirements. Protection is required to prevent freezing of the uncombined free water in saturated new concrete, and accelerating the hydration process by the addition of calcium chloride or similar admixture will reduce the duration of protection required. Surface-applied insulation is commonly used during cool and cold weather conditions to regulate the loss of heat. Such heat losses create adverse thermal gradients that may lead to cracking of the surface concrete or the entire structure.

Besides being important in the dissipation of internal heat, thermal properties of concrete contribute to damping ambient cyclic variations. Few buildings, residential or industrial, are designed without consideration for thermal insulation. Lightweight aggregate concrete and foamed or cellular concretes provide a measurable degree of insulation for the interior, in addition to their lower dead-weight structural advantage. In consideration of heat and energy costs, designers now utilize the damping effect, expressed by thermal diffusivity, of leveling out the cyclic air temperature variation. Normal-weight concrete ideally combines strength and durability with a favorable thermal diffusivity property that is most effective in reducing the heating and cooling requirements of the building interior.

Restraint and Volume Changes

Restrained volume changes that cause tensile strains are a primary concern in mass concrete structures, particularly in the first few days after the placement of the concrete when the tensile capacity of the concrete can be quite low [31]. Bond or frictional forces between the concrete and the foundation or underlying lifts cause external restraint (K in Eq 1). The degree of external restraint depends upon the stiffness and strength of the concrete and restraining material and upon the geometry of the section. Internal restraint is caused by temperature gradients within the concrete. The warmer concrete in the interior of the lift provides restraint as the concrete in the periphery of the lift cools due to heat transfer to its surroundings. The degree of restraint varies depending on the shape of the structure and where in that structure stresses are being evaluated. An additional factor that influences restraint is the relative elasticity of the restraining structure compared to the restrained structure. Volume change, in the form of a linear length change, is then computed by combining the heat dissipation from the peak temperature of the structure to its final stable temperature, and restraint factors and the coefficient of thermal expansion as shown in the formula above.

Analytical Methods

The prediction of stresses, strains, and cracking in mass concrete at early times presents special problems, because many of the properties of concrete depend on the degree of hydration of the cementitious materials. The rate of hydration of the cementitious materials is affected by the type of materials used and by the temperature and moisture history during the period of hydration. At the same time, the internal environment of mass concrete is affected by the hydration of the cementitious materials. Elevated temperatures generated by hydration are maintained for long periods of time in the center of mass concrete structures and affect mechanical properties that are essential in determining the stress/strain condition of the concrete, such as elastic modulus, compressive strength, creep, and volumetric changes associated with hydration.

The analysis of these early-age and time-dependent properties is complex. The analytical methods range from simplified manual computation processes to a complex modeling approach that requires finite element methods [24]. Commercially available software is not necessarily suitable for these problems involving concrete materials and long-term construction schedules. The Corps of Engineers adapted a general-purpose, heat transfer and structural analysis finite element program with a user-defined material model to the problem of thermal analysis of mass concrete structures [25]. The heat-transfer capability of this program uses the finite element method to numerically solve the governing differential equation for heat transfer. Material properties necessary for input to the program are thermal conductivity, specific heat, a mathematical description of the applied heat flux (adiabatic temperature rise), and density. Calculations are carried out in time steps to model the incremental construction of mass concrete structures. The temperature-time history obtained in the heat-transfer analysis is used as the loading in a stress analysis. The effects of time-dependency and temperature on modulus, strength, and creep compliance along with interactive cracking criteria for concrete are incorporated into the calculations with the user-supplied material model [26].

The thermal and incremental construction analysis procedures have been used to develop specific recommendations to limit thermal-related cracking during construction of mass concrete projects [27]. A number of parameters may be controlled to limit cracking caused by restrained volume changes. These parameters can be generally classified as either material or construction parameters. Concrete material parameters include: (a) heat generation; (b) thermal properties (coefficient of expansion, specific heat, and conductivity); (c) mechanical properties (strength, modulus of elasticity, and tensile strain capacity); and (d) time-dependent deformations (shrinkage and creep or stress relaxation). Construction parameters include: (a) lift height; (b) time between placement of lifts; (c) concrete placement temperature; (d) ambient temperature; (e) use of insulation; (f) use of cooling coils; and (g) monolith geometry (section thickness, monolith length, location and size of inclusions such as galleries, culverts, etc.).

In addition to new construction, thermal stress analysis procedures have also been used to address thermal cracking problems in rehabilitation of existing navigation lock walls [28,29]. Improvements in material properties and new design and construction procedures resulting from these studies have significantly reduced the extent of cracking in lock walls resurfaced with cast-in-place concrete.

For arch dams and similar massive structures containing joints that must be subsequently closed by grouting, the product of temperature drop and coefficient of expansion (or contraction) should be such that joint openings of at least 1 to 2 mm (0.04 to 0.08 in.) will occur. A minimum opening of this magnitude is required to permit successful and complete grouting. A recent example of thermal evaluation to assist in joint grouting is contained in Ref 30.

The design of prestressed concrete reactor vessels should include consideration of stresses or strains originating from temperature gradients during operation as well as during shutdowns. Thermal conductivity at higher temperature levels and the resulting thermal expansion or contraction can be determined analytically for introduction into the stress and strain patterns.

The principal thermal property to be considered in rigid pavement design is coefficient of thermal expansion under the cyclic variations in ambient temperature. Spacing with the width of expansion joints is determined mainly by the amount of thermal expansion caused by the amplitude of the ambient temperature cycle and solar heating of the pavement slab.

Conventional building construction, principally rigid frame or continuous structures, warrants consideration of thermal expansion coefficients as they may affect either joints or stresses.

Thermal incompatibility is a primary concern in repair of concrete with polymer mortars. Depending on the type of polymer, the coefficient of expansion for unfilled polymers is 6 to 14 times higher than that for conventional portland cement concrete. Adding fillers or aggregate to polymers will improve this thermal mismatch, but the coefficient of expansion for the polymer-aggregate combinations will still be about 1.5 to 5 times more than that of concrete. The modulus of elasticity of the polymer and the thickness of the repair will also affect the magnitude of the stresses caused by thermal incompatibility. Thick repairs with stiff materials present the greatest potential for failure.

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Pore Structure, Permeability, and Penetration Resistance Characteristics of Concrete

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Preface

THIS CHAPTER WAS FIRST PUBLISHED IN THE PREvious edition *ASTM STP 169C* [1] under the authorship of N. Hearn, R. D. Hooton and R. H. Mills. Since publication there have been significant developments in modeling of penetration resistance of deleterious substances (e.g., [2]), and in the way pore structure and permeability of the cementitious systems are viewed. Even the word "permeability" is more cautiously used and is frequently replaced by the more general term "penetration resistance." The new version of this chapter explores the changes associated with defining and measuring concrete pore structure and permeability.

This chapter is dedicated to the late Ronald H. Mills, who made significant contributions on porosity and permeability, and who was a co-author of the previous version of this chapter in *ASTM STP 169C.*

Introduction

The evolution of modern concrete has been propelled by the need to improve mechanical properties and subsequently to ensure sufficient durability to meet the specified design life. Permeation characteristics of concrete have been studied for over 100 years, with one of the first publications on the water permeability of concrete published in 1889 by Hyde and Smith [3].

In general, durability can be defined as the ability of a material to withstand the intended exposure conditions. The concept of durability encompasses both porosity and permeability properties of concrete. Concrete porosity is complex, as it is not a static system and can simultaneously undergo evolution during the hydration process and deterioration during exposure. Measurements of mass transfer through this continually changing pore structure become a function of testing parameters and the type of transport that is being tested. The following sections will review the fundamental peculiarities of the cementitious pore systems and mass transfer within them.

Porosity of Concrete

Most of the important properties of hardened concrete are related to the quantity and the characteristics of the various types of pores in the concrete. The engineering properties, such as strength, durability, shrinkage, creep, permeability, and ionic diffusion are directly influenced or controlled by the relative amounts of the different types and sizes of pores.

The initial porosity of concrete is determined by the sum of the volume of mixing water, intentionally entrained air, and accidental voids due to incomplete compaction (aggregate voids are discussed in Chapter 38). As the cement reacts with water, the new solids (cement hydrates) occupy space partly within the original grain boundary and partly in the original water-filled space between cement grains and around aggregates. Thus, the porosity of concrete may be classified as follows:

1. Porosity of the aggregates.
2. Water- and air-filled voids after consolidation and final set.
3. Water- and air-filled voids after partial hydration of the cement.

The pores can exert their influence on the properties of the concrete in various ways. In regard to the strength and elasticity of the concrete, it is primarily the total volume of the pores that is important, not their size or continuity. The permeability is influenced by the volume, size, and continuity of the pores. Shrinkage, at least that part of drying shrinkage that is reversible, is largely a function of changes in surface energy at the solid-to-pore interface and, therefore, depends upon the nature of the solid surface and the total surface area of the pore system. Irreversible drying shrinkage may involve capillary phenomena. The resistance of concrete to freezing and thawing and deicer scaling is controlled by the volume, size, and spacing of air voids. Therefore, it is not surprising that there has been considerable interest in developing ways of measuring and characterizing the pore structure and permeability of concrete and in elucidating the various mechanisms by which they influence the properties of the concrete.

The constituents of concrete–hardened cement paste (HCP) and aggregates–are characterized by widely different porosity but potentially similar permeability depending on rock type and

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TABLE 1—Comparison of Permeability and Capillary Porosity Between Well-Hydrated Cement Paste and Natural Rocks [4,93]

Rock		Permeability Coefficient, m/s (rock and paste)	Cement Paste	
Type	Porosity, %		Porosity, %	w/c
Sandstone	4.3	1.7×10^{-11}	30	0.71
Limestone	3.1	8.0×10^{-13}	28	0.66
Fine-grained marble	1.8	3.3×10^{-14}	15	0.48
Dense trap	0.6	3.5×10^{-15}	6	0.38

water to cement ratio of the paste (Table 1 [4]). When cement paste is mixed with aggregates, the resulting concrete has lower porosity than the original cement paste. For instance, concrete, which is mixed with cement paste having 25 % to 50 % by volume of pores (depending on w/c), will have a final porosity between about 7 % and 15 % (respectively) by volume. Few normal density aggregates have porosities greater then 5 % by volume.

Porosity Formed During Concrete Production

Porosity can be introduced into concrete at the time of mixing and placing purposely to improve the concrete's performance, such as by air-entrainment, or it can stem from faulty construction practices allowing excessive bleeding, or from voids such as entrapped air or honeycombing due to incomplete compaction. Adequate workability is the key to reduction of unintentional voids. Workability can be controlled by the quantity, size, and surface characteristics of aggregate, w/c ratio, and judicious use of admixtures. The sizes of the various pore types are compared in Table 2, indicating the possible contribution of the accidental flaws to increased permeability. Potentially, entrained air, entrapped air, or honeycombing (large irregular voids due to poor compaction) are less damaging than bleeding, as they are isolated and do not form continuous flow paths in the cement matrix. Bleeding has more serious effects [5]. The formation of bleed water channels creates continuous flow paths and as the upward movement of the bleed water is impeded by aggregate particles, zones of low density occur below the aggregates. Both effects enhance continuity of the pores. The latter, in particular, contributes to the difference in permeability between concrete, mortar, and pure cement paste.

Those purposely entrained voids, that have a significant effect on the resistance of the concrete to freezing and thawing and deicer scaling, range from a few to several hundred micrometres in size. The air voids may constitute from less than 1 % to occasionally more than 10 % of the concrete volume, the volume and size depending upon several factors including the amount of air-entraining agent used, size distribution of fine aggregate, concrete consistency, and duration of mixing [5].

TABLE 2—Size Distribution Pores and Cracks [6]

	Typical Dimensions of Pores (μm)	Micro-Crack Dimensions (μm)
Interparticle spacing between C-S-H sheets	0.001 to 0.03	1 to 60
Capillary voids	0.01 to 50	
Entrained air bubbles	1 to 50	
Entrapped air voids	1000 to 3000	

The small air voids having a significant effect on concrete durability can be seen and studied using a microscope at a magnification of approximately 50 to 125 times. The volume of the air voids in hardened concrete and the characteristics of the air-void system (void size, voids per inch of traverse, and spacing factor) can be determined in accordance with ASTM Practice for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete (C 457).

Porosity in Hardened Concrete

Porosity decreases with time due to continuing cement hydration if the pores have a high degree of saturation. Other than the larger air voids discussed above, cement paste contains pores within the solid hydrates and often capillary pores, depending on the water to cementitious ratio and degree of hydration. Porosity is affected by the curing and the exposure of concrete to the environment. Continuous channels can form due to either a lack of hydration or due to microcracking in the HCP and in the mortar surrounding the coarse aggregate. Microcracking can result from drying shrinkage, carbonation shrinkage, thermal shrinkage, and externally applied loads. The cracks are larger than most capillary cavities (Table 2) and generally provide continuous flow paths throughout the cement matrix [6].

Methods of Porosity Measurement

Liquid Displacement Techniques

A liquid (water) displacement technique for measuring total porosity of cementitious systems has been standardized in ASTM Method for Specific Gravity, Absorption, and Voids in Hardened Concrete (C 642).

The porosity measured using water as a displacement fluid is always higher than that using isopropanol, methanol, or even mercury intrusion [7] or initial helium displacement. This indicates that water can enter into space that other fluids cannot and this is likely the interlayer space described by Feldman and Sereda [8].

Mercury Intrusion Porosimetry

The early use of mercury intrusion porosimetry (MIP) for cementitious systems was reported by Winslow and Diamond [5]. Because of the ease of the test and the wide range of effective pore sizes that can be measured (200 μm to 2 nm), this test has become very widely used to study the pore structure of concrete. Unfortunately, it suffers from a number of problems when applied to the complex pore structure in cementitious systems [9–11].

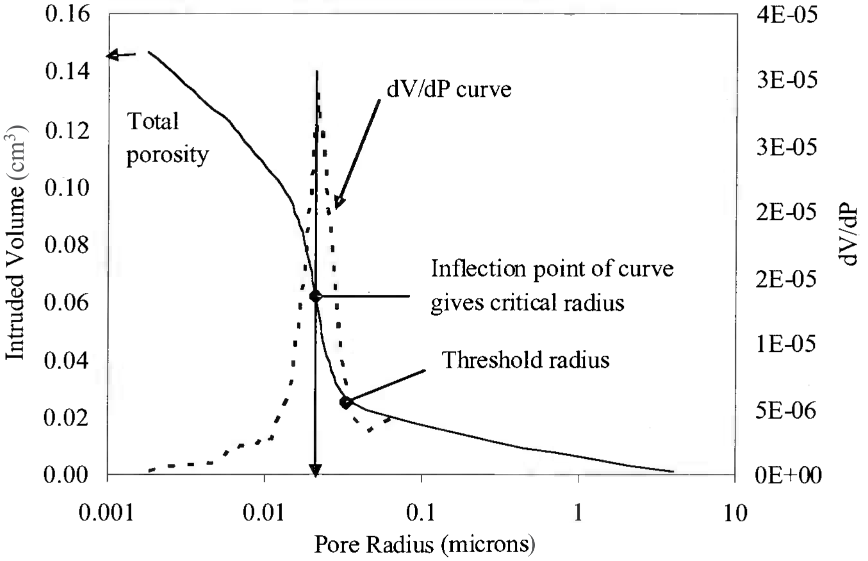


Fig. 1—Determination of total porosity, threshold, and critical pore radii from MIP curve [33].

The method of sample drying can influence pore-size distributions [12] as can sample-size effects [13] and selection of contact angle [14]. The biggest problem is with the assumption that the voids are conical in shape and decrease in diameter from the surface to the core. The presence of so-called ink-bottle pores, as well as continuous pores with narrow openings, which HCP almost certainly have, will not be correctly measured in the recorded pore-size distribution. Recent work by Diamond [15] suggests that the only accurate pieces of information that can be obtained from MIP are the threshold pore radius (the radius where the pore system becomes percolated, as suggested by Bentz and Garboczi [16,17]) and the total intruded porosity. Some authors prefer the use of the critical pore diameter, taken as the maximum of the dV/dP plot, which gives numerical values smaller than the threshold diameter [18] shown in Fig. 1.

Supplementary cementing materials such as slag or fly ash, which often hydrate after the initial pore structure is established, were postulated by Bakker [19] to result in blockages of capillary pores without a huge reduction of total porosity. These would certainly be examples of ink-bottle pores. Feldman [20], using double intrusions of mercury, showed that this indeed was the case with blended cement pastes where, unlike Portland cement pastes, the mercury forced its way through these blockages, resulting in damage to the pore system. However, not all agreed with his interpretation [14].

Other Methods

Other methods, which are not discussed in detail, include capillary condensation, BET, nitrogen or water vapor adsorption, helium inflow, image analysis, low-angle X-ray scattering, and nuclear magnetic resonance. These methods have been reviewed by Diamond [9] and by Feldman [10] and, with the exception of image analysis, are largely research techniques [21,22].

One problem with using image analysis to automate ASTM C 457 has been in distinguishing between entrained air voids in the paste, and cracks as well as voids in the aggregates. Better sample preparation techniques and shape factor analysis, which automatically separate out the spherical entrained air voids, are needed to allow ASTM C 457 to be fully automated. Commercial equipment is now available which uses a digital microscope and a motorized X-Y stage to perform a modified point count on a contrast-enhanced polished specimen. Collecting the required number of points takes about 6 min and the standard C 457 calculations are used to calculate air-void parameters [23]. Also, developments are taking place using high resolution flat-bed scanners and multiple scans of polished surfaces after different preparation steps [24,25].

Summary of Porosity

Concrete can be visualized as consisting of a heterogeneous mixture of components, each component having its own characteristic pore dimensions. Excepting compaction porosity from poor placement practices, the air voids are normally the coarsest of all pores and may constitute from less than 1 % to occasionally more than 10 % of the total volume of the concrete. Approximately 65 to 75 % of the volume of concrete is aggregate, frequently heterogeneous, with an internal pore volume varying from almost 0 to 20 % or more (most commonly about 1 to 5 %). The cement paste component usually contains both extremely fine gel pores and the coarser but submicroscopic capillary spaces [26]. Over the life-cycle of concrete, the pore structure undergoes changes due to continued hydration, drying and wetting, and deterioration processes. Thus, porosity analysis only provides data for a particular mixture at a particular time.

Transport Mechanisms in Concrete

(This section was adapted from a paper by Hearn and Figg [27].)

Factors that govern the mass transfer of fluids include pore structure, moisture content, and boundary conditions. In concrete, the movement of water is controlled by cracking and the properties of the hardened cement paste (HCP) because in most cases, the aggregates are, by comparison, effectively impervious. Moreover, the movement of water in HCP is complicated by changes in its pore structure due to continued hydration and changing solubility of the constituent materials. Changes in the boundary conditions, such as pressure, temperature, or chemical gradient will affect the state of water held in the HCP. The adjustment to such changes, in order to restore thermodynamic equilibrium, is controlled through humidity flux, which includes adsorption, surface diffusion, vapor diffusion, and bulk flow.

Historically the effectiveness of concrete containment structures was measured assuming Darcian flow, where water flow is driven by a static applied head. In assessment of durability of concrete, not only Darcian flow, but all the possible means of water transport must be evaluated. Five primary transfer mechanisms can be defined:

1. adsorption,
2. vapor diffusion,
3. liquid assisted vapor transfer (film transfer),
4. saturated liquid flow, and
5. ionic diffusion under saturated conditions.

Figure 2 [28] shows the various transport mechanisms and the driving forces behind them. At relative humidities (RH) approaching zero, any external source of water will be initially adsorbed onto highly hydrophilic surfaces of the cement hydration products. According to BET theory, at 11 % RH a single monolayer of water molecules will exist on all the hydrated surfaces [10,29]. With increasing relative humidity, the energy of surface adsorption decreases as the subsequent water molecule layers are further away from the hydration product surfaces; e.g., when the energy of surface adsorption of the first and fourth monolayers of water molecules are 1523 and 8 kJ/kg of H_2O, respectively. Adsorption is not only important at low relative humidities, but acts as a driving force along the pore walls transmitting the flow ahead of the meniscus at higher relative humidity, and as part of the water movement at the initiation of bulk fluid flow in a dry pore [30].

Below 40 to 45 % RH, a capillary meniscus cannot be sustained inside the pores. With the thin film of adsorbed water on the pore surfaces, the mass transfer is in the form of molecular diffusion [31] (Fig. 2). For relative humidity between 45 and 100 %, menisci form in the pore system, with increasing RH corresponding to increasing pore sizes forming menisci. As the pore widths are not uniform, menisci are formed in the necks of pores, so that the transfer is propelled by condensation at the higher pressure side and evaporation at the lower pressure side [31] (Fig. 2). The result is that a short circuit is created, allowing rapid moisture transfer without coherent flow. This type of transfer also has been termed wick action, except wick action is measured on the bulk specimen, while liquid-assisted vapor transfer occurs on micro-scale within the hydrate structure.

At saturation, the fluid transport is controlled by the viscosity of the fluid and the pressure gradient. Darcian flow is a direct relationship between pressure gradient and resulting flow.

The presence of water within the cement structure in liquid form allows movement of dissolved, ionic species driven by the molecular concentration gradient (Fig. 2). The rates of ionic diffusion are defined by Fick's law. Ionic diffusion is frequently the most significant transport phenomenon that determines the rates of physical and chemical deterioration.

Most deterioration in aggressive exposures occurs under conditions where water is present in a liquid form rather than vapor. In liquid conditions, dissolved species can migrate through concrete driven by concentration gradients and following Fick's law of diffusion. If an evaporation front exists, the aqueous solution is pulled into concrete through capillary suction, and at the evaporative front precipitation and crystallization of dissolved species can create tensile stresses in the pores. Under freezing conditions, saturated pore systems will undergo tensile stresses due to expansion of the forming ice.

Inter-Relationship of Transport Mechanisms

There is a considerable body of work on the measurement of permeation characteristics of concrete; however, very little work has been done to compare permeation rates obtained using various flow mechanisms. Conceptually, it is obvious that some transport mechanisms will be more effective than others at

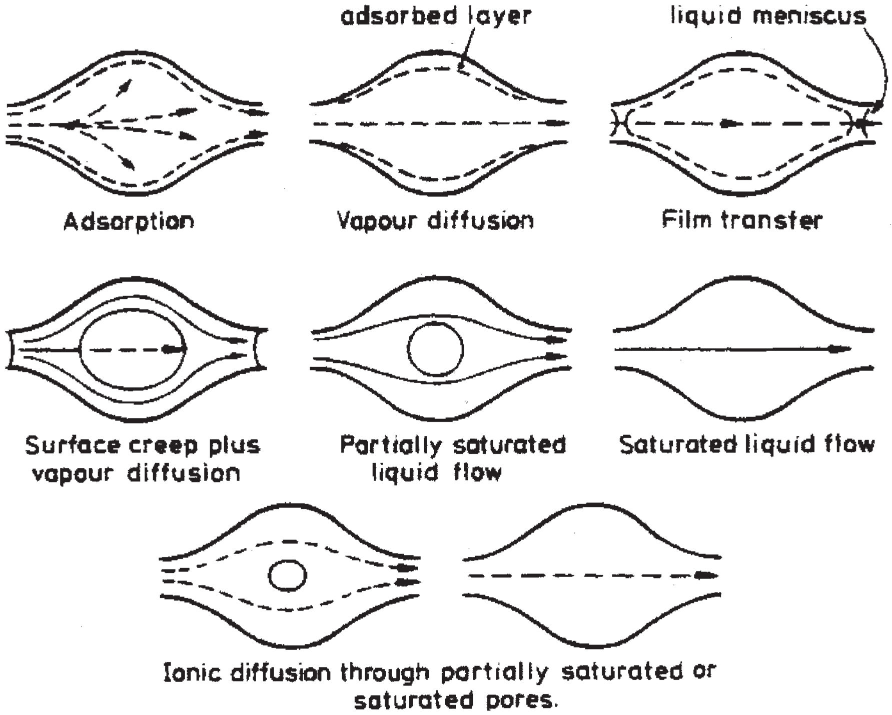


Fig. 2—Idealized model of movement of water and ions within concrete (adapted from [28]).

TABLE 3—Comparison of Mass Transfer of Water through Various OPC Pastes using Darcian Flow Measured under High Pressure 10 MPa (1450psi) and by Wick Action [32]

Mixes	Rate of Water Transmission (g/day)	
	Darcian Permeability	Wick action
0.3	< 0.0024	2.32
0.4	0.041	5.24
0.5	1.42	5.90

transmitting a unit mass per unit time of liquid water, water vapor, or ionic species. In concrete, these rates of transmission are a function of the microstructural characteristics of the cementitious system. A study by Leong compared transmission rates of OPC and blended pastes [32]. For low permeability materials the effect of wick action on liquid ingress was 1000 times greater than transmission driven by pressure head as shown in Table 3. Nokken [33] also found that flow due to wicking was orders of magnitude greater than in saturated permeability. Other papers showing the effect of wick action are Nokken et al. [34], Buenfeld et al. [35], and Aldred [36]. Hong and Hooton [37] showed that cyclic absorption will also accelerate ingress of chlorides.

Figure 3 relates depth of penetration achieved through externally applied pressure and the size of the pore radius. Thus, the smaller the pore radius the greater the pressure required to achieve a given depth of penetration. In concrete, the transmission rates are further complicated, due to possible discontinuity of the pore structure, where both Darcian flow and capillary suction become less significant and transmission may become diffusion controlled. In order to better comprehend the interaction between transmission rates and microstructural characteristics, key elements of the cementitious microstructure must be examined.

Summary

Three stages of moisture front propagation in concrete can be defined:

1. for partial vapor pressures below 0.45, before a meniscus is formed, the moisture movement is controlled by adsorption and surface diffusion;
2. for partial vapor pressures between 0.45 and close to 1, moisture transfer is achieved through vapor diffusion and capillary tension; and
3. in saturated or nearly-saturated material, moisture transfer is mainly due to laminar flow, controlled by viscosity and defined by Darcy's law.

Flow into unsaturated concrete may be supported by all of the preceding mechanisms. The complexity of the pore structure of concrete, its variation with mix proportions, curing and conditioning, creates difficulties in numerical analysis even when only one flow mechanism is examined. Moreover, the presence or introduction of water into the concrete's pore system obscures analysis even further, because of processes such as swelling, continued hydration, and dissolution and precipitation of alkalis.

Methods of measuring multi-phase moisture flux in concrete suffer from variability or simplistic modeling divorced from the real environment. The following sections discuss procedures for measuring mass transfer in concrete and their advantages or disadvantages over direct measurement of saturated flow.

Transport Test Methods and Standards

Gas

Gas Flow

Pressure-induced gas flow through a sample appears to follow Darcy's law, and the porous solid is often characterized by the intrinsic permeability coefficient, *K*.

$$\frac{dq}{dt} = KA\frac{\rho g}{\mu}\frac{dh}{dL} \qquad (1)$$

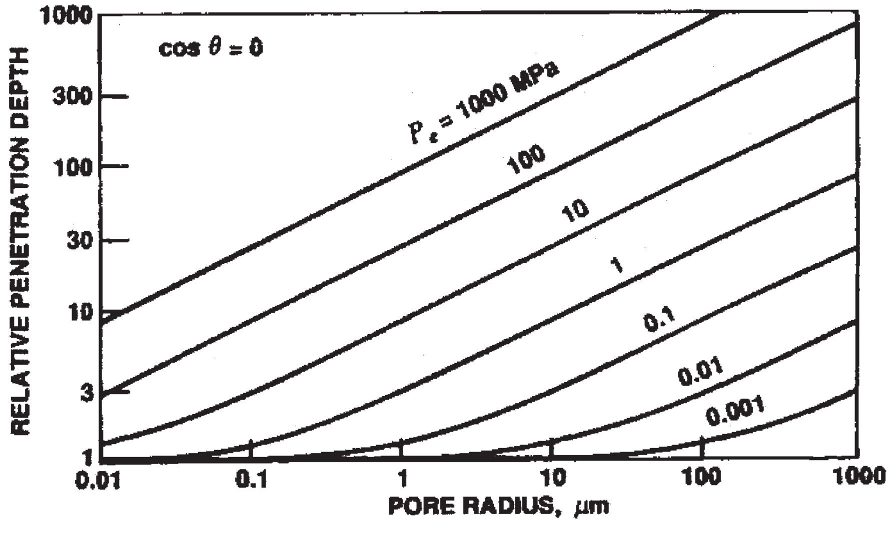


Fig. 3—Influence of external pressure (Pe) on penetration (θ = contact angle of water) [51].

where

ρ = density of the permeant (kg/m^3),
g = acceleration due to gravity (9.81 m/s^2),
μ = viscosity of the permeant (Pa·s), and
K = intrinsic permeability coefficient (m^2).

Equation 1 incorporates Carman's findings [38] (which show an inverse relationship between the rate of flow and viscosity of the permeant) with Darcy's equation. The intrinsic permeability coefficient thus depends only on the microstructural properties of the porous medium and is supposed to be independent of the properties of the permeant.

The test can be rapid and reproducible, but only if the moisture condition of the concrete sample is known and carefully controlled. As the moisture content may vary from surface dry to nearly saturated, so will intrinsic permeability, because water is an effective barrier to gas flow. Estimating the wetted surface area and calculating the hydraulic mean radius can estimate the permeable porosity at various moisture levels. The moisture level, however, must be constant throughout the specimen. Conditioning of the test samples to a constant moisture content is a lengthy process, especially for thick samples. Though oven drying is a quick and uniform method of conditioning, it can alter the microstructure and cause extensive cracking. The alteration of pore structure and flow can be minimized by drying techniques such as freeze-drying or solvent replacement using isopropanol [12,39,40].

There is no direct relationship between gas and water permeabilities. For materials of low porosity, gas permeability is always greater than that of water. This discrepancy was attributed by Klinkenberg [41] to the gas-slippage effect. He proposed the following relationship between gas and liquid permeabilities for a porous medium:

$$K_a = K\left(1 + \frac{b}{P_m}\right) \tag{2}$$

where

K_a = gas permeability (m^2),
K = actual intrinsic permeability (m^2),
b = constant (m), and
P_m = mean pressure (m).

Bamforth [42] derived a relationship, using Eq 2, for concrete. There is, however, some doubt regarding the application of Eq 2 to cementitious materials, because of changes to the microstructure, including cracking, that accompany drying.

Gas Diffusion

Gas diffusion measures the concentration of a chosen gas (usually oxygen) across the sample, due to exposure at the upstream face. Typically, a nitrogen stream is initially maintained at equal pressure on either side of the specimen. A slight over-pressure of oxygen is applied at the upstream face and the resultant gas transfer is detected by means of a gas chromatograph on the downstream side [43]. This technique is useful for dense concretes, and has also been used to predict carbonation rates and reinforcement corrosion rates. The limitations discussed with regard to gas flow also apply to gas diffusion testing and interpretation because water in a partially saturated system has the same effect as a solid barrier.

Depth of Carbonation

Depth of carbonation is taken as a measure of the rate of reaction between carbon dioxide from the atmosphere and the cement paste. Carbon dioxide initially reacts with calcium hydroxide as shown below:

$$Ca(OH)_2 + CO_2 = CaCO_3 + H_2O$$

The hydrates (C-S-H) are also affected once the pH drops and breaks down into hydrous silica and calcium carbonate. The rate of reaction depends on the diffusion of carbon dioxide into the concrete, and the carbonation front is often taken to be revealed by the color change resulting from the application of a phenolphthalein indicator onto the freshly exposed concrete cross section. This test, of course, detects the advance of a pH change and indicates acidity from all possible sources, not exclusively carbonation. This test is most useful for rapid on-site evaluation where the depth of acidity together with the age of the structure is used to estimate the rate of carbonation and the possible onset of corrosion in reinforcing steel.

Water

Water-Vapor Diffusion

Water-vapor diffusion refers to the movement of water vapor across the sample driven by a difference in partial pressure between the upstream and downstream faces. The common methods of testing water vapor diffusion are either by measuring the rate of drying of a sample, or by creating a vapor pressure gradient and then monitoring moisture transfer [31]. The latter is achieved by using a dry cup filled with desiccant on the downstream side of the specimen and storing the whole in a controlled humidity environment, as in ASTM Standard Test Methods for Water Vapor Transmission of Materials (E 96). Periodic weighing of the sample and the desiccant monitors the movement of moisture through the sample. The diffusion coefficient, D_p, based on Fick's law, is calculated using Eq 3. The rate of water vapor transmission has its most practical application when the sample is in contact with water on one side and a desiccant on the other. These conditions, under which both saturated and unsaturated flow take place, are common in concrete water retaining structures. Not much research has been done in this area, but the intractability of analyzing the combined effect of saturated flow, capillary action, and vapor diffusion is well known [30]. The rate of water vapor diffusion is governed by the vapor pressure gradient and the microstructure of the specimen and can be described by Fick's first law:

$$Q = -K_v A\, dp/dx \tag{3}$$

where

Q = vapor transport rate (m^3/s);
K_v = vapor diffusion coefficient (m/s);
A = cross-sectional area (m^2); and
dp/dx = vapor pressure gradient (m/m).

ASTM subcommittee F06.40 dealing with flooring developed Standard Test Method for Measuring Moisture Vapor Emission Rate of Concrete Subfloor Using Anhydrous Calcium Chloride (F 1869), the so-called "moisture dome test," for measurement of moisture loss from a concrete surface. A container of calcium chloride is placed inside a known area of concrete, which is sealed from the environment, then the rate of moisture emitted from the concrete is measured by the increase in mass of the calcium chloride desiccant. More recently, that committee has also developed a relative humidity test, ASTM Standard Test Method for Determining Relative Humidity in Concrete Floor Slabs Using in situ Probes (F 2170).

Absorption and Rate of Absorption

Most concrete is only partly saturated and the initial ingress of water and dissolved salts is dominated, at least initially, by capillary absorption rather than either water permeability or ion diffusion. A wide variety of water absorption tests on concrete have been developed. These tests measure the weight gain of a sample, volume of water entering the sample, depth of penetration, or a combination thereof, by either complete immersion of dry samples in water, exposing only one face to water, or spraying the specimen surface with water. Absorption is either measured at a single, arbitrary time or by measuring the rate of absorption (by change in mass). Although in all these tests the absorption process is proportional to the square root of time over a specified time period, the sorptivity varies a great deal between test methods. The absorption tests have been standardized in many countries (for example, in Great Britain: BS1881 Test for Determining the Initial Surface Absorption of Concrete; in North America: ASTM Test Method for Specific Gravity, Absorption, and Voids in Hardened Concrete (C 642), ASTM Test Method for Evaluating the Effectiveness of Materials for Curing Concrete (C 1151), and ASTM Test Method for Measurement of Rate of Absorption of Water by Hydraulic Cement Concretes (C 1585); and in Australia: AS1342); however, the differences in the test limits and procedures create considerable variation in sorptivity measurements [44].

In ASTM C 642, the water absorbed by an oven-dried specimen is measured after a 48 h immersion or after such immersion followed by 5 h in boiling water. The ratio of the water absorbed to the dry weight is the absorption. In ASTM C 1151, which has been discontinued, the absorptivity of 10-by-25-mm-diameter, methanol-dried cores are measured after 60 s of contact of one surface with water. The difference in absorptivity between the top, cured surface and the bottom, molded surface is taken as a measure of the effectiveness of curing.

In British Standard 1881, a closed reservoir is sealed onto the concrete surface. The reservoir is flooded with water and sealed except for an open-ended capillary tube that is used to monitor the volume of surface absorption after 10, 30, 60, and 120 min.

The major difficulty in the assessment of absorption tests is the determination of the initial moisture condition, which affects sorptivity measurements [30,45,46]. Moreover, in poorly compacted concrete, where entrapped air voids do not get filled with water, the absorption may be lower than for a thoroughly compacted sample. The new ASTM Test Method for Measurement of Rate of Absorption of Water by Hydraulic-Cement Concretes (C 1558-04) addresses some of these issues and provides a method to evaluate the rate of absorption of concrete in the laboratory. Slices of concrete cylinders or cores are conditioned at 80 % RH and 50°C for three days either in a humidity-controlled chamber, or by drying in an oven with the samples sealed in a desiccator over a saturated solution of potassium bromide. This is followed by sealing the samples inside closed containers at 23°C for another 15 days. Then, the sides of the specimen are sealed and the top face is fitted with a loose fitting plastic film. The test face is exposed to water and the change in mass is measured at various time intervals from 1 min up to 192 h.

The water uptake in volume per unit of surface area is plotted against the square root of time. From this, the initial rate of absorption is plotted as the initial slope of this graph. After several hours, the rate of absorption drops off sharply, and the volume of absorbed water is calculated at this “nick point” in the curve. A secondary rate of absorption at times beyond the nick points is also calculated.

The advantage of this test is that absorption is one-dimensional. Absorption from a single face of interest can be measured, unlike other tests where rates of transport are averaged over the thickness of the specimen. This makes the rate-of-absorption test suitable for evaluation of the effects of curing on a surface in addition to assessing the quality of the concrete mixture.

The C09.66 subcommittee is currently considering an in-situ rate of absorption test, based on the work of DeSouza et al. [47–49], which also makes use of in situ moisture data to correct the measured absorptions. While a number of commercial devices exist, assessing and correcting for in-situ moisture contents is the most important issue with respect to interpreting in-situ test results [46].

Capillary absorption tests, besides measuring degree of imperviousness, also give an indication of mean pore radius and porosity of the sample. The Concrete Society [43] in their summary has provided the following equations for determination of mean pore radius and porosity

$$r = \sqrt{\frac{4\mu}{P_0}} \cdot \frac{d}{\sqrt{t}} \tag{4}$$

where

r = mean pore radius (m);
μ = viscosity of water (Pa s);
P_0 = atmospheric pressure (Pa);
d = depth of penetration, for example, from a splitting test (m); and
t = time (s).

$$v = \frac{1000\ M}{Ad} \tag{5}$$

where

v = porosity
M = gain in mass (g),
A = area of penetration (m^2), and
d = depth of penetration (m).

Water Penetration

The water penetration test, standardized in Germany as the DIN 1048 test, involves subjecting one end of the unsaturated concrete to a pressure head. The measure of water penetration is achieved either by measuring the volume of water entering the sample or by splitting open the cylinder and measuring the average depth of discoloration (due to wetting) taken as equal to the depth of penetration. As discussed earlier in the section on Bulk Flow, the usual external hydraulic forces in structures are small compared to that of capillary tension. There are many equations used to model water penetration (a good summary can be found in Emerson [50]). In penetration tests where artificially high pressures are applied, the equations must be modified. Reinhardt [51] presented the combined effects of capillary tension (P_c) and hydraulic pressure (P_e) on the fluid penetration coefficient (B), in the following form

$$B = \frac{r}{2}\left(\frac{P_e + P_c}{\eta}\right)^{1/2} \tag{6}$$

where B is equivalent to sorptivity (S), as defined by Hall [52].

Besides the DIN 1048 test, several in-situ tests utilize water penetration or absorption, or both, for estimating the quality of concrete; for example, Figg's tests [53] and several modified versions (see Refs [54,55]).

The water penetration results are often complicated by the initial moisture state of the specimen and the nonuniformity of moisture distribution. Moreover, the microstructural characteristics in concrete change with the introduction of water, modifying the pore size distribution of the matrix.

Saturated Flow

Pressure-induced water flow through a saturated sample follows Darcy's law.

$$\frac{dq}{dt}\frac{1}{A} = K\frac{\Delta h}{L} \tag{7}$$

where

dq/dt = rate of flow (m^3/s)
A = cross-sectional area of the sample (m^3)
Δh = drop in hydraulic head across the sample (m)
L = thickness of specimen (m)

Saturated water permeability testing involves subjecting the sample to a pressure gradient. The measurement of the outflow enables the determination of the permeability coefficient, k (described in Eq 2), or the intrinsic permeability coefficient, K, which are related by:

$$K = \frac{\mu}{\rho g}k \tag{8}$$

where
ρ = density of the permeant (kg/m^3),
g = gravitational acceleration ($9.81\ m/s^2$), and
μ = viscosity (Pa s)

so that conversion factors can be calculated for the various fluids at various temperatures. For water at 23°C, for example,

$$k = 9.75 \times 10^6 K \tag{9}$$

The intrinsic permeability is solely a function of the pore structure, not of the permeating fluid.

One of the major objections of the saturated water flow test is that the boundary conditions are not representative of the usual concrete environment. Even so, it has drawn considerable attention over the past century (from Hyde and Smith [3] to Reinhardt, Hearn, and Sosoro [56].

The major attractions of this test are:

1. Saturated flow gives intrinsic permeability as defined by Darcy's law.
2. Saturated flow has only one fluid transport mechanism—flow of water through voids—while unsaturated flow encompasses diffusion, absorption, capillary, and saturated flows.
3. The conditioning process for saturated flow (water saturation of the sample) does not damage the existing microstructure.

The major disadvantages of saturated water permeability testing are:

1. Potential problems with saturation of specimens [57–59].
2. Establishing equilibrium flow conditions [60].
3. Decrease in flow with the progress of the test (the self-sealing phenomenon [61]).

Ionic Diffusion and Chloride Ingress

While concrete can be penetrated by many aggressive ions, the main concern with ionic diffusion is chloride ion migration into concrete, because of the extent of reinforcement corrosion damage due to deicing and marine salts. However, sulfate ions can also be of relevance for ionic diffusion as well as other mechanisms of ionic transport.

If a concrete surface is not saturated at the time it is exposed to ionic solutions, then capillary tension will rapidly draw the solutions into the surface layer until the surface layer becomes saturated. This is a very common mechanism of chloride ingress.

If the concrete surface is exposed to wetting and drying cycles and is intermittently exposed to chloride solutions, the capillarity will continue to absorb chlorides, concentrating them and building up an interior "surface" concentration at the depth of the convection zone. For the de-icing salt exposure, the chloride concentration in the outer convection zone will build up during the winter and reduce due to rain washout in warmer weather [62]. This is the scenario for bridge or parking decks subject to de-icer salts or for the tidal and splash zones in marine structures.

When concrete is saturated, both surfaces are inundated, and at least one surface is exposed to a chloride solution (for example), then a chloride concentration gradient exists between the surface and the pore solutions and pure diffusion will result. The magnitude of the concentration gradient is the driving force for diffusion as solutions seek to come to an equilibrium concentration. When the external source of chlorides is constant then the surface concentration builds up rapidly. In the case of de-icing salts, the surface concentration will build up slowly over many years and only then will the surface concentration become constant.

Wick action can occur where relatively thin structural elements are exposed on one side to ionic solutions and on the other to air at a relative humidity less than 100 % (e.g., parking deck soffits or the inside of tunnel liners or for slabs-on-grade), causing the ionic solutions to be drawn toward the air-exposed surface and evaporated. This will result in an increased ionic concentration inside the concrete at the depth of evaporation. The effect of wick action will be to increase the rate of chloride ingress beyond that predicted by diffusion alone [63].

For tunnel linings and other elements under hydrostatic head, pressure-driven fluid movement will also increase the rate of chloride ingress beyond that predicted by diffusion alone.

Ionic Diffusion Tests

Chloride diffusion of in-situ concrete is usually determined by analyzing a concrete core at successive depths, thus establishing a chloride ion concentration profile. The diffusion coefficient, D_c, is often calculated by using Crank's solution (Eq 10) to Fick's second law, after fitting an equation to the chloride profile in order to first establish an estimate of the surface chloride concentration, C_s

$$C(x,t) = C_s[1 - erf\,(x/2\sqrt{D_c t}) \tag{10}$$

where

$C(x,t)$ = chloride concentration at depth x (m) at age t (years),
C_s = surface chloride concentration (%), and
erf = error function (from standard tables).

This solution to Fick's second law assumes both surface concentration and diffusion coefficient are constant with time.

ASTM Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion (C 1556-03) is based on the Nordtest NT Build 443 [64]. In this test, slices from concrete cylinders or cores are sealed except on one face, vacuum saturated, then immersed in a 2.8N NaCl solution for at least 35 days. The sample is then profile ground inward from the exposed face, and an equation is fitted to the chloride concentration versus depth and using Eq 10.

This provides a chloride bulk diffusion coefficient for the concrete at that age of test. These values can be used as input parameters for chloride service-life prediction models [65].

Since the total chloride content (free plus bound chlorides) is measured at each depth in this test, this bulk diffusion coefficient is an apparent chloride diffusion coefficient. For use of this value in service-life modeling, chloride binding needs to be considered since not all of the chlorides will be free to penetrate further into the concrete. An effective chloride diffusion coefficient is one where only the free chlorides in the pores are measured with depth, most commonly measured using steady-state migration tests.

Another issue is that as a particular concrete matures, due to reasons including continued hydration, the value of its apparent diffusion coefficient will reduce with age as the capillary pore network is altered. The effect of this was considered by Maage et al. [66], Thomas and Bamforth [67], Stanish and Thomas [68], and Nokken et al. [69]. Therefore, the age at chloride exposure and the length of chloride ponding are important parameters when D_a values are calculated.

This effect can be evaluated by performing the ASTM C 1556 test on a concrete at a series of ages. From this, a log-log plot of bulk diffusion versus age should yield a straight line. The slope of this line can be taken as the time-dependent coefficient, typically with values between 0 and 1 (see Eq 11). This coefficient, m, is also often used in service-life prediction models.

$$D_{(t)} = D_{ref} \cdot \left(\frac{t_{ref}}{t}\right)^m \tag{11}$$

where

$D_{(t)}$ = diffusion coefficient at time t;
$D_{(ref)}$ = diffusion coefficient at a reference time, t_{ref} (often taken at 28 days)
m = constant (slope of line)

Other methods involve the use of a concrete slice as a membrane between two salt solutions. One of these methods uses the concentration gradient of a salt as a driving potential across a thin slice of paste or mortar. The rate of diffusion is monitored by the analyses of the ion concentration change on the lower concentration side [70,71]. Once steady state has been established, often taking several weeks, Fick's first law (Eq 12) can be used to calculate an effective ionic diffusion coefficient

$$Q = Dc\,\frac{dc}{dx} \tag{12}$$

where

Q = mass transport rate (mol/m^{2}s),
D_c = diffusion coefficient (m^2/s), and
dc/dx = concentration gradient of the ion (mol/m^3/m).

However, such static or steady state tests are very time consuming, so an electric potential is often simultaneously applied to accelerate the rate of chloride ingress. These have been termed migration tests and require the use of the Nernst-Planck equation to determine migration-diffusion coefficients. A review of related issues is given by Andrade [72] and details of such a migration test method are given by McGrath and Hooton [73].

Another test, used by highway agencies, has been the AASHTO T259 chloride ponding test. In this test, concrete slabs are cast and moist-cured for 14 days, then air-dried for 14 days. The tops of the slabs are then ponded with a 3 % NaCl solution, typically for 90 days. The sides of the slab are sealed, but the bottom of the slab is exposed to laboratory air at 50 % relative humidity. At the end of this exposure, cores are taken and sliced at about 10–12 mm intervals. Each slice is crushed and digested in acid, then titrated to determine the chloride content.

No standard method of analyzing the results is given but some have adopted an integrated chloride content (the sum of the chloride content multiplied by thickness of the slice for all the slices). This value has been found to be misleading [74] in comparing concretes where in one case, the outer slice has a high chloride content but the chlorides did not penetrate deeper, relative to another case where the chloride has penetrated deeper but the concentrations in each slice are not as high. Having a lower depth of penetration is desirable, but this is not always consistent with a lower integrated chloride content. Recently, a version of T259 was adopted as ASTM C 1543-02, Test Method for Determining the Penetration of Chloride Ion into Concrete by Ponding. In this test, numerous improvements over the previous method are provided, including the option for precision grinding of millimetre-thick layers and use of penetration depth as a figure of merit.

A rapid method, known as the Rapid Chloride Permeability Test, AASHTO T277 and also ASTM Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration (C 1202), has become widely used in North America and abroad. It involves measurement of the total charge passed in 6 h across a sample sandwiched between NaOH and NaCl solutions. The driving force is provided by 60-V of direct current applied between the two sides of the sample. In this test, the conductivity of the saturated sample, including the effects of all dissolved ions, is measured without emphasis on a particular ion.

While this test has become widely used as an index of concrete quality by highway and other specifying agencies, it has also been criticized. Fundamentally, it is simply a conductivity or inverse-resistivity test, and in spite of it often being referred to as the Rapid Chloride Permeability Test, it does not measure the “permeability” or “chloride diffusion” of concrete. Numerous researchers have pointed this out [75–77]. Because current flow is a function of pore fluid conductivity, admixtures which result in large changes in conductivity are unfairly judged by this test (e.g., calcium nitrate corrosion inhibitors) and this is noted in the standard.

Others have criticized it for apparent lack of relationship with salt ponding test data [78] and for the fact that high current flows will result in heating of the sample and solutions during the 6 h test, raising the measured conductivity. However, others have found that ASTM C 1202 test data, if corrected for temperature, do relate to salt ponding data [74,79], and that some of the lack of correlation found by others is in large part due to poor sampling and analysis procedures used in salt ponding tests (i.e., AASHTO T259) [74].

Regardless, it is easy to envisage substitution of the ASTM C 1202 test with a simpler resistivity test or simply to take the initial current reading to calculate a resistivity value. The C09.66 subcommittee is conducting tests to develop such a simplified procedure.

In spite of its shortcomings, after over 20 years of use, the ASTM C 1202 test is found in numerous laboratories in North America and internationally, and continues to be specified for evaluating the quality of supplied and in-place concrete for highway bridges, tunnel liners, and parking structures. Therefore, in spite of other developments, it is likely that this test will remain as an index test for permeability for many years to come.

An alternate rapid index test to ASTM C 1202 for chloride penetration resistance of concrete is a rapid migration test based on the procedure developed by Tang and Nilsson [80] and recently adopted as a Nordtest standard (NT Build 492). Similar to C 1202, this nonsteady state migration test involves measurement of the depth of chloride ingress under an applied DC potential. The depth of penetration is measured by splitting the specimens open after the test and spraying the fracture surfaces with a 0.1N $AgNO_3$ solution. Above about 0.07N chloride concentration, the $AgNO_3$ will covert to $AgCl_2$ and turn white in color. The nonchloride areas will turn brown. A nonsteady state migration-diffusion value can be calculated from the depth of penetration together with knowledge of the magnitude and period of the applied potential. Based on this, AASHTO has adopted a version of this Rapid Migration Test as TP164-03, Predicting Chloride Penetration of Hydraulic Cement Concrete, based on the development work of Stanish, Hooton, and Thomas [81,82].

The test is of similar rapidity to the ASTM C 1202 procedure and gives a very similar ranking of results, but has the advantage that the results are not influenced by the pore fluid conductivity. This makes it applicable to concrete with calcium nitrite corrosion inhibitor.

Relationship Between Porosity and Permeability

The relationship between porosity and transport is of particular interest. Many tests that directly measure transport properties require specialized equipment and long periods of time to complete (e.g., saturated water permeability). Powers and colleagues “observed that the degree of permeability was controlled mainly by the capillary porosity” [83]. In the course of further hydration, the capillary pores become disconnected and the permeability is controlled by the “gel pores.” Given that the capillary pore system presents the pathway for the ingress of deleterious substances, it follows that the formation of a discontinuous capillary pore system is highly desirable. Powers [84] also suggests that moist curing of field concrete past the point of achieving discontinuity is of little value.

The theories of permeability in porous media arise from two schools of thought. One is the application of the Poiseuille-Hagen law (used by Hughes [85]); the other considers viscous drag of moving fluid on a particle. Powers and his coworkers [86] took the second approach, which uses Stoke’s law as a basis. (Stoke’s law is also used when determining particle size distribution of small particles using sedimentation, such as silt and clay.) As the concentration of particles increases, the Stoke’s velocity decreases. Steinour [87] determined a function for the variation in particle concentration using data derived from tapioca suspended in oil. The actual function used is proportional to the inverse of the hydraulic radius, dependent on particle concentration and temperature. The theory was extended to hardened cement paste since it is a porous solid with particle connections involving a small fraction of the surface, and can be thought of as a collection of particles.

To define discontinuity, permeability experiments were performed on cement paste specimens approximately 600 days old, leached of all alkalis [83]. The alkali was leached into the curing water placed on top of the cement paste at the time of casting. The experiments were designed to minimize variations due to osmotic pressure by using leached specimens. The cement is described as ultrafine with a Blaine of 8000 cm^2/g. Fine cement hydrated for an extended period led to completely hydrated specimens free of capillary pores. Four water-to-cement ratio pastes were tested at four different temperatures. A line was fit to the four data points at each temperature relating the inverse of the hydraulic radius to a function of permeability, leading to the following relationship:

$$\log 10^{12} K \frac{C}{(1-C)^2} = 2.13154 - \log\eta(\theta) - \left(\frac{539}{T} + 0.3\right)\frac{C}{1-C} \tag{13}$$

where

K = coefficient of permeability in cm/s,
C = particle concentration, i.e., volume of solids per unit volume of specimen,
$\eta(\theta)$ = viscosity of the fluid [poises] as a function of temperature (°C),
T = temperature (K).

At 27°C, the relationship becomes:

$$\log 10^{12} K \frac{C}{(1-C)^2} = 4.2 - 2.097\left(\frac{C}{1-C}\right) \tag{14}$$

Given that porosity of the gel pores is 26 %, the capillary porosity, p, at complete hydration is given by:

$$p = (1-C) - 0.26 \tag{15}$$

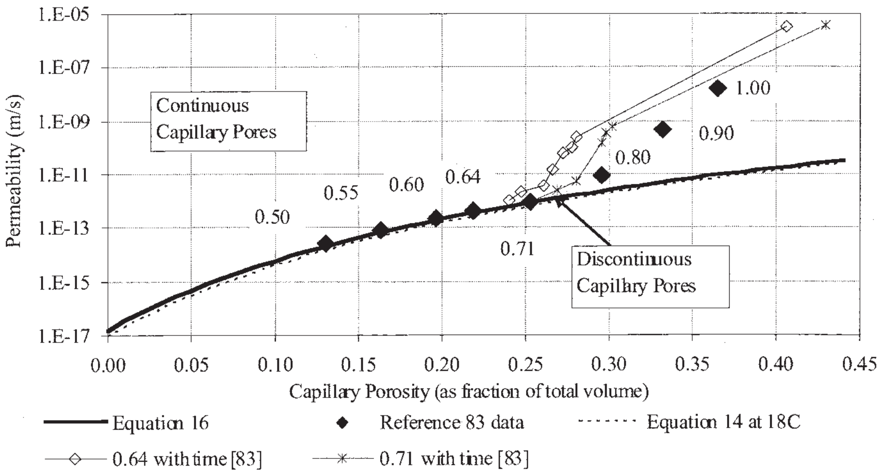


Fig. 4—Definition of discontinuity.

Solving for K (in cm/s) in terms of p yields:

$$K = 10\exp\left(\left[4.2 - 2.097\left(0.74 - \frac{0.74-p}{0.26+p}\right) - \log\left(\frac{0.74-p}{(0.26+p)^2}\right)\right] - 12\right) \tag{16}$$

Figure 4 shows Eq 16 plotted with data from Powers et al. [84] for cement pastes of various water to cement ratios (shown in the figure). The line defines discontinuity. Once the paste has hydrated enough to reach this line, it will continue to move along it. For comparison, a dashed line is shown in Fig. 4 solving Eq 14 for 18°C. It can be seen that temperature differences are minimal. The decrease in porosity and permeability with time for a cement paste with 0.64 water to cement ratio can be seen in the figure. The paste was produced using cement with a fineness of 1800 cm^2/g Wagner. The highest permeability corresponds to the permeability calculated from bleeding rates. The next point was established on the fourth day and the final point on the 24th day after casting. Later permeability measurements yielded points slightly below the line signifying capillary discontinuity. However, alkali remaining in the specimens accounted for the somewhat lesser permeability. Had the specimens been leached of all alkalis, the terminal points for the paste made with the coarser cement would likely lie on the line. The particle concentration was calculated by non-evaporable water content (hydration) of pastes. Mercury intrusion porosimetry only became available commercially after the work of Powers [88]. Although the chemistry and fineness of cement has changed significantly since the time of Powers, the discontinuity relationship was recently validated in concrete by Nokken and Hooton [89] using modern cements and supplementary cementing materials. Figure 5 shows the results of this study showing that capillary discontinuity will not occur with water to cement ratio greater than approximately 0.70.

"The conclusion about continuity or lack of continuity of capillaries rests on conformity or lack of conformity of the data to an equation for the permeability based on the assumption that resistance to flow through a granular body is determined by viscous drags on the individual particles composing that body" [83]. When the flow through the paste does not follow the law, it is assumed that continuous capillary pores exist. The assumption that no continuous capillary pores existed was

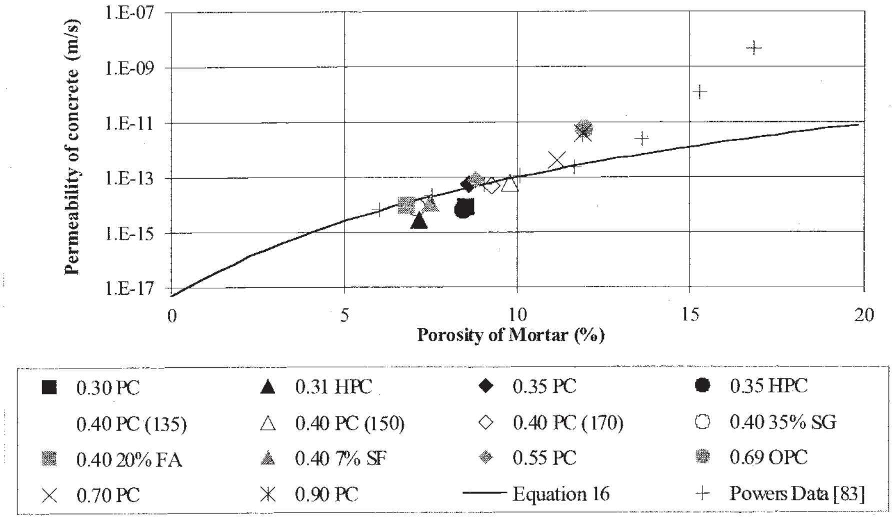


Fig. 5—Discontinuity of concrete [89] compared with Powers [83].

TABLE 4—Time Required to Achieve a Discontinuous Pore Structure [83]

W/C	Time Required	Approximate degree of hydration required
0.40	3 days	0.50
0.45	7 days	0.60
0.50	14 days	0.70
0.60	6 months	0.95
0.70	1 year	1.00
>0.70	Impossible	>1.00

verified by confirming that the solid surface area calculated by the permeability relationship matched that measured by water absorption. The presence of continuous capillaries would not produce the same result.

Table 4 lists the estimated time required to achieve discontinuity of pastes under standard laboratory conditions. The time was based on hydration of ordinary Type I cement (which is certainly of lower C_3S and coarse particle size distribution). The degree of hydration, or maturity, required to achieve discontinuity was estimated from the intersection of measurements of permeability with time for pastes at two water to cement ratios (0.64 and 0.71) with the line signifying discontinuity, resulting in the following relationship:

$$\alpha = \frac{3.15 \cdot w/c \cdot (C/1 - C) - 1}{0.568[1 + (C/1 - C)]} \tag{17}$$

Geiker [90] showed that there is a critical degree of hydration for autogenous shrinkage that can be thought of as achieving discontinuity [17]. Larger cement particle size distribution requires a larger degree of hydration to achieve depercolation [91].

Winslow and Liu [92] found that pore size distributions of the paste, mortar, and concrete at lower degrees of hydration (~37 %) are similar and diverge at ~ 60 % hydration, which seems to indicate that pore continuity is the overriding factor at early ages.

Characterizing Pore Systems

(This section was largely taken from a paper by Hearn and Figg [27].)

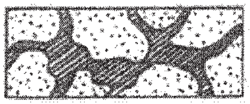
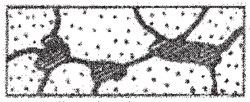
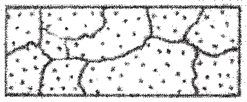
In concrete, various flow mechanisms have different rates of transmission in a singular pore structure, and concrete pore structures undergo dynamic changes. As no two concrete samples are exactly alike, a broad classification was developed to categorize concrete into three groups. Table 5 classifies the porosity-permeation system, from low to high porosity and the corresponding transfer mechanisms. The three systems in Table 5 represent increasing cementitious content and/or levels of hydration.

The most porous system corresponds to high w/c ratios (above w/c = 0.7) and/or low levels of hydration, where high porosity is compounded by high connectivity. In this system, most of the water is free and unaffected by the surface forces of the hydration products. The discontinuity of the pore structure is never achieved, as discussed by Powers (Table 4). As the internal flow paths are unobstructed, the pressure-induced flow would provide the highest transmission rates, through the pore structure.

Most structural concretes are cast at w/c ratios of 0.45 to 0.65. The existing porosity (as determined by the initial w/c) is divided into a multitude of finer pores and, under a proper curing regime, complete discontinuity of the pore system is possible (Table 4). Under these conditions, the water is affected by the surface forces and the Darcian flow becomes less effective as a mass transfer mechanism. Higher pressures are required to achieve measurable flow. Work done by Leong [32] showed that the time required to reach zero flow conditions for w/c = 0.3, 0.4, and 0.5 hardened cement pastes tested at 1.0 MPa were 2, 5, and 17 days, respectively. Subsequent tests on these samples, using a higher pressure of 13 MPa, showed that only w/c = 0.3 had achieved zero flow conditions (i.e., permeability less than 1×10^{-16} m/s), while w/c = 0.4 and 0.5 had permeability of $17 \pm 2 \times 10^{-16}$ m/s and $780 \pm 150 \times 10^{-16}$ m/s, respectively.

High performance cementitious systems have w/c ratio below 0.45, are well cured, and often incorporate supplementary cementing materials. In this system, the pore structure is mostly that of the internal C-S-H gel porosity, so that permeability is a function of the transmission through gel and tortuous pathways through small poorly connected capillaries. Darcian permeability of gel has been determined

TABLE 5—Three Cementitious Systems Based on the Porosity/Permeation models [27]

	System 1 w/c > 0.70	System 2 0.45 to 0.65 w/c	System 3 < 0.45 w/c
Continuous pore radius	Large 	Medium 	Small 
Pore sizes (nm)	10–100	0.1–10	0.001–0.1
Controlling transmission mechanisms	Darcian flow Ionic diffusion	Capillary suction Evaporation Ionic diffusion	Vapor diffusion

by Powers [93] to be 2×10^{-17} m/s, which was also taken to be the limiting permeability of the cementitious systems. This gel permeability value, however, is a theoretical determination. Experimentally, a number of researchers have reported "zero" flow due to inability to measure Darcian flow [39]. This limiting value is a function of the applied pressure gradient during the experiment. For low pressure systems (less than 1 MPa), the Darcian permeability was found to be 10^{-13} m/s, while under high pressure testing (above 7 MPa) $-$ 10^{-16} m/s. Limiting permeability values, however, does not necessarily mean that the concrete at hand is completely impermeable to any aqueous transmission. Under these conditions, where gel segments the pore structure, the governing mass transfer mechanism becomes diffusion of aqueous and ionic species instead of pressure-induced flow.

With the onset of a deterioration process, these systems can become interchangeable. For instance, extensive cracking of the highly impermeable concrete would result in the flow characteristics being determined by the crack parameters rather than the original mix design. Therefore, the initially discontinuous pore structure turns into a continuously interconnected system that will easily support transmission through wick action and, under severe cracking, Darcian flow. In the same way, very permeable concrete at the initial stages of chemical degradation processes can become less permeable as the reaction products fill in the existing pore spaces (for example, alkali aggregate reaction), or through self-healing of the cracks.

Conclusion

As stated in the introduction, the concept of durability encompasses both porosity and permeability properties of concrete. Concrete porosity is complex and covers pores ranging from nanometer to centimeter sizes. Measurement of porosity is significantly influenced by sample history and conditioning. Also, concrete porosity is not a static value and can simultaneously undergo evolution during the hydration process and deterioration in extreme exposures. Measurements of mass transfer through this continually changing pore structure become a function of testing parameters and the type of transport that is being tested. There have been significant advances in the theories and test methods involving the measurement of transport mechanisms and porosity in the intervening years since the last edition. For example, new test methods have been standardized for: Determining the Penetration of Chloride Ion into Concrete by Ponding (C 1543-02), Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion (C 1556-03), and Measurement of Rate of Absorption of Water by Hydraulic-Cement Concretes (C 1558-04). Other test methods are under development.

Currently, for porosity and absorption measurements, C 642 is commonly used. Rate of absorption measurements, such as C 1585, are useful to determine the rate of uptake of liquids into unsaturated concretes, and since they measure uptake from one face, they are useful for evaluation of the combined effects of compaction, composition, and curing history on an exposed surface. For vapor transmission through concrete exposed to liquid water on one side, E96 can be used. To measure surface moisture emissions from floor slabs, F 1869 has been used for many years and F 2170 has been more recently developed. These tests were developed to determine when it was suitable to apply floor finishes, but have been also used to estimate vapor transmission rates through concrete. Chloride ingress by diffusion (C 1556) and combined transport mechanisms (C 1543) exist. The C 1556 test, when run for a range of chloride exposure periods, can provide a bulk chloride diffusion value and time-dependent change for input into some service-life prediction models. As rapid indices of penetration resistance, the C 1202 test continues to be useful for quality control and quality assurance purposes, although other tests, such as AASHTO TP 64 and resistivity/conductivity tests may prove to be more useful in the future. Progress will continue to be made on test methods, due to the impetus to produce highly durable concrete structures, and to more accurately predict their service lives.

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Chemical Resistance of Concrete

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Preface

THIS CHAPTER BUILDS UPON CHAPTERS ON THE same topic from previous editions of the *ASTM STP 169* series. Due to the fundamental nature of the subject, much of what appeared before in *ASTM STP 169, 169A,* and *169B,* authored by Lewis H. Tuthill, and *169C,* authored by G. W. DePuy, appears again here. This chapter will review and update the topic as addressed by the previous authors, introduce new technology that has been developed, and include updated references.

Introduction

Under ordinary processing and environmental conditions, properly designed concrete is a very durable material and may be expected to have a long service life. In addition to being a strong and versatile material with good constructability and low cost, the durability of concrete should be the main reason for concrete being the material of choice. In most situations concrete is a stable material, and well-made concrete resists deterioration for many years. However, there are instances where concrete is subjected to chemical attack by aggressive substances, and the durability of concrete may become a serious concern.

The common forms of chemical attack discussed are leaching by soft water, acid attack, various forms of sulfate attack, carbonation, exposure to industrial chemicals, and exposure to saline water. Two other types of destructive chemical reactions, alkali-aggregate reaction and corrosion of reinforcing steel and other embedded metals, are discussed in other chapters of this volume. Chemical reactions per se in concrete are not necessarily harmful; some may be beneficial, such as pozzolanic reactions. Other reactions may be beneficial in some circumstances but harmful at other times, as is the case with carbonation. Most harmful reactions involve chemicals in a liquid or gaseous form. Most chemicals in a dry and solid form are not harmful to concrete until they are brought into contact with water or other solvents.

The two questions most commonly faced by engineers with regard to concrete exposed to aggressive agents are how long will the structure stand up to the exposure and what can be done to prolong the service life? The answers to these questions require information on the specific exposure conditions and chemical reactions so that appropriate protective strategies can be developed. The more commonly occurring situations involving exposure to aggressive chemicals have been extensively investigated over the years and some general guidelines have been developed for dealing with these situations; however, the attack in a specific situation is a complex matter and may vary greatly depending on the variables governing the reaction. This is particularly true in unusual situations where concrete is exposed to high temperatures or where the performance of concrete is especially critical, as in hazardous waste containment or nuclear waste storage [1]. In these instances, the general guidelines that have served so well in the past may not adequately cover the situation and it becomes increasingly more important that the engineer have a good understanding of the chemical reactions, factors governing the rate of reaction, and mechanisms causing deterioration in concrete.

There are three main types of mechanisms involved in the deterioration of concrete from chemical reactions: (1) mechanical expansive forces arising from the chemical reactions leading to formation of expansive reaction products or products that have a larger volume than the original materials, (2) chemical alterations that weaken or soften the concrete constituents, and (3) dissolution and leaching of soluble components. Under certain conditions, the above mechanisms may occur and cause damage simultaneously.

Concrete may be thought of as a man-made rock, and in this context concrete structures exposed to the elements are subject to the same natural chemical weathering processes (dissolution, oxidation, hydrolysis, and acid hydrolysis) that attack rock [2]. In industrial exposures, concrete faces a wider variety of chemical reactions, ranging from dissolution (dissolving of a solid substance into a solvent such as water) and decomposition (dissociation of one or more compounds and chemical combination into other compounds, such as redox and acid-base reactions) to ion exchange (substitution of one ion for another in a molecular structure that may change the physical properties of the compound).

The rate and severity of attack is governed by the quality of concrete (e.g., permeability), reactivity of the reactants, supply of the reactants, and severity of exposure conditions, including temperature. The reaction is also affected by other factors, such as effects of other substances that alter, inhibit, or promote the reaction; the physical configuration of the reaction site (e.g., access of water and external chemical substances); the nature of the reaction products; and whether

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the reaction goes to equilibrium or completion. Some reactions, such as dissolution and leaching may tend to accelerate future deterioration; other reactions form products that may produce a physical blocking or passivation effect and tend to impede, sometimes only temporarily, further deterioration.

The supply of aggressive agents for reaction is governed by the velocity that the reactants are brought into contact with concrete and the penetration of the reactants into the concrete, which is generally considered in terms of permeability and diffusion [3]. The primary defense against the penetration of reactants into concrete is its quality; specifically, low density (low w/c), low permeability, and minimal cracking. Permeability, the penetration of a substance through fractures, voids, and capillaries in concrete, generally is linearly related to pressure and viscosity of the aggressive agent. Diffusion, the passage of molecules or ions of an aggressive substance through the microstructure of the cement hydration products, is a function of the concentration gradient and is generally related to the square root of time. The velocity of the aggressive agent coming into contact with concrete is a key factor in surface reactions.

Although both the hydrated cement paste and aggregate particles may be involved in harmful chemical reactions, most attention will be given to reactions with cement hydration products. The main cement hydration products are calcium silicate hydrates and calcium hydroxide, but damaging reactions often relate to formation of varying amounts of other reaction products such as a variety of calcium sulfoaluminate hydrates or their analogs.

The portland cement hydration product of greatest significance to chemical resistance is calcium hydroxide ($Ca(OH)_2$, also referred to as lime, hydrated lime, or portlandite). In portland cement concrete calcium hydroxide constitutes about 25–30 % by mass of the hydrated cement paste and is more soluble in water and more chemically reactive than the other hydration products. It reacts readily with acids and with carbonate and sulfate ions. One strategy to improve the chemical resistance of portland cement is to minimize the calcium hydroxide content of the hydrated cement by using a "low-lime" cement (contains more dicalcium silicate and less tricalcium silicate), by replacing part of the portland cement with pozzolans that will react with calcium hydroxide to form more stable calcium silicate hydrates, or by using ground granulated blast furnace slag as a partial replacement for portland cement.

Four general strategies are used to improve the service life of structures under the threat of chemical attack. These are the design of the structure to avoid or minimize contact with water and aggressive agents, the use of supplementary cementing materials, the production of high-quality impermeable concrete, and the provision of a physical barrier to exclude aggressive agents.

The best strategy starts with the design of the structure. This would include avoiding or minimizing contact with aggressive agents, providing good drainage to intercept or eliminate the water migrating through concrete or ponding behind structures, using contraction and construction joints that are water tight and frequent enough to prevent intermediate cracking, and attention to details to control cracking. Design measures may also include the provision of sacrificial concrete (using sections with added thickness to compensate for the chemical attack).

The intrinsic durability of concrete may be improved by the following:

1. Production of high-quality, well-designed, well-consolidated, well-cured, impermeable concrete that requires selection of the appropriate materials, proper mixture design, and good quality control and workmanship. This involves good workmanship throughout the entire production process from batching and mixing, placing and consolidation, finishing and curing to produce a crack-free concrete with a hard and dense surface.
2. In addition to good workmanship, attention must be given to the selection and proportioning of concrete materials, which includes:
    - *a.* optimum cement type and content for the intended use; both strength and durability need to be considered;
    - *b.* good mix design to obtain a workable and compactable mix;
    - *c.* a low water-cement (w/c) ratio (usually 0.40 or lower which can be easily provided by using a water-reducing admixture or high-range water reducer) to reduce shrinkage, capillarity, and permeability;
    - *d.* blending the cement with pozzolan or slag to minimize permeability;
    - *e.* selection of an optimum amount of a well-graded, hard, clean, physically sound, and chemically resistant aggregate; and
    - *f.* proper selection and use of chemical admixtures; if multiple admixtures are used, their interactions and effect on concrete properties should be known.
3. Special surface treatments to produce a hard, impervious surface.
4. Use of properly selected chemically resistant materials, such as chemical-resistant cement and aggregates, or polymer concretes.
5. Use of properly selected impermeable barriers, such as chemically resistant membranes and protective coatings [4–6].

Scaling, Efflorescence, and Leaching

Concrete exposed to water is subject to leaching and the deposition of mineral deposits. Leaching and deposition are related effects produced by the dissolution and precipitation of minerals in water from any source, including groundwater, surface water, or water contained in or transported by concrete structures [7–12]. Leaching refers to the chemical dissolution and removal of water-soluble constituents in concrete. The deposition of mineral deposits includes both scaling or the formation of incrustation and efflorescence.[3] Scale deposits are generally hard, layered precipitates of calcium sulfate, calcium carbonate, or magnesium salts, such as produced by the heating or evaporation of water containing dissolved salts. Efflorescence refers to a white, powdery-to-hard deposit (commonly calcium carbonate or sodium sulfate hydrate, but may consist of other salts), such as is formed when water containing dissolved minerals migrates through concrete and subsequently evaporates on exposed surfaces.

Mineral deposits may form on exposed surfaces where water seeps through concrete along joints and cracks of concrete structures, such as dam faces, pipelines and tunnels, retaining

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[3] In regards to concrete technology, the term "scaling" is used more often in an entirely different sense to refer to the physical process of the removal of thin layers of surface concrete.

walls and abutments, and building foundations. Occasionally, surface evaporation leads to transport of water containing various ions through permeable concrete, leading to deposition of salts on horizontal slabs or stem walls close to the soil line. These kinds of deposits may be objectionable for several reasons–they are unsightly, and they may adversely affect the performance of a structure by reducing the effectiveness of heat exchanger surfaces or by obstructing the passage of water in drain holes and water tunnels [13,14]. The deposits are sometimes taken as evidence of leaching in the concrete, but the leaching in such cases is often relatively minor.

Leaching may erode concrete either externally on exposed surfaces, as in the case of water flowing in canals and pipelines, or internally in the case of water migrating through more porous paste, cracks, and joints in a concrete structure. Relatively cold and pure water from mountain streams and reservoirs, and product water from desalting plants, are capable of leaching concrete and create the sandy appearance sometimes observed in some concrete conduits, canals, and flumes. Internal leaching by water seeping through cracks and joints in concrete is sometimes suspected of creating voids and weakening the concrete.

The principal constituent of concrete usually involved with leaching or mineral deposition is $Ca(OH)_2$ (calcium hydroxide). $Ca(OH)_2$ is soluble to a certain extent in water and is the main cement hydration product susceptible to leaching. The other principal hydration products (calcium silicate hydrates and calcium aluminate hydrates) are much less soluble. The observable differences in resistance to leaching due to the use of different types of portland cements are very minor and usually attributed to differing amounts of $Ca(OH)_2$ formed from the hydration of the cement. Other factors affecting the permeability of the concrete, such as water/cement ratio, the appropriate use of pozzolans or slag, and the presence of microfractures and cracks, are much more important.

$Ca(OH)_2$ dissolves in water by dissolution and by acid-base reaction in acidic water. The amount of $Ca(OH)_2$ that can be dissolved is dependent upon the pH of the water, its temperature, and the concentration of other soluble materials in the water. The pH of natural water low in dissolved solids is related to the absorption of carbon dioxide (CO_2) gas from the air. The solubility of CO_2 in water increases with pressure and decreases with temperature. On this basis, cold water is more aggressive towards concrete than warm water as calcium hydroxide is more soluble in cold water than in hot water.

The effects of other dissolved salts in water are major factors governing the dissolution of lime in concrete by water. Hard water (water containing dissolved calcium, magnesium, and iron salts) will not dissolve calcium hydroxide to any appreciable extent, but soft water (water relatively free of the dissolved salts) readily dissolves calcium hydroxide and may become quickly saturated with calcium.

Saturated lime water is in a delicate equilibrium condition, and slight changes in conditions can quickly result in the formation of a precipitate. A common example is that of lime-saturated water migrating through concrete. When the water becomes exposed to the atmosphere, calcium carbonate frequently precipitates on the surface of the concrete. The precipitation occurs as a result of a shift in equilibrium of CO_2 in solution due to a decrease in pressure, a probable increase in temperature, and evaporation.

Leaching and deposition processes usually tend to either increase or decrease with time, rather than continue on at a more or less constant rate. Most commonly, the processes tend to diminish. These processes are complex and depend on the internal chemistry of concrete as well as on the (often changing) environmental conditions of use. The rate of increase or decrease may be taken as evidence of whether the problem is becoming worse or is solving itself. At times, leaks are observed to plug themselves through the formation of calcium carbonate precipitates–the so-called autogenous crack healing sometimes observed in concrete pipes and canals.

The tendency of water to dissolve or to form a precipitate of a calcium salt is an issue related to the degree of saturation of the salt in a particular situation, and the other factors governing solubility. A common indicator of the tendency of a particular water to dissolve or precipitate calcium carbonate is content of the water, pH, total alkalinity, dissolved solids, and temperature [15]. This is applicable to concrete, as calcium usually precipitates in the form of calcium carbonate. More recently, chemical equilibrium models, such the U.S. Geological Survey's WATEQ model, have been developed to evaluate mineral saturation [16,17]. A more definitive answer would require a chemical mass balance approach [18–20].

Questions on the cause of mineral deposits, or on whether leaching is damaging to a particular concrete structure, are difficult to answer as usually there is insufficient information on the chemical equilibrium condition of the water before and after exposure to the concrete. An indication of the potential for the water to dissolve lime could be obtained by testing the water immediately upstream of the structure to obtain a Langlier, or preferably a WATEQ saturation index. This may be difficult to do as it would require sampling of the water upstream of the structure and also at the location where the water seeps through the concrete. Drilling and coring may also be required to determine if leaching has weakened the concrete; however, some caution in interpretation should be exercised since internal voids can also be produced by defective workmanship in the construction of the structure. In most cases, the appearance is worse than the damage, and very little good-quality concrete has actually been destroyed or made unserviceable by leaching of lime [21].

The Bureau of Reclamation investigated the effects of warm to hot distilled water on portland-cement concrete in a study on the use of concrete in desalting plants, and concluded that concrete was not suitable in exposure to flowing distilled water [22]. The studies showed that concrete does not resist leaching in warm to hot distilled water, and that severity of leaching increases as temperature increases. The use of up to 23 % fly ash was not effective in reducing the leaching by warm to hot distilled water.

In addition to the general steps to improve chemical resistance, other means to consider include:

1. Mineral deposits
   a. Stop the water seeps and leaks.
   b. Removal by physical or chemical means (such as sulfamic acid, or using a protective coating as an aid).
2. Leaching
   a. Use sacrificial concrete.
   b. Stop or divert the flow of water.
   c. Pretreat the water to saturate it with calcium before it comes into contact with the concrete.
   d. Use of special cements, such as aluminous cement, "low-lime" portland cement (contains more dicalcium silicate and less tricalcium silicate), a polymer modified

cement, or a polymer concrete. These options are usually unavailable.

e. Use of mineral admixtures blended with portland cement (may not be effective against hot distilled water), such as ground blast furnace slag, pozzolans, such as 20–30 % of a good natural pozzolan or fly ash, or silica fume.

Sulfate Resistance

External Sulfate Attack

Sulfate attack, sometimes called "sulfate corrosion," is a particularly severe type of deterioration resulting from chemical reactions occurring when concrete components react with sulfate ions (SO_4^{2-}) present in solutions in contact with concrete or, under certain conditions, within the concrete mixture itself. Sulfate attack has been reported in many parts of the world, including the western United States, the northern Great Plains area of the United States and Canada, Spain, Great Britain, the Middle East, and in industrial situations where concrete has been exposed to solutions containing sulfates. The attack is particularly prevalent in arid regions where naturally occurring sulfate minerals are present in the water and ground in contact with the structures. The necessary conditions for sulfate attack are known and preventive measures can be taken to eliminate or minimize the risk of sulfate attack. A comprehensive description of the different forms of sulfate attack and methods of prevention was recently published by Skalny et al. [23].

Without taking adequate precautions, concrete structures such as floors, foundations, drainage pipe, and lower parts of canal structures may completely disintegrate in only a very few years when exposed to water containing dissolved sulfates. The severity of the attack can be affected by the presence of other dissolved substances in the water, but generally increases as the concentration of sulfates in the water increases, and becomes even more severe if the concrete is subjected to frequently alternating periods of wetting and drying. On the other hand, concrete exposed to dry sulfate-bearing soils will not be attacked.

The source for the sulfates involved in the chemical reaction may be either external or internal to the concrete [23–30]. In the case of external attack, the sulfates may originate from groundwater or from sulfates leached from adjacent soil as the reaction progresses from the surface into the interior of the concrete. In the case of internal attack, the sulfates may come from minerals in the aggregates, sulfates dissolved in the mix water, admixtures and additives, and possibly from sulfates in the portland cement. It is therefore necessary to have a chemical analysis of the ingredients of the concrete and of the groundwater and soil surrounding the structure in order to assess the probability of sulfate attack.

The gypsum normally added to portland cement to control setting characteristics is not sufficient to cause sulfate attack. Portland cement normally contains gypsum, which is added in amounts of about 2.5–4 % as SO_3 to control setting characteristics. This gypsum is consumed during the normal course of the cement hydration process.

The main constituents of hardened portland-cement paste susceptible to sulfate attack are the hydration products of the tricalcium aluminate ($3CaO{\cdot}Al_2O_3$ or C_3A) phase. Other cement hydration products can also be attacked in more severe sulfate environments.

The major factors governing the sulfate reactions are (1) the permeability of the concrete to water, which is a control on the rate at which the reactants are brought together; (2) the composition of the hydrated phases of the cement paste, as not all cement hydration products are equally susceptible to reaction with sulfates; (3) the chemical composition and concentration of the sulfate-laden water, which affects the course and the severity of the reaction; and (4) the exposure conditions (i.e., concrete exposed to cyclic wetting and drying, or concrete exposed to moisture gradient will be more vulnerable than concrete continually immersed in sulfate-contaminated groundwater; temperature is also important). There are several different sulfate reaction mechanisms depending on the composition of the sulfate water and the environmental condition. The course of the reaction and the mechanism for deterioration may vary according to the situation. As the reaction proceeds inward into the concrete, the composition of the pore water may change and influence the course of the reaction.

Although sulfate attack has been extensively investigated and mechanistic aspects of the problem are known, it is still not completely understood. The situation is not as straightforward as might be expected since several different chemical reactions rather than a single reaction may be involved simultaneously. Some controversy still remains over the mechanism responsible for producing expansion and deterioration of the concrete. Most of the knowledge on sulfate attack has been developed from laboratory studies involving relatively simple chemical systems of pure materials, yet in actual practice the situation is more complicated as the exposures may involve varying and changing combinations of impure materials in different proportions. In such cases, the water may contain other aggressive anions and cations that attack hydrated cement constituents by chemical reactions other than those associated strictly with the sulfate anion. At times these reactions may impede the attack, and at other times may aggravate the attack.

Deterioration due to sulfate attack is generally attributed to chemical decomposition of certain portland-cement hydration products after hardening, and formation through solution and precipitation of less soluble reaction product [31–37]. This may produce expansive forces and a subsequent physical disruption of the concrete. Sulfate solutions may also react with the cement paste to form products that have little cementing value, and thereby turn the concrete into mush with little apparent change in volume [34,38].

The main reaction products involved in sulfate attack are calcium sulfoaluminates. Calcium sulfoaluminates form during the normal hydration of portland cement, and also form as reaction products of sulfate ion with calcium hydroxide and calcium aluminate hydrate. Calcium sulfoaluminate hydrate occurs in two forms, depending largely upon the supply of sulfate and alumina: a low sulfate form called calcium monosulfoaluminate or monosulfate ($3CaO{\cdot}Al_2O_3{\cdot}CaSO_4{\cdot}12H_2O$), and a high sulfate form called calcium trisulfoaluminate or ettringite ($3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}32H_2O$). The monosulfate form may be converted to ettringite when exposed to sulfate solutions, and the associated volume increase is potentially destructive.

Formation of **ettringite,** often from **monosulfate**, is the reaction that is considered the main destructive force in sulfate attack. However, there is some controversy over the formation of ettringite and the deterioration of the concrete [23,35,39,40]. The presence of ettringite alone is insufficient evidence of a destructive sulfate attack, as ettringite is also a secondary reaction product from the hydration of portland cement. If ettringite crystals are found in fractures, there may be a question of whether the fracture was caused by crystal growth itself (primary ettringite) or if the crystals happened to recrystallize into the space provided by the fractures (secondary ettringite). Ettringite as a normal cement hydration product is usually not considered to be destructive. There is also a lack of correlation between the amount of ettringite formed and the amount of expansion observed. There are some other unconfirmed theories: a) that there is more than one type of ettringite, and not all types are expansive [38,41,42]; b) that the mode of formation may also have an effect, that is, ettringite formed by solid-state reactions is more expansive than that formed by through-solution reactions [35,43]. However, according to Brown and Taylor, ettringite forms typically by through-solution rather than by topochemical mechanism [44]. Mechanisms for the expansion of ettringite have been reviewed by Cohen [45], Ping and Beaudoin [46], and others [23].

Thaumasite ($CaCO_3 \cdot CaSO_4 \cdot CaSiO_3 \cdot 15H_2O$) is structurally similar to ettringite and was observed by numerous authors in concrete attacked by sulfate solutions [47–57]. Thaumasite may form as a conversion product from ettringite by carbonate and silicon substitutions, or may form directly under favorable conditions when there is a supply of alumina, calcium silicates or free silica gel, sulfate, and carbonate [49,52]. It would appear that the presence of C_3A is not necessarily a prerequisite for thaumasite formation, and therefore such precautions as a Type V cement would not necessarily provide protection against the formation of thaumasite. Thaumasite forms preferentially at low temperatures due to the higher solubility of calcium salts at low temperatures, and appears to form rather quickly at temperatures around 5°C [49,52]. The carbonate ions can be derived from sources within the concrete such as limestone used as an aggregate or a cement addition or can be supplied as carbonate or bicarbonate ions dissolved in the soil or groundwater.

In some cases, other reaction products are formed such as **gypsum** ($CaSO_4 \cdot 2H_2O$) and silica gel. Gypsum is also considered by some to be an expansive reaction product, but there is some question as to whether the formation of gypsum produces the same disruptive effect as is attributed to ettringite formation [42]. Even so, gypsum formation under sulfate attack conditions is associated with producing a deterioration effect on concrete as it involves the conversion of cementitious constituents into products having little cementing properties [34].

The American Concrete Institute (ACI) divides sulfate attack into two general categories: (1) a sulfate reaction with calcium hydroxide to form gypsum (sometimes called gypsum attack, gypsum corrosion, and acid type of sulfate attack), and (2) a sulfate reaction with calcium aluminate hydrate to form ettringite (sometimes called sulfoaluminate attack or corrosion) [5]. In more severe forms of the reaction, other constituents of concrete are susceptible to sulfate attack, such as calcium silicate hydrate, and even some types of aggregate particles such as weathered feldspars. The reactions are more complex, however, as both anions and cations are involved.

The type of chemical reaction that will be prevalent is related, among other effects, to the cations associated with the sulfates in solution, which most commonly are Ca^{++}, Na^{+}, K^{+}, and Mg^{++}. The cations affect the solubility of sulfate minerals and the concentration of the sulfates in solution, and also the course of the reaction. With extremely high concentrations of sulfate, the reaction may intensify into acid attack that forms a different reaction product [10,42]. These reactions will be discussed in the following paragraphs. The course of the reaction and destructive effects are also affected by the presence of other dissolved constituents in the system that can tend to either ameliorate or intensify the destructiveness of the reaction. Sulfate attack is also affected by environmental factors such as ambient and internal temperature and wetting and drying, and frequency of their changes.

In addition to the chemical form of attack involving chemical reactions, the so-called "physical" form of sulfate attack is sometimes included under the general category of sulfate attack [23,58]. Crystallization pressures resulting from the precipitation of sulfate salts, such as Na_2SO_4 or $MgSO_4$, and subsequent growth of crystals, as may occur when concrete is exposed to sulfate solutions under wetting and drying conditions, can cause destruction of concrete [58–60]. This mechanism is employed in ASTM Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88).

In the view of some contemporary authors, *sulfate attack* involves all deleterious reaction mechanisms that involve sulfate ions. All such reactions are chemical in nature and, under certain processing and ambient conditions, may lead to compositional, microstructural, physical, and, finally, mechanical changes in the concrete material structure [23,61].

Internal sulfate attack should be then categorized as either a) composition induced internal sulfate attack, caused by an excess of sulfate ions in the concrete itself (from clinker, aggregate, admixtures, and rare excessive addition of calcium sulfate) or b) heat-induced internal sulfate attack (also referred to as *delayed ettringite formation* or DEF), caused by thermal decomposition and subsequent reformation of ettringite due to thermal conditions during concrete processing (high heat of hydration cement, high surface area cement, high ambient temperature during placing and curing, possibly affected by cement composition) [28–30]. This type of mechanism involves only monosulfate and ettringite (tri-sulfate) and will be discussed in the next section.

External sulfate attack relates to all chemical mechanisms of deterioration caused by external sources of sulfate anions, independent of the cation. Per this view, sulfate attack involves mechanisms such as a) reactions of sulfates with cement paste components leading to formation of gypsum (the so-called gypsum attack); b) expansion by primary ettringite formation (the "classical" sulfate attack characterized as sulfoaluminate attack); c) damage caused by magnesium sulfate—namely, a combined attack on calcium-silicate-hydrate and calcium hydroxide and involving the alumina-containing phases of the cement paste; d) thaumasite sulfate attack; and e) even the so-called *physical sulfate attack* of thenardite-to-mirabilite recrystallization, which is, in reality, a *chemical* process of repeated through-solution recrystallization leading to physical damage [23].

Calcium Sulfate Reaction

Calcium sulfate (most commonly occurring as gypsum, $CaSO_4 \cdot 2H_2O$) is not highly soluble in water, and sulfate attack on concrete in contact with soils containing gypsum may range from negligible to severe, depending upon the amount of gypsum dissolved in water in contact with the concrete. In concentrations approaching saturation (2000 mg/L water), calcium sulfate becomes reactive with calcium aluminate hydrates present in the system. The reactions of calcium sulfate with calcium aluminate hydrate ($4CaO \cdot Al_2O_3 \cdot 19H_2O$) and calcium monosulfoaluminate have been described [10,33,36,38,62–64], but the latter reaction (2) is today considered to be the primary deleterious reaction in the unlikely case when excess gypsum from cement is still present in the system:

$$\begin{gathered} 3(CaSO_4\cdot 2H_2O) + 4CaO\cdot Al_2O_3\cdot 19H_2O + 17H_2O \\ \rightarrow 3CaO\cdot Al_2O_3\cdot 3CaSO_4\cdot 32H_2O + Ca(OH)_2 \\ \text{gypsum + calcium aluminate hydrate + water} \\ \rightarrow \text{ettringite + calcium hydroxide} \end{gathered} \tag{1}$$

$$\begin{gathered} 2CaSO_4\cdot 2H_2O + 3CaO\cdot Al_2O_3\cdot CaSO_4\cdot 12H_2O + 18H_2O \\ \rightarrow 3CaO\cdot Al_2O_3\cdot 3CaSO_4\cdot 32H_2O \end{gathered} \tag{2}$$

$$\text{gypsum + calcium monosulfoaluminate + water} \rightarrow \text{ettringite}$$

Usually, when mature concrete is exposed to sulfate-bearing solutions, gypsum from cement has been already consumed in the normal hydration reactions between the calcium aluminate and alumino-ferrite phases present in most cements.

Under some conditions, such as periodical temperature changes leading to evaporation of water from the concrete surface, sulfates–independently of the cation they were originally associated with–may concentrate in the concrete matrix, especially close to evaporative surfaces, to dangerous levels even if the original concentration of sulfate in groundwater or soil were within the limits assumed to be safe.

Sodium Sulfate Reaction

Sodium sulfate is potentially more destructive than gypsum as it is more soluble (40 000 mg/L water) than calcium sulfate, and because it enters into two types of reactions with the hydrated cement phases in the hardened cement paste. Sodium sulfate reacts with calcium hydroxide as well as the calcium aluminate hydrate phases. At lower concentrations (SO_4 content less than 1000 mg/L), sulfate reacts with the hydrated calcium aluminate phases to produce ettringite and subsequent deterioration; at higher concentrations, sodium sulfate reacts with calcium hydroxide to produce gypsum [10]. Gypsum formation results in a volume increase, but there is some controversy as to whether the formation of gypsum produces a harmful expansion in concrete. The evidence for gypsum producing expansion and deterioration of the concrete does not appear as straightforward as does the case for ettringite formation [42].

The sodium sulfate reactions have been variously described [10,36,63,65,66].

$$\begin{gathered} Na_2SO_4 + Ca(OH)_2 + 2H_2O \rightarrow CaSO_4\cdot 2H_2O + 2NaOH \\ \text{sodium sulfate + calcium hydroxide + water} \\ \rightarrow \text{gypsum + sodium hydroxide} \end{gathered} \tag{3}$$

and

$$\begin{gathered} 3(CaSO_4\cdot 2H_2O) + 4CaO\cdot Al_2O_3\cdot 19H_2O + 8H_2O \\ \rightarrow 3CaO\cdot Al_2O_3\cdot 3CaSO_4\cdot 32H_2O + Ca(OH)_2 \\ \text{gypsum + calcium aluminate hydrate + water} \\ \rightarrow \text{ettringite + calcium hydroxide} \end{gathered} \tag{4}$$

$$\begin{gathered} 2Na_2SO_4 + 3CaO\cdot Al_2O_3\cdot CaSO_4\cdot 12H_2O + 2Ca(OH)_2 \\ + 2H_2O \rightarrow 3CaO\cdot Al_2O_3\cdot 3CaSO_4\cdot 32H_2O + 4NaOH \\ \text{sodium sulfate + calcium monosulfoaluminate} \\ \text{+ calcium hydroxide + water} \rightarrow \text{ettringite} \\ \text{+ sodium hydroxide} \end{gathered} \tag{5}$$

Magnesium Sulfate Reaction

Magnesium compounds are potentially more destructive to concrete as the magnesium ion is capable of completely replacing the calcium in hydrated portland cement. Magnesium sulfate is highly soluble (70 000 ppm) and can form more highly concentrated sulfate solutions than sodium sulfate. Strong solutions of magnesium sulfate are capable of reacting with the calcium silicate hydrate phases as well as calcium hydroxide and calcium aluminate hydrate phases. The concentration of magnesium sulfate may affect the course of the reaction [10]:

- At low concentrations (less than 3200 mg SO_4/L–or less than 4000 mg $MgSO_4$/L), the attack is characterized by ettringite formation. However, in the continued presence of $MgSO_4$, ettringite is eventually decomposed to gypsum, magnesium hydroxide, and hydrated alumina [36].
- At intermediate concentrations (between 3200 and 6000 mg SO_4/L or 4000 to 7500 mg $MgSO_4$/L), the reaction is characterized by ettringite and gypsum formation. Deterioration and cracking may slow down and may be hardly perceptible.
- At high concentrations (above 6000 mg SO_4/L or above 7500 mg $MgSO_4$/L), the reaction is characterized by the formation of magnesium hydroxide, gypsum, and silica gel; ettringite does not form. Deterioration becomes very severe as the magnesium concentration increases.

The various magnesium sulfate reactions have been described elsewhere [10,34,36,64,67].

$$\begin{gathered} MgSO_4 + Ca(OH)_2 + 2H_2O \rightarrow CaSO_4\cdot 2H_2O + Mg(OH)_2 \\ \text{magnesium sulfate + calcium hydroxide + water} \\ \rightarrow \text{gypsum + magnesium hydroxide} \end{gathered} \tag{6}$$

$$\begin{gathered} 3MgSO_4 + 3CaO\cdot Al_2O_3\cdot 6H_2O + 6H_2O \\ \rightarrow 3(CaSO_4\cdot 2H_2O) + 3Mg(OH)_2 + 2Al(OH)_3 \\ \text{magnesium sulfate + calcium aluminate hydrate} \\ \rightarrow \text{gypsum + magnesium hydroxide} \\ \text{+ aluminum hydroxide} \end{gathered} \tag{7}$$

$$\begin{gathered} 3MgSO_4 + 3CaO\cdot Al_2O_3\cdot CaSO_4\cdot 12H_2O \\ + 2H_2O \rightarrow 4(CaSO_4\cdot 2H_2O) \\ + 3Mg(OH)_2 + Al_2O_3\cdot 3H_2O \\ \text{magnesium sulfate + calcium monosulfoaluminate} \\ \text{hydrate} \rightarrow \text{gypsum + magnesium hydroxide} \\ \text{+ aluminum hydroxide} \end{gathered} \tag{8}$$

$$\begin{gathered} 3MgSO_4 + 3CaO\cdot 2SiO_2\cdot nH_2O + zH_2O \\ \rightarrow 3(CaSO_4\cdot 2H_2O) + 3Mg(OH)_2 + 2SiO_2\cdot xH_2O \\ \text{magnesium sulfate + calcium silicate hydrate +} \\ \text{water} \rightarrow \text{gypsum + magnesium hydroxide + silica gel} \end{gathered} \tag{9}$$

Magnesium silicate hydrate may also be produced from this reaction. Magnesium hydroxide may have a beneficial effect as it is highly insoluble and forms surface deposits, which tend to block pores and hinder the further penetration of sulfate solutions into the concrete, but one should not rely on this process as a prevention against sulfate attack.

Thaumasite Form of Sulfate Attack

Thaumasite is a calcium-silicate-sulfate-carbonate hydrate ($CaSiO_3 \cdot CaCO_3 \cdot CaSO_4 \cdot 15H_2O$) that can form in concrete by reaction of the calcium-silicate hydrates (C-S-H) with sulfates in the presence of carbonate ions [23]:

$$3Ca^{2+} + SiO_3{}^{2-} + CO_3{}^{2-} + SO_4{}^{2-} + 15H_2O \rightarrow 3CaO \cdot SiO_2 \cdot CO_2 \cdot SO_3 \cdot 15H_2O$$

The process only occurs in very wet environments and the rate of formation is increased at low temperatures. It should be noted that in all of the laboratory and field cases reported, the sulfates for this reaction come from external sources. The conversion of C-S-H into thaumasite results in a loss of binding material and hence the integrity of the concrete (or other cement-based material). The thaumasite form of sulfate attack (TSA) is a relatively rare form of sulfate attack and risk of its occurrence is thought to be negligible unless the following conditions are met [68]:

- Presence of sulfates and/or sulfides in the ground,
- Presence of mobile groundwater,
- Presence of carbonate, generally in the concrete aggregates,
- Low temperatures (generally below 15°C).

However, a series of cases were recently presented in which TSA had occurred without all four of the above conditions being fully met [69], and other workers have provided evidence that TSA may occur in concrete containing siliceous aggregates [70], in concrete stored at temperatures above 15°C [71], and possibly even concrete with no access to external sulfate [72]. Also, in some cases the source of carbonate has been identified as acidic, carbonate-bearing groundwater [73], or even seawater [74]. It has been argued that the apparent scarcity of TSA is partly attributed to the following: 1) the conditions of most standardized sulfate resistance tests do not favor thaumasite formation, 2) forensic examinations may have failed to detect TSA in deteriorated concrete, and 3) buried concrete is rarely inspected [75]. For example, TSA was recently diagnosed as the cause of damage to an aqueduct where previous investigations had wrongly attributed the deterioration to classical sulfate attack involving ettringite formation [76].

Other Reactions

The most common types of sulfate attack in natural environments are from calcium, sodium, magnesium, and occasionally potassium sulfate. Potassium sulfate reacts with concrete in a manner similar to that of sodium sulfate. In some industrial situations, concrete may be exposed to other types of sulfate solutions and to bisulfate solutions ($HSO_4^{=}$). Bisulfate solutions tend to be more acidic and can contribute to acid attack as well as sulfate attack. The aggressiveness of various sulfate salts towards concrete is partly related to solubility in water, in that sulfates of very low solubility (barium sulfate and lead sulfate) are relatively harmless. Ammonium sulfate is highly soluble in water (~800 g/L) and is highly aggressive towards concrete. Sulfates of copper, manganese, aluminum, iron, cobalt, and zinc, which are moderately soluble in water, are also aggressive towards concrete [77,78].

In low concentrations, chlorides have been shown to decrease the sulfate resistance of concrete [79,80], yet in high concentrations chloride is thought to temper sulfate attack as chloride increases the solubility and decreases the stability of ettringite and gypsum [10,36,41,81]. Chloride ions react with hydrated aluminates and monosulfoaluminate to form chloroaluminates.

There is conflicting evidence concerning the effects of solutions containing mixtures of sulfate salts and other dissolved substances. In some cases, sulfate solutions containing chloride, magnesium, sodium bicarbonate, and carbon dioxide are highly aggressive towards concrete, yet seawater, which contains these substances, is not as aggressive as might be expected based upon magnesium and sulfate contents [36,63,69,82].

Control of External Sulfate Attack

The widespread occurrence and destructiveness of sulfate attack led to many investigations over the years into the mechanism for the deterioration and means to combat the reaction. There have been many studies in many parts of the world and an extensive body of knowledge on sulfate attack has been developed. It is not possible to adequately recognize the many contributions and only a few of the contributions can be mentioned in passing.

Some of the earlier studies in the United States include those conducted by the Metropolitan Water District [31], the U.S. Department of Agriculture and the University of Minnesota [83], the Portland Cement Association [32,34,84–86], the California Division of Highways [45], the Bureau of Standards [87], the Corps of Engineers [88,89], the Bureau of Reclamation [79,90–93], and at the University of California for the Bureau of Reclamation [94].

Extensive studies have been conducted elsewhere in the world such as in Canada, England, France, Russia, South Africa, Australia, Belgium, Sweden, Hungary, and Israel (for example, see Refs 10, 33–36, and 95–98 for brief summaries, and more recently Refs 23, 27, 37, 38, 40, 42, 66, and 99–109).

Most of these investigations seem to reach (or confirm) the same conclusions on the resistance of concrete to sulfate attack:

The four main strategies for improving resistance to sulfate solutions are (1) making a high quality, impermeable concrete; (2) preventing or limiting, or both, the access of sulfate solutions to concrete; (3) using a sulfate-resistant cement (limitations mentioned above); and (4) using pozzolans or slag [5]. If access of sulfate cannot be easily prevented, the service life of concrete also may be increased by protecting the concrete from exposure to sulfates by limiting the sulfate content of the materials used to make the concrete and using impermeable barriers to prevent sulfate solutions from coming into contact with the concrete. Impermeable barriers are not recommended as a long-term solution to an aggressive sulfate condition as there is no guarantee the barriers will perform effectively over a long term, and may even aggravate the situation.

It is essential to have a high-quality impervious concrete to improve sulfate resistance [104,105,110–112]. This involves using a low water-cement ratio and a reasonably high cement content in the mix, the use of pozzolans or slag (or both), and also good workmanship and construction techniques. This includes good mixture proportioning and selection of materials, consolidation of the concrete during placement, and good finishing and curing to produce a hard, uncracked and dense surface.

The sulfate resistance of portland cements may be improved by limiting the C_3A content in the cement clinker, by limiting the amount of $Ca(OH)_2$ formed in the hydrated cement paste, or by converting the $Ca(OH)_2$ to a more stable form either as C-S-H or $CaCO_3$. Carbonation of the surface layer, and air entrainment also may aid by reducing the permeability of concrete and improving sulfate resistance

[87,113–115], but this should not be used alone as a prevention technique. The use of calcareous fillers has been reported as having a beneficial effect due to a reaction with C_3S to form carboaluminate ($C_3A{\cdot}CaCO_3{\cdot}12H_2O$) [116]. It may be noted that the development of high alumina cements (which contain calcium aluminates in forms other than C_3A) was in part stimulated by a search for sulfate-resistant cements. The high alumina cements generally have very good sulfate resistance [36,117]. These cements are rarely used today for sulfate resistance as there are other issues involved.

One means to reduce the C_3A content of the cement is to convert the C_3A phase to C_4AF and CF by the addition of iron oxide during the cement manufacturing process. But, as mentioned above, the hydrated ferrite phases are ultimately susceptible to sulfate attack in severe environments, the relevant specifications for highly sulfate-resistant cements place a limit on the total amount of C_3A and C_4AF + CF. Again, this is of no help in the presence of magnesium sulfate.

Although the deterioration generally increases as the amount of tricalcium aluminate (C_3A) in the cement increases, some exceptions have been noted. In some cases, low C_3A cements were observed to have poor sulfate resistance, some moderately high C_3A cements were observed to have good sulfate resistance, and in several instances zero C_3A cements have shown poor sulfate resistance [37,96]. This is most probably related to the reactivity of alumina present in other hydrated phases, such as those formed by hydration of C_4AF (calcium aluminate ferrite). Please note that use of ASTM Type V sulfate-resisting cements is not a remedy against sulfate waters containing magnesium cations ($MgSO_4$).

The $Ca(OH)_2$ content of the hardened cement paste can be controlled to a certain extent by increasing the amount of C_2S and decreasing the C_3S phase in the portland cement as the C_2S phase liberates less $Ca(OH)_2$ than C_3S during hydration; however, production of low-C_2S cements is uncommon and the evidence does not clearly indicate that increasing the relative proportion of C_2S with respect to C_3S is effective in controlling sulfate resistance [36].

The $Ca(OH)_2$ formed during the hydration of portland cement can be converted to a more stable C-S-H form through a chemical reaction with reactive forms of SiO_2. This can be accomplished by adding finely ground SiO_2 to the mix followed by high pressure steam curing at 100°C or higher, or by combining portland cement with active siliceous mineral admixtures such as natural or calcined pozzolans, fly ash, silica fume, or slag. In regards to heat-treated or low-temperature steam-cured concrete (around 75 or 80°C), it should be noted that excessive heat may lead to delayed formation of ettringite (DEF), which in some cases has been proven to cause deterioration in precast and other concrete products [26,28–30,118–121].

Pozzolans and slag improve sulfate resistance by chemically reacting with $Ca(OH)_2$ to form C-S-H and by reducing the permeability of the concrete. Slags have been shown to be effective when substituted for at least 50 % of the cement as long as the Al_2O_3 content is limited [122]. However, the addition of a pozzolan or slag does not automatically guarantee sulfate resistance, and tests should be made to determine effectiveness in improving sulfate resistance.

There are some apparent inconsistencies regarding the effectiveness of pozzolans in improving sulfate resistance. Not all pozzolans are equally effective in improving sulfate resistance. The apparent inconsistencies are due in part to differences in the composition, chemical reactivity (e.g., thermal history), and fineness of the pozzolans, and also to the amount of pozzolan and the amount and type of cement in the mixture. Some differences in performance may depend upon whether a particular pozzolan is used as a replacement or as an addition to the cement. Other differences may arise depending upon the criteria selected to base performance (comparison with other mixes or against an absolute scale).

A pozzolan that meets a specification such as ASTM C 618 does not guarantee effectiveness for sulfate resistance [123], but at the risk of over simplification, the following generalizations may be made regarding the effectiveness of pozzolans classified according to ASTM C 618 for improving sulfate resistance:

- Class N pozzolans are variable but generally good especially those of higher reactivity such as metakaolin [124].
- Most Class F fly ashes generally improve the sulfate resistance of Type II and Type V cements, and generally are more efficient in improving sulfate resistance than Class C ashes [125–128]. However, some ashes with high alumina content are not effective in improving sulfate resistance. The influence of the alumina in fly ash will depend on whether it is available for reaction with sulfate (e.g., alumina present as C_3A or in glass) or whether it is tied up in stable crystalline compounds (e.g., mullite).
- Low calcium Class C fly ashes (e.g., 10–20 % CaO) are often good; but high calcium Class C (e.g., > 20 % CaO) ashes are variable, and may reduce sulfate resistance by introducing additional calcium ions into the system [93,125,127,129–131]. In general, the efficacy of fly ash in terms of increasing sulfate resistance increases as the calcium content of the fly ash decreases [132]. Some Class C ashes must be used at replacement levels greater than 75 % to achieve sulfate resistance or may be used at moderate levels in combination with silica fume [127,133].
- Silica fume is generally very effective in improving sulfate resistance, perhaps even more so than fly ash or ground slag [128,134–137].

Two general approaches have been taken for the specification of sulfate-resistant cements: (1) base specification on the chemical composition of the cement such as ASTM Specification for Portland Cement (C 150); or (2) performance tests such as ASTM Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution (C 1012), ASTM Test Method for Potential Expansion of Portland-Cement Mortars Exposed to Sulfate (C 452), or Bureau of Reclamation (USBR 4908) Procedure for Length Change of Hardened Concrete Exposed to Alkali Sulfates. There are some cases where a specification based on chemical composition alone would exclude a sulfate-resistant cement. In these cases, performance tests may be specified in lieu of a chemical composition specification. Performance tests should be specified for combinations of portland cements and pozzolans or slags.

Portland cement with a C_3A content below 5 % is generally considered to be sulfate resistant [34,106]. ASTM C 150 designates Type II cements containing no more than 8 % C_3A as having moderate sulfate resistance and Type V cements (containing less than 5 % C_3A and 25 % C_4AF + 2(C_3A) or C_4AF + C_2F) as having high sulfate resistance. However, it needs to be stated that the designated type of cement by itself does not guarantee sulfate resistance. Sulfate resistance cements should be considered to be a secondary defense against sulfate waters, the primary being good quality, low-permeability concrete matrix. Also, as mentioned earlier, under some environmental and processing circumstances–such as excessively high heat input or presence in ground water of magnesium sulfate–using sulfate-resisting type of cement is useless.

Studies have been made for the development of specifications for sulfate-resistant fly ashes. These have included - specifications based on chemical composition and mineralogical composition [138]. For example, an "*R* value" based on chemical composition of fly ash (where $R = \%CaO - 5 \div \%Fe_2O_3$) has been proposed as a method to estimate sulfate resistance [123,139,140]. For replacement mixed in the 15–25 % range, *R* values below three indicate improved sulfate resistance. The *R* values has been questioned as it does not directly take into account the alumina content of the fly ash; however, the *R* value indirectly allows for alumina since alumina combined with lime is available for attack and not when combined with iron [140]. *R* values are not commonly used today.

The recommendations for sulfate resistance developed originally by Tuthill are fairly well accepted and can be updated as follows [31,141]:

- When long-range durability of concrete is at stake in an aggressive environment, it is more important to specify performance limits that will provide the most sulfate resistance, rather than to require compositional limits on ingredients that most manufacturers can not meet.
- Use as low as possible w/c ratio. A w/c ratio below 0.50 is recommended for mild exposures, 0.45 or below for moderate exposure, and 0.40 or below for severe and extremely severe exposures [4,5,80]. At very low w/c ratios, the effects from the composition of the cement are less apparent, which has to be taken into consideration if magnesium sulfate in aggressive water is an issue [83].
- Use a portland cement with the lowest possible C_3A content [83,142]. Consider the availability of alumina originating from the hydrated ferrite phases.
- Use ample amounts of cement. The lower the C_3A content of the cement and the richer the mix, the better will be the resistance of concrete to sulfate attack. An additional sack of Type V sulfate-resistant cement will increase resistance at least 50 %. However, an additional sack of Type II cement will not provide additional sulfate resistance equal to that provided by Type V cement with 4.5 % C_3A, unless the Type II cement contains less than 5.5 % of C_3A. Extending the curing period provided prior to sulfate exposure also improves sulfate resistance.
- If a pozzolan or slag is to be used, the amount required with a particular cement to produce the desired level of sulfate resistance should be determined by appropriate testing (e.g., ASTM C 1012).
- A low-lime and low-aluminate cement, having less than 50 % C_3S and less than 12 % $C_3A + C_4AF$, in which less than 4 % is C_3A, makes the most sulfate-resistant portland cement assuming the concrete quality is good.
- Air entrainment is generally recognized as being beneficial to sulfate resistance, especially when air entrainment is used to advantage in reducing the water-cement ratio [113,114,128].
- Precast units such as concrete pipe and block can be made appreciably more resistant by introducing a period of several weeks of drying following good curing. Presumably, carbonation occurring during this period contributes to improved sulfate resistance, as it may reduce permeability.
- Steam curing at high temperatures may increase the sulfate resistance of concrete if the concrete is dense and uncracked [83,96,143]. However, high heat input (ambient temperature, processing temperature, high cement surface area-heat of hydration) may lead to internal sulfate attack problems (see section on DEF).

Two aspects of controlling sulfate attack are identification of the severity of the exposure conditions and selection of appropriate precautionary measures. Table 1 [5] shows the severity of sulfate attack to be expected from the sulfate concentrations encountered in both soil and in water, and recommended precautionary measures. These recommendations are generally consistent with those given by the Bureau of Reclamation [80], the Corps of Engineers, and the Portland Cement Association [128], but have yet to be fully adopted by the ACI 318 Building Code [144].

The table is intended for use in concrete construction under ordinary service life conditions, but may not necessarily be reliable for the ultimate protection of concrete in exposures to exceptionally severe conditions such as magnesium-sulfate exposures, high temperature exposures, or for mega year service life, such as may be encountered in severe industrial exposures or hazardous waste containment [42,145]. For example, water containing magnesium sulfate, especially in concentrations of more than 6000 ppm SO_4 (equivalent to 0.75 % $MgSO_4$), is capable of attacking all portland cement hydration products regardless of cement type or combination of pozzolan or slag [10,37]. In this case, even more than in other cases, the quality of concrete rather than the cement type is crucial. Good judgment must be exercised as the table lists only the concentration of the sulfate anion (SO_4) as a criterion for potential severity of sulfate attack and does not include cations that also have an effect on the conditions and severity of the reaction. It is also possible to have cases where a potentially destructive sulfate attack condition may exist even though a sulfate analysis indicates the groundwater or soil to have a low sulfate content. These are situations where the exposure conditions may cause the water in contact with the structure to become more concentrated in sulfates due to ponding and evaporation, or where under severe and repeated wetting and drying conditions, water may evaporate and crystals of sulfate salts may crystallize, decompose and re-crystallize, leading to physical destruction of the concrete matrix [34,42,59,60,146].

Tests for Sulfate Resistance

It should be noted that there is no perfect test for sulfate attack; the known sulfate reaction mechanisms are numerous and complex and they may occur simultaneously. For the same reason, there cannot exist a single test that applies to all situations. As discussed above, the best protective measure is quality concrete, thus testing of cements and other concrete ingredients is important but of only limited value.

Tests for sulfate resistance are made to study the sulfate resistance of different cements and various combinations of materials. These tests include field exposures under natural conditions, and laboratory tests of concrete or mortar specimens exposed to artificial and generally accelerated exposure conditions. As the exposure conditions vary, some differences in the rate and severity of attack may be expected. Laboratory tests involve exposing mortar or concrete specimens to sulfate, either internally by adding the sulfate to the mixture, or externally by soaking the specimen in a solution containing sulfate. Sulfate exposure conditions are accelerated

TABLE 1—Requirements to Protect Against Damage to Concrete by Sulfate Attack from External Sources (ACI 201.2R-01)

Severity of potential exposure	Water-soluble sulfate (SO_4)[1], %	Sulfate (SO_4)[1] in water, ppm	w/cm by mass, max. [2,3]	Cementitious material requirements
Class 0 exposure	0.00 to 0.10	0 to 150	No special requirements for sulfate resistance	No special requirements for sulfate resistance
Class 1 exposure	$>$0.10 and $<$0.20	$>$150 and $<$1500	0.50	C 150 Type II or equivalent[4]
Class 2 exposure	0.20 to $<$2.0	1500 and $<$10 000	0.45	C 150 Type V or equivalent[4]
Class 3 exposure	2.0 or greater	10 000 or greater	0.40	C 150 Type V plus pozzolan or slag[4]
Seawater exposure	-	-	See section 2.4 (ACI 201.2R-01)	See section 2.4 (ACI 201.2R-01)

[1] Sulfate expressed as SO_4 is related to sulfate expressed as SO_3, as given in reports of chemical analysis of portland cements as follows: SO_3 % × 1.2 = SO_4 %.

[2] ACI 318, Chapter 4, includes requirements for special exposure conditions such as steel-reinforced concrete that may be exposed to chlorides. For concrete likely to be subjected to these exposure conditions, the maximum *w/cm* should be that specified in ACI 318, Chapter 4, if it is lower than that stated in Table 2.3.

[3] These values are applicable to normal-weight concrete. They are also applicable to structural lightweight concrete except that the maximum *w/cm* ratios 0.50, 0.45, and 0.40 should be replaced by specified 28 day compressive strengths of 26, 29, and 33 MPa (3750, 4250, and 4750 psi), respectively.

[4] For Class 1 exposure, equivalents are described in Sections 2.2.5, 2.2.6, and 2.2.9. For Class 2 exposure, equivalents are described in Sections 2.2.5, 2.2.7, and 2.2.9. For Class 3 exposure, pozzolan, and slag recommendations are described in Sections 2.2.5, 2.2.8, and 2.2.9 (of ACI 201.2R-01).

by using high sulfate concentrations, by using magnesium sulfate solutions, or by using a wet-dry exposure cycle. A variety of test methods have been devised; none of them is absolute [10,26,90,106,115,147–149].

ASTM C 452 involves adding gypsum to a mortar bar (1 by 1 by 10-in. or 25 by 25 by 250-mm size prism) in amounts so that the total sulfur trioxide content of the specimen is 7 % by mass. The specimens are stored in water and length changes are measured over time. The test is considered suitable for portland cements, but not for blended cements or blends of portland cement with pozzolans or slags. Results of this test generally conform with the long-term behavior of cements in concrete exposed to aggressive sulfates and with predictions based on the C_3A content of the cement. This test is used to establish that a sulfate-resisting portland cement meets the performance requirements of ASTM C 150. The sulfate resistance of concrete is not guaranteed, however, if the concrete is of inferior quality.

ASTM C 1012 involves expansion of mortar specimens in a 5 % Na_2SO_4 solution (50 g/L) at 23°C (73.4°F) and measuring length change. The test is considered suitable for portland cements, blends of portland cements with pozzolans or slags, and blended hydraulic cements. The test does not simulate sulfate attack by solutions of sulfate compositions other than that used, and if evaluation of behavior in exposure to a sulfate solution of a given composition is desired, then that solution should be used in the test.

The Bureau of Reclamation Procedure 4908 [150] describes three methods for testing 3 by 6-in. (76 by 152-mm) cylindrical specimens. Method A consists of continuous soaking of test specimens in a 2.1 % Na_2SO_4 solution at room temperature; Method B consists of continuous soaking of test specimens in a 10 % Na_2SO_4 solution at room temperature; and Method C consists of alternating wetting and drying tests of specimens soaked in a 2.1 % Na_2SO_4 solution. Methods B and C produce failure in about one-sixth the time required in Method A. An expansion of 0.5 % is considered failure in the test. A failure criterion of a 40 % reduction in dynamic modulus of elasticity has also been used.

These test methods are irrelevant if the reasons for sulfate attack are high-heat processing of concrete or other specific situations, such as the thaumasite destruction mechanism or magnesium sulfate exposure.

Internal Sulfate Attack

In the case of internal attack, the sulfates may come from minerals in the aggregates, sulfates dissolved in the mix water, admixtures and additives, and possibly from sulfates in the portland cement or supplementary cementing materials. Internal sulfate attack may be induced by either (1) an excess of sulfate ions in the concrete (contributed by one or more of its ingredients) or (2) exposure to an elevated temperature (usually at early age) leading to thermal decomposition and subsequent reformation of ettringite. The latter process is commonly referred to as delayed ettringite formation (DEF). The presence of excessive sulfates in the concrete can be prevented by the appropriate selection and testing of the materials used to produce concrete. Prevention of delayed ettringite formation requires either control of the manufacturing and curing processes, or the materials used for concrete production, or both.

Delayed Ettringite Formation

The term delayed ettringite formation, or DEF, was first used to describe a form of deterioration in heat-cured precast concrete railway ties in the early 1980s in Germany [151]. It was postulated that the normal early formation of ettringite was postponed as a result of exposure to excessive temperatures during manufacture and that ettringite formed during subsequent exposure to moisture in service. This delayed formation of ettringite in the hardened concrete led to deleterious expansion and cracking of the concrete. Since this time DEF has been implicated as a cause of deterioration in numerous other cases. Most of these cases have involved precast concrete elements that have been subjected to heat curing during the production process. For some reason, railway ties appear to be particularly susceptible to this form of deterioration with cases involving ties being reported in Germany [119,151,152], Finland [118], the former Czechoslovakia [153], Canada, the United States [154], South Africa [155], and Australia [120,156].

The occurrence of these problems and the apparent role of elevated-temperature curing have led many countries to impose restrictions on the heat-curing process used during the manufacture of precast concrete. These restrictions include limits placed on pre-set times, rates of heating and cooling, and the maximum allowable concrete (or curing) temperature. Experience with the German recommendations dates back to 1977 and there is evidence that these and similar practices in Europe have been successful in eliminating damage due to DEF [157,158].

It has also been claimed that concrete that has not been exposed to elevated temperatures may suffer deterioration due to so-called "ambient-temperature" DEF [154,159–161]. Such cases apparently involve certain modern-day high-sulfate clinkers, which reportedly result in a slow or late release of sulfate over time (i.e., long after the concrete has hardened). All of the evidence to support this viewpoint comes from investigations of damaged nonheat-cured precast and cast-in-place concrete elements where it has not been possible to prove unequivocally that DEF is the primary cause of deterioration or that the concrete had not been exposed to elevated temperature either deliberately or adventitiously (due to autogenous temperature rise effects). To date, there is no laboratory evidence to confirm that damage due to DEF can occur without an excursion to elevated temperature (i.e., somewhere above 60–70°C).

A number of comprehensive reviews on the DEF phenomenon have been published recently [27–30,162–164]. The mechanisms of DEF are now fairly well established as are means for minimizing the risk by controlling the maximum temperature reached within the concrete or by incorporating pozzolans or slag [151,162,164–171].

The solubility of ettringite increases with temperature and pH, and exposure to elevated temperatures increases both the sulfate and alumina concentration in the pore solution of concrete. Much of this sulfate and alumina becomes encapsulated by the rapidly forming calcium-silicate hydrates (C-S-H). Immediately after the early excursion to elevated temperature, little or no ettringite is detected in the concrete, and poorly crystalline monosulfate appears to be the main sulfate-bearing phase [164]. During subsequent exposure to moisture at normal ambient temperatures most of the sulfate, but only a small amount of the alumina, is released by the C-S-H. The increased availability of sulfate results in a conversion of the monosulfate into ettringite. This process may occur many months or years following the exposure to elevated temperature, hence the term "delayed ettringite formation." This delayed formation of ettringite may under some circumstances result in expansion of the paste and consequent cracking of the concrete. It is now generally believed that the paste expansion results from the growth of ettringite crystals in the very small pores (~ 100 nm) of the cement paste [164,172]. The expansion of the cement results in the formation of gaps around aggregate particles and cracking of the cement paste [173]. Ettringite eventually reprecipitates into these gaps and the cracks, but this process is harmless being a symptom of the DEF process rather than a cause of damage [173].

A number of workers have attempted to develop models to predict the expansion due to DEF from the composition of the portland cement [119,151,163,165–167,174]. However, it has not been possible to extend the models developed for one set of cements and test conditions to the results of other studies using different materials and test conditions [164].

It has been demonstrated by numerous studies that expansion due to DEF cannot be induced in the laboratory without an excursion to an elevated temperature [162,165–169]. The precise temperature necessary to produce expansion varies between studies and is likely affected by the composition of the cement (particularly alkali content) and, possibly, other material and production parameters. Most studies have indicated that a temperature somewhere above 70°C is required; although expansion has been observed both in the field and the laboratory at somewhat lower temperatures (~ 60°C) with high-alkali cements [168,175].

Damaging expansion can be prevented in mortars and concretes cured at temperatures up to at least 95°C by the incorporation of sufficient quantities of pozzolans or slag [151,168,170,171]. Fly ash, slag and natural pozzolans (e.g., metakaolin) are all effective in this role whereas silica fume is not. It appears that the alumina content of the pozzolan is artly responsible for mitigating the damaging effects of DEF [168–171].

Acid Attack

As concrete is an alkaline material, it is subject to deterioration in exposure to acid solutions. Fortunately, most natural waters are neutral or only slightly acidic and do not present a problem. However, natural waters can become acidic, either through natural processes or from contamination, and attack concrete. Natural processes for acidifying water include the decay of organic matter in marshes and swamps, and absorption of gases and pollutants from the atmosphere such as CO_2 and SO_2. Natural waters can also be contaminated by acid mine drainage, and agricultural and industrial wastes. In industrial exposures, concrete may be exposed to a wide variety of acidic solutions.

Acid solutions attack concrete through dissolution and acid-base chemical reactions. The reaction attacks concrete on the exposed surface and works inward, and generally involves removal of soluble reaction products. The acid attacks cement hydration products and, in some cases, may also attack certain types of aggregate particles. The hydration product most susceptible to reaction with acid is $Ca(OH)_2$, which is readily attacked and dissolved by even relatively mild acid solutions. More aggressive acid solutions react with other cement hydration products, and ultimately all cement hydration products may be attacked. As an example, ASTM Test Method for Portland Cement Content of Hardened Hydraulic-Cement Concrete (C 1084) is based on dissolution of all hydrated cement paste in hydrochloric acid. Certain types of aggregates, such as limestone and dolomite that contain carbonate minerals, react with acids.

The two main factors involved in the chemical reactivity of an acid are the concentration and strength of the acid.

The concentration of a particular acid solution refers to the weight of acid per unit volume of water and is commonly expressed either in terms of normality or molarity.

The strength of an acid is determined by its tendency to dissociate in water and form protons (H^+ ions). The intrinsic property of a particular acid to dissociate in water is indicated by the dissociation constant, K_a, for the acid, which also may be expressed in terms of pK_a (defined as the negative logarithm of the equilibrium constant, K_a, for the acid, $pK_a = -\log K_a$). Strong acids have a large dissociation constant and a small pK_a. Strong acids ionize completely in water; weak acids do not completely ionize. For example, battery acid is concentrated H_2SO_4, is highly ionized ($pK_a = 1.92$), and is a strong acid. Vinegar is dilute acetic acid, dissociates to a much lesser extent ($pK_a = 4.5$), and is a weak acid.

The chemical reactivity of an acid is due to the hydrogen ion (H^+), and reactivity increases as the H^+ concentration increases. The H^+ concentration of a particular solution is related to both the strength and concentration of the acid in the solution, and is commonly expressed in terms of pH (originally defined as equal to the negative logarithm of the H^+ concentration). A 1-normal solution of a strong acid (one gram equivalent weight of a completely dissociated acid per litre of water) theoretically has a pH of 0, and a 0.1-normal solution has a pH of 1. A neutral solution has a pH of 7. A decrease of a full pH unit represents a 10 times increase in the concentration of the acid, that is, an acid solution of pH 4 is 100 times more concentrated than an acid solution of pH 6.

pH measurements are commonly used as an indicator of the aggressiveness of a solution, but may be misleading. For example, it is possible for a dilute solution of a strong acid to have the same pH as a concentrated solution of a weak acid. Both solutions may initially attack concrete at about the same rate, but on an equal volume basis, a given volume of the weak acid ultimately will be more destructive to concrete than an equal volume of the stronger acid as the weaker solution contains more acid [77].

The rate and extent of attack is governed by several factors in addition to the strength and concentration of acid. These factors include exposure conditions (exposure to static or running solutions), the transport of the acid into the concrete (diffusion and permeation), the nature of the reaction product formed, and in some cases, the rate at which the reaction product is removed. Flowing solutions are more aggressive than static solutions as more acid is brought into contact with the concrete and as the flowing solutions tend to remove the reaction products. The extent of the attack is governed by the rate the acid penetrates into the concrete. Acids that form soluble reaction products are generally more aggressive than acids that form insoluble reaction products. Most acids react to form soluble products; oxalic and phosphoric acids are exceptions and form insoluble reaction products that are not readily removed. Insoluble reaction products, such as SiO_2 gel, $CaCO_3$, or $CaSO_4$, may tend to seal the concrete surface from the acid and impede further reaction of the acid with the concrete.

Hydrochloric acid (muriatic acid), nitric acid, and sulfuric acid are among the more common acids and are highly reactive with concrete. Sulfuric acid is particularly aggressive since the sulfate ions it contains are also available for sulfate attack reactions. Dilute hydrochloric acid has been used to clean concrete surfaces and for architectural purposes in making exposed aggregate surfaces; however, hydrochloric acid is not recommended for these purposes as the reaction is difficult to control and as it is difficult to make sure the surfaces have been washed clean and all acid removed. Organic acids range from highly to slightly reactive. Lactic acid, acetic acid (vinegar), citric acid, and tartaric acid are highly to moderately reactive and oxalic acid is only slightly reactive. Oxalic acid is sometimes beneficial as it has been used as a floor treatment to improve the resistance of the floors to other weak organic acids. The literature contains tabulations on the exposure of concrete to various acids [4,36,77,176].

The leaching of concrete ascribed to soft water is largely due to carbonic acid (H_2CO_3) which forms when the water absorbs carbon dioxide (CO_2) from the atmosphere. Such waters are quite aggressive towards concrete [9,10,12,177,178]. The effect of CO_2 on the pH of soft water is quite complex [179,180]. The amount of CO_2 that can be dissolved in water is directly proportional to the partial pressure of CO_2 in the atmosphere and inversely proportional to temperature. As the dissolved CO_2 content of the water increases, the CO_2 combines with water to form carbonic acid. Carbonic acid dissociates to produce H^+ in two steps. In the first step, H_2CO_3 dissociates to form $H^+ + HCO_3^-$, lowering the pH of the water. In the second step, as more CO_2 is dissolved and the pH decreases, the $HCO_3^=$ ionizes to $H^+ + CO_3^=$, which further lowers the pH.

Concrete sewer lines are susceptible to acid attack although the sewage itself is usually neutral or only slightly acidic. The deterioration of concrete in sewer lines is observed above the water line, rather than below where the sewage is in direct contact with the concrete. Problems of acid attack above the water level in sewers arise indirectly from the bacterial decomposition of sewage, which produces hydrogen sulfide gas. The gas rises and combines with oxygen and with moisture condensed on upper surfaces of the sewer conduit to form sulfuric acid where it reacts with the cement paste [181]. Acid corrosion problems in the crown portions may be reduced by design or operating modifications that results in the sewers running full or with ventilation at higher velocities. Lower temperatures also tend to reduce the production of acid-forming hydrogen sulfide gas. When sewage is stagnant or moves slowly, bacterial creation of the sulfide may be too rapid for the sulfide to be oxidized by air dissolved in the sewage. But if industrial wastes are discharged into sewage lines, the situation may significantly change and present a special problem.

Engineers are often faced with questions concerning whether concrete will be resistant to acidic solutions of varying concentrations. As acid solutions are commonly described in terms of pH, most engineers are interested in rules of thumb regarding the resistance of concrete to acids at various pH levels. Although pH by itself is not necessarily a good indicator of aggressiveness, most investigators seem to feel above pH 5.5 the prospective severity of acid attack is zero to very slight, from about pH 4 to 5.5 acid attack may range from slight to somewhat aggressive if the acid is replenished and some precautionary measures may be advisable (such as chemically resistant slag cements or supersulfated cement), and below pH 3.5 acid attack may be severe and the concrete should be protected [4,6,10,77,182–184]. For neutral to moderately acidic natural waters, the American Water Works Association recommends using an Aggressiveness Index, which is a function of pH, calcium hardness, and total alkalinity [185].

Testing for acid resistance usually consists of soaking test specimens in acid solutions followed by periodic testing. Most tests are run under static conditions, and may show some inconsistencies as the acid is consumed during the test. ASTM

Test Method for Chemical Resistance of Mortars (C 267) consists of immersing test specimens in the particular solution under investigation over a period of time. Specimens are removed periodically and tested for visual appearance, weight loss, and loss of compressive strength. Some feel such tests should be conducted on a depth of scaling basis rather than weight loss.

Surface treatments or protective coatings are sometimes prescribed for concrete exposed to various acids, but usually there is no guarantee as to their long-term effectiveness [4,5,176]. If the exposure is severe, it is better to use an acid-resistant cement than to rely on a protective coating system.

Seawater and Brines

The fact that plain concrete (nonreinforced) of good quality can have good resistance to seawater is amply demonstrated by the many examples of structures showing good service life after many years exposure to seawater. Examples of good performance of concrete made using seawater as mix water can also be found. But there are also many examples of concrete deterioration in exposure to seawater. Most of these examples involve the deterioration of poor quality, permeable concrete or the corrosion of reinforcing steel. The permeability of concrete is one of the key factors in seawater attack [81].

There are several mechanisms involved in the deterioration: leaching and chemical attack of the cement paste by seawater, corrosion of steel reinforcement, wetting and drying effects, freezing and thawing, and wave action [8,9,36,43,81,186–190]. Biological attack has also been identified [191].

The type of attack on concrete depends on the exposure conditions of the concrete to seawater. Generally speaking, concrete exposed above the high tide line is subject to attack from corrosion of reinforcement and freezing and thawing; concrete in the tidal zone or splash zone is susceptible to cracking and spalling from wetting and drying, frost action, corrosion of reinforcement, and erosion of material from chemical reactions by wave action; and below the water line by chemical reactions between seawater and cement paste that soften the concrete and make it susceptible to erosion [81,192]. The mechanisms for deterioration are complicated as the reactions involved in seawater attack may vary according to the exposure and temperature conditions and according to the depth of penetration of the various seawater constituents into the concrete.

Seawater contains about 3.5 % dissolved salts, mainly magnesium sulfate and sodium chloride. The most commonly described reaction of seawater with cement paste is sulfate attack although other reactions involving chloride and magnesium ions and carbonation also occur (see discussion of $MgSO_4$ reactions under the section on sulfate attack) [10,36]. The reaction is somewhat different than ordinary sulfate attack in that the reactions seem to be more complex, the concrete generally deteriorates from erosion and loss of concrete constituents rather than by expansion and cracking, and the chemical composition of the cement seems to play only a minor role if at all [36,64,87]. The observed deterioration does not seem to be related to the presence of ettringite per se. Two types of ettringite have been observed: one type is associated with Type I and II cements and is considered expansive[4] and the other type is associated with Type V cement and does not appear to be expansive [41].

Most investigators observe that seawater is not as destructive to concrete as the total sulfate content of the water would indicate, and suggest this is due to the effects of dissolved chloride on the reactions. The role of chloride appears to be quite complex. Chloride ions penetrate cement paste more readily than sulfate and other ions, with the result that the course of chemical attack may vary with depth within the concrete according to rate the reactive species penetrate into the concrete. Chloride and dissolved CO_2 in the seawater also act to dissolve $Ca(OH)_2$ in the cement paste. Leaching and subsequent erosion of concrete is more severe in seawater as calcium hydroxide and gypsum are more soluble in seawater than in ordinary water.

Chloride reactions appear to have three main effects on the cement paste: lowering of pH and dissolution of $Ca(OH)_2$ and gypsum, reaction with calcium aluminate to form chloroaluminate and suppression of the formation of calcium sulfoaluminate, and enlargement of pores and increase in the permeability of the cement paste [36,43,193,194]. Chlorides tend to reduce the pH of the system, and as the pH is lowered, the solubility of $Ca(OH)_2$ is increased. The leaching of $Ca(OH)_2$ is only very slight at pH 13, but increases as pH is lowered [193]. Ettringite is also more soluble in solutions containing chloride [194]. The presence of chloride favors the formation of chloroaluminate over ettringite; however, in the presence of sufficient sulfate the chloroaluminate will convert to ettringite.

The main line of defense against seawater attack is making good quality impervious concrete. This generally means using a high cement content, low water/cement ratio, good consolidation, and workmanship. The use of pozzolans such as fly ash and silica fume, or slag cements can result in substantial reductions to the permeability of concrete and hence increase the resistance of the concrete to chlorides [10,36,95,195–198]. In very severe cases, special cements such as high alumina cements and polymer concretes work well [199].

The question of the durability of concrete in exposure to brines and seawater, especially at high temperatures, arises in industrial situations, hazardous waste containment, and in saline water conversion projects involving the use of concrete structures.

The durability of concrete exposed to distilled water and synthetic seawater brine at normal and elevated temperatures has been investigated fairly extensively by the U.S. Bureau of Reclamation in a study on the use of portland-cement concrete in saline water distillation plants [22,200]. These investigations showed that conventional portland-cement concrete is not affected by exposure to seawater for four years at temperatures up to 200°F (93°C), and showed only slight surface deterioration at exposures up to 250°F (121°C). Conventional portland cement concrete is considered suitable for use in exposures ranging up to 200°F (93°C), and up to 250°F (121°C) if sacrificial concrete is provided. Concrete exposed to seawater at 290°F (143°C) for 40 months showed surface deterioration to a depth of about 15 mm and was not considered suitable for long-term use at this temperature. Hot seawater exposure studies performed on concrete made with a crushed limestone coarse aggregate showed that the limestone is susceptible to

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[4] This does not mean all ettringite in concrete made with Types I and II cements has caused expansion.

dissolution when directly exposed to hot brines, such as on sawed surfaces, and that the concrete undergoes abnormally large expansions. The use of limestone aggregates in such exposures is not recommended. However, concrete made with natural siliceous aggregates did not show signs of deleterious sulfate attack.

Carbonation

The reaction of carbon dioxide gas with hydrated portland cement, called "carbonation," is known to produce both beneficial and detrimental effects in concrete (the reactions of carbon dioxide dissolved in water is discussed in the sections on Scaling, Efflorescence, and Leaching and Acid Attack). Carbon dioxide gas reacts with calcium hydroxide in the hydrated cement paste to form calcium carbonate [36,64, 201–205]. All calcium silicate hydration products are susceptible to carbonation, and over long periods of time will be ultimately converted to calcium carbonate and hydrated silica [65,203,204,206]. The basic reactions are

$$\underset{\substack{\text{carbon}\\\text{dioxide}}}{CO_2} \quad + \quad \underset{\text{water}}{H_2O} \quad \rightarrow \quad \underset{\substack{\text{Carbonic}\\\text{acid}}}{H_2CO_3}$$

$$\underset{\substack{\text{Carbonic}\\\text{acid}}}{H_2CO_3} \quad + \quad \underset{\substack{\text{calcium}\\\text{hydroxide}}}{Ca(OH)_2} \quad \rightarrow \quad \underset{\substack{\text{calcium}\\\text{carbonate}}}{CaCO_3} \quad + \quad \underset{\text{water}}{2H_2O}$$

The long-term exposure of concrete to the atmosphere often results in shrinkage of the concrete and sometimes cracking if the shrinkage is great enough. The shrinkage is generally attributed to two main causes: (1) a gradual loss of moisture (drying shrinkage), and (2) carbonation. In situations where the long-term volume stability of concrete is important, carbonation becomes of interest not only in studies to separate the two causes for shrinkage, but also as a means to control shrinkage. Intentional carbonation of freshly placed concrete is an effective means to control shrinkage and also to improve strength, hardness, and imperviousness of concrete [207].

Moisture is an important factor in carbonation. Studies have indicated that carbonation does not occur in dry cement or cement at 100 % relative humidity (RH) [204]. Apparently, at 100 % RH, the moisture blocks carbon dioxide from passing through the pores. The optimum conditions for carbonation appear to be around 50 % RH.

Carbonation is of concern in Europe in regard to the deterioration of concrete balconies, facades, parapets, etc., although these problems do not appear to be quite so wide spread in North America [208]. The deterioration is caused by the corrosion of reinforcing steel. Carbonation reaction consumes calcium hydroxide, destroys the passivation effect of calcium hydroxide in preventing the corrosion of reinforcing steel, and lowers the pH to a point where corrosion readily occurs. Carbonation also causes shrinkage of the cement paste that may produce cracking or surface crazing of concrete [205,209–211]. Carbonation often acts in concert with other destructive mechanisms, such as chemical attack or freeze-thaw action [212].

Both fresh and hardened concrete are susceptible to carbonation, and processes have been developed to use carbonation as a treatment to produce beneficial effects. The treatments are reported to harden surfaces, increase compressive strength, increase imperviousness, and reduce shrinkage [202,207,213]. The process has been used as a means to prevent crazing, decrease permeability and improve frost resistance, control dusting and increase wear resistance, of the surface, and to control aggressive chemical reactions including further carbonation and alkali-aggregate reaction. However, carbonation of low-strength concrete with a high water-to-cementing-materials ratio (W/CM) can result in considerable weakening of the cement matrix [214].

The partial replacement of portland cement with higher than normal levels of pozzolans (such as fly ash) or slag, while improving many properties of concrete, may result in an increased rate of carbonation in concrete, especially in poorly cured, low-strength concrete [215,216]. Consideration should be given to extending the period of moist curing or reducing the water-to-cementing-materials ratio (W/CM), or both, when using high levels of pozzolans or slag.

Attack by Other Chemicals

Concrete has good resistance to attack by dry chemicals, dry soils containing chemicals, most alkalies, mineral oils, neutral salts and many salts, of strong bases, although it is susceptible to varying degrees of attack by other chemicals not already covered in this chapter. These include acid-producing substances, ammonia salts, magnesium salts, concentrated solutions of strong bases such as NaOH, chlorides, sulfates, nitrates, salts of weak bases, animal wastes, vegetable and animal oils, fats, sugar, glycol, glycerol, phenol, and creosote. The literature contains fairly comprehensive lists of various substances and their effects on concrete [4,5,10,36,77,176,217–219].

Such generalities on the resistance or susceptibility of concrete to chemical attack should be taken with some caution as the length of exposure, temperature, exposure conditions, concentration, and reactivity of the particular chemical must also be considered. Some chemicals ordinarily considered as not particularly aggressive towards concrete may react very slowly under prolonged exposure conditions or at elevated temperatures, and progressively attack concrete over a period of many years, and ultimately may prove to be aggressive towards concrete. Some substances, which by themselves may be harmless, may react with other substances in the environment and become aggressive. Examples of these materials are vegetation that may decompose to form organic acids and sulfides that may oxidize to form sulfuric acid. The severity and rate of attack may vary greatly and an actual test under anticipated exposure conditions is recommended to assess the potential for deterioration.

Vegetable and animal oils usually contain fatty acids that may be oxidized to form more acid during the exposure and become even more aggressive towards concrete. Many oils, fats, and glycerides are saponified by the lime in hydrated cement to form alcohols that can react with more lime to further deteriorate the concrete. On the other hand, drying oils (linseed oil and tung oil), undergo oxidization and polymerize when exposed to air. These oils form hardened films and have been used as protective coatings on concrete.

Many salts are not particularly harmful to plain portland cement concrete under ordinary circumstances, but in some particular situations may cause damage in other ways, such as by accelerating the corrosion of reinforcing steel or by "salt attack" (corrosion is discussed in the chapter on Embedded Metals and Materials Other Than Reinforcing Steel). Salt attack includes both "salt scaling" and "salt weathering." These forms of salt attack are caused by destructive physical processes rather than by a strictly chemical type of attack, and therefore will only be mentioned briefly.

Salt scaling refers to spalling, cracking, and deterioration of concrete surfaces associated with the use of deicing salts, usually sodium or calcium chloride, on concrete in freezing weather [220–227]. The attack is quite rapid and destructive to concrete surfaces. The deterioration appears to be primarily caused by frost action (see the chapter on Freezing and Thawing), but at times other phenomena may also be involved, such as corrosion of reinforcing steel, leaching, carbonation, osmotic pressures, and salt heaving. Non-air-entrained concrete is quite susceptible to salt scaling.

Remedial measures are similar to those recommended for freeze-thaw resistance: use a high quality, dense, air-entrained concrete with a water-cement ratio of 0.45 or less. Some sealers and surface treatments have also shown promise in reducing salt scaling [222,228]. Pozzolans, including silica fume and fly ash, may also be effective. Particular combinations of materials should be tested to determine their effectiveness, such as by ASTM Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals (C 672) [226,229,230].

Salt weathering is a destructive physical process commonly occurring in arid climates, coastal regions, and in wet-dry exposures. Deterioration of concrete by salt weathering has been relatively limited and has been reported only infrequently although the process is fairly widespread [58–60,113,231,232]. The subject has been explored more thoroughly as a geologic weathering process, and also in regards to historic preservation and the weathering of historic and cultural artifacts [233–236]. Most investigators attribute the mechanism of the attack to expansive pressures generated by the formation of salt crystals in fractures and pores [39,146,237]; others feel other mechanisms may be involved, such as osmotic, surface tension, and adsorption effects that cause swelling and shrinkage [238]. The crystal growth mechanism for salt attack has been fairly well established [40,237,239–242].

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Resistance to Fire and High Temperatures

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Preface

THE FUNDAMENTALS OF CONCRETE PERFORMANCE at high temperatures, including fire resistance, are well established. This subject was authored in *ASTM STP 169* and *169A* by P. H. Peterson under the title "Resistance to Fire and Radiation." Peter Smith authored the chapter in *STP 169B* entitled "Resistance to High Temperature" and the one in *STP 169C* with the present title. Accordingly, major revisions have not been necessary, though new products, applications, and research results are addressed, such as those arising from the use of high-strength concrete.

Introduction

One of the main reasons why portland cement concrete is so widely used in building construction is that it can help satisfy the cardinal need for public safety in the face of the hazards of fire better than most alternative materials. Concrete is noncombustible and a reasonable insulator against the transmission of heat. These qualities alone are considered in design for fire containment and limiting the extent of the damage. Though portions of the surface of concrete elements may crumble or spall, more often than not the essential engineering properties of the body of the concrete remain intact. In many applications, the main role of concrete in a fire is to protect any embedded steel for as long as possible against a rise in temperature to the point where its physical properties are reduced significantly, causing excessive structural deflections that might lead ultimately to collapse.

Building codes contain minimum fire protection requirements based on a combination of knowledge of the physical properties and past experience of the behavior of various building materials when exposed to fire, and upon fire endurance ratings specified by the survival times of specific structural assemblies or components in standard laboratory fire tests. Usually only the ambient temperature regime to a maximum is controlled in these tests. Therefore, such tests do not provide much information about the effect of specific high temperatures on the properties of concrete, its constituent materials, or reinforcing steel except in a very general way. To improve fire resistance in building design, or to assess the condition and possibilities of repair of a structure damaged by fire, more needs to be known about the thermal and mechanical properties of steel and concrete at elevated temperatures and residual properties after slow or quench cooling. It is relatively easy to determine the residual properties by standard test methods and the results provide much of the information needed to determine what can be saved after a fire. However, experiments to determine the same properties at sustained or cyclical high temperature require special equipment and measuring techniques. This kind of information has become increasingly in demand for several reasons. First, it is needed for the development and validation of computer-based modeling techniques of heat flow and structural behavior. Computer modeling offers promise for extending the results of fire tests on discrete assemblies to the more general cases, and demonstrating the beneficial effects of restraint and support from unaffected cooler parts of the structure. Second, advanced industrial applications, in particular for nuclear reactors, require a greater knowledge of the physical properties of various types of concrete when subject to complex, sustained or repetitive, mechanical and thermal stress regimes at moderately high temperatures. Third, as new concrete constituents and proportions continue to become available, such has concrete with specified compressive strengths in excess of 83 MPa [12 000 psi], information is needed to determine if and what modifications should be made to methods for determining fire endurance. While some industrial and military applications require special concrete that is resistant to specific service temperature regimes; fortunately for most constructions, three of the most important factors are within the control of the designer and specifier prior to placing concrete. These are: the structural system and design details chosen, the thickness of cover provided to any embedded steel, and the design of concrete mixtures. By selection of the appropriate combination of these for a particular application, the desired resistance to high temperatures, including fire, can be achieved.

Fire Testing, Endurance Standards, and Modeling

In North America, the fire resistance of structural and fire protection components of buildings are usually based on the fire endurance as determined by ASTM Method for Fire Tests of Building Construction and Materials (E 119). A standard fire exposure is defined by a time-temperature curve in which 538°C (1000°F) is attained in 5 min, 927°C (1700°F) in 1 h, and 1260°C (2300°F) in 8 h (assuming an end point has not yet been reached). Exposure to the furnace temperature is on one side

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only for slabs and walls, beams are exposed from beneath and from the sides, and columns are exposed on all vertical surfaces. Minimum specimen sizes (areas or lengths exposed) and a moisture condition requirement prior to testing are specified and superimposed loads and restraint may be added to simulate in-service conditions.

End point criteria in the test are intended to simulate modes of failure in an actual fire. They include the following:

- Transmission of heat.
- Transmission of hot gases sufficient to ignite cotton waste.
- For load bearing elements, load carrying ability during the test exposure.
- For walls, resistance to the passage of a hose stream.

For load bearing elements, the maximum load condition is defined as that allowed under nationally recognized structural design criteria. These design criteria consider factored loads increasing the load to account for potential variations over the life of the building. Load factors are assigned for specific combinations of loads. In general, some degree of conservatism is provided because the maximum load experienced is less than the maximum load conditions during the fire tests. ASTM E 119 requires load combinations that result in the maximum load, in accordance with nationally recognized structural design criteria, be used to determine the applied load on the test specimen.

For walls, the assembly must also resist the passage of a stream of water when subjected to a hose stream test in accordance with ASTM Practice for Application of Hose Stream (E 2226). Fire endurance is then stated in terms of time to reach whichever end point occurs first (such as 1, 2, 3, or 4-h rating). The appropriate fire endurance requirements, necessary to ensure safety in a particular set of circumstances, are prescribed in the applicable building code along with related design criteria. It must be emphasized that the prime purposes of fire tests and related criteria are to ensure life safety of the building occupants and to provide property protection in case of fire and not to ensure structural survival or ease of repair after the fire (though often the more fire resistant the construction is, the more likely that it will be salvageable). The requirements and significance of standard fire test procedures and the influence of structural design and material parameters on fire endurance have been debated for over 100 years, with improvements being introduced as the development of knowledge and experience dictated. Uddin and Culver [1] cite 149 historic references covering the period from 1884 to 1961, when an American Concrete Institute (ACI) Symposium was held. The proceedings of that symposium provide a good opening to present-day thinking on fire resistance through four comprehensive papers. Carlson [2] looked at the effect of moisture, specimen size, restraint, surface temperature criteria, and radiant heat. Benjamin [3] reported on thermal factors arising from moisture content and type of aggregate, on structural factors such as concrete strength, cover to reinforcing steel, spalling, and lateral restraint as affecting fire endurance ratings. Sheridan [4] discussed concrete as a protection for structural steel. Troxell [5] addressed the special problems of prestressed concrete sections. Since then, researchers and fire testing laboratories around the world have worked actively to evaluate new types of construction and improve fundamental knowledge.

The establishment of fire endurance ratings by means of standard fire tests is both expensive and time consuming. While the results are specific and particular to the component or assembly tested, the results are generally extended to other concrete elements having similar configurations, constituents, and thermal properties. It is difficult and much more expensive to simulate the effects of restraint by cooler parts of a concrete structure that are clear of the actual fire [6]. Alternative mathematical modeling methods to more precisely determine fire endurance without the expense of full scale testing continue to be developed. The mathematical models tend to predict the heat flow process in concrete elements to establish temperature distributions and, taking material and structural parameters into account, to then predict fire endurance and structural behavior at high temperatures.

The almost limitless number of constituents and their proportions, configurations, and details for concrete elements demanded the development of analytical methods to determine the fire resistance rating of concrete elements for compliance with building code provisions. Methods such as the American Concrete Institute Standard Method for Determining Fire Resistance of Concrete and Masonry Construction Assemblies (ACI 216.1) [7] and American Society of Civil Engineers Standard Calculation Methods for Structural Fire Protection (ASCE 29) [8] are based on ASTM E 119 test data. Further guidance on determining the fire endurance of concrete elements is provided in a report of ACI 216 [9] and is beyond the scope of this chapter to further discuss fire-resistant structural design.

Factors Influencing Behavior and Affecting Test Methods

For most applications, interest in the behavior of structural concrete exposed to high-temperatures begins at a lower bound temperature of 100°C (212°F) and immediately above as free water starts to be driven off. Generally speaking, the engineering properties and behavior of concrete at these temperatures vary by only a few percentage points from those measured at room temperatures. However, above about 149°C (300°F), the progressive continuum of cement dehydration reactions, thermal incompatibilities between paste and aggregate, and physical-chemical deterioration of some aggregates leads to high thermal stresses, microcracking, and a rapid worsening in most mechanical properties of structural value. However, the end of structural usefulness of a particular concrete does not occur suddenly at a specific temperature.

Previous chapters focused on the review by Uddin and Culver [1], which list six interrelated material properties and conditions as having particular influence on the high-temperature performance of structural concrete: (1) type of aggregate, (2) free moisture in the concrete, (3) stress levels in both steel and concrete, (4) cover over reinforcing steel, (5) modulus of elasticity, and (6) thermal conductivity, diffusivity, specific heat, etc. Three other factors may significantly influence the fire endurance of concrete elements. Kodur et al. [10] found that (7) the amount, configuration, and placement of reinforcing steel ties has a significant influence in high-strength concrete, when the specified compressive strength is greater than 83 MPa (12 000 psi). Tests reviewed by Benjamin [3] suggest that (8) the volume and relationship of volume to surface area of the concrete elements also has a significant influence as temperature drop from the exposed surface is steep. Lin et al. [11,12] found that increasing the cross section size of columns, even in one direction significantly increases fire resistance. Kodur et al. [10] also found that (9) the use of silica fume in the concrete mix influences the performance of concrete having a specified compressive strength greater than 83

MPa (12 000 psi). The use of silica fume appears to reduce the permeability of the concrete restricting the loss of moisture during curing, drying, and the fire test. This latter concept has not been thoroughly researched or evaluated. While silica fume and other changes in the basic constituent of structural concrete may influence the fire endurance, factors such as different types of portland cements, are of much less importance.

Most of the factors interrelate and the format has been adopted of first considering the influences of component materials, then the mechanical and thermal properties of ordinary concretes and, finally, concretes in special applications. Within the space available, it is not possible to reproduce the many graphical displays of test results in the references discussed. All the essential ones covering steel properties, mechanical and thermal properties of concrete, and temperature distribution across various sections can be found in the references.

Throughout the discussion, experimental techniques to measure various properties will be identified or, where this is not essential, left to the details provided by the referenced sources. Two references, Bertero et al. [13] and Purkiss and Dougill [14], are specific to experimental techniques and support a general observation that a property easily be determined at normal temperatures by standard tests often can only be determined at elevated temperatures with difficulty. Modification of existing standard methods or the development of new methods is often required to determine some relevant properties at elevated temperatures. As a result, many of the differences reported in measured values of various properties may be ascribed to differences in test conditions. However, while the actual numbers may differ, there is a usually good agreement on the general trends reported.

Two tests have proved useful in obtaining an understanding of the observed behavior of concrete at high temperatures. Thermogravimetry tests provide insight into chemical reactions involving loss of weight while dilatometry tests that measure change of length may detect other reactions not necessarily accompanied by loss of weight. Harmathy and Allen [15], among other investigators, used these to examine the overall stability of a variety of concretes and to pin-point specific chemical and physical changes that occur with increasing temperature. Cement dehydration or the decomposition of carbonate aggregate and the chemical stability of lightweight or instability of siliceous aggregate concretes are very evident in their loss-of-weight test data. One important result from the change-of-length test is to show that the α-β quartz transformation at 573°C (1063°F), while certainly causing an increase in volume in siliceous aggregates, did not cause a corresponding change in the concrete. This, Harmathy and Allen [15] attributed to the accommodating plasticity of the cement paste at high temperatures. The overall pattern of their length change data confirms the complex superposition of dehydration shrinkage in the cement paste and expansion of the aggregate as being a major factor in high-temperature performance of concrete. The corresponding cooling curves demonstrate that most of the reactions and events that occur during heating are irreversible.

Many of the tests discussed in this chapter, including but not limited to Purkiss and Dougill [14], Hertz [16], Malhotra [17], Zoldners [18], and Philleo [19] involved small specimens or coupons of concrete and while the tests identify the phenomena occurring at specific temperature exposures, the exposure and specimens are not representative of actual concrete elements used in structures. Frequently when small specimens or coupons are used the maximum aggregate size is also limited. Such limitations may not be representative of typical concrete used in structural members as the surface area of the aggregate in contact with cement paste is larger and the smaller aggregate typically results with an increase in the amount of paste for a given volume. Both may exaggerate the effects of the cement paste. Even "full-scale" tests, such as Kodur et al. [10], are not representative of actual concrete elements. In the latter, the largest cross-sectional area of the high strength concrete columns tested was less than one-half the cross-sectional area of high strength concrete columns currently used in typical building design practice today.

Influence of Cement Paste Component

Choosing one portland cement over another will do little to improve the properties of concrete at high temperature. High alumina cements are used in refractory concretes but may not be appropriate for structural purposes because of the conversion phenomena that may occur under normal service conditions.

While a case may be made for using fly ash or blast furnace slag cements mainly because these produce lesser amounts of calcium oxide as a result of dehydration in the heat of a fire, in practice these are little used for such benefit alone. At lower temperatures such as those that might be experienced in mass concrete in nuclear reactors, Nasser and Marzouk [20] attributed an increase in compressive strength and decrease in the modulus of elasticity in the temperature range of 121–149°C (250–300°F) to the formation of tobermorite that is much stronger than tobermorite gel. After six months in a temperature range of 177–232°C (350–450°F), there was a great reduction in structural properties of fly ash concrete, thought to be due to the formation of crystalline alpha dicalcium silicates that are poor binders. Fly ash as it affects lightweight concrete is discussed under that section.

Silica fume additions have been shown to increase the risk of explosive spalling in very high strength concrete [16]. In the same investigation, it was noted that when a melamine-based high range water reducer was incorporated, it decomposed releasing ammonia gas. Otherwise, admixtures appear to have no unusual effects.

The development of distress and the change for the worse in the thermal and mechanical properties of portland cement pastes that occur with increasing temperature are the result of an uninterrupted series of physical-chemical reactions that are accompanied by shrinkage and microcracking. Desorption of what is commonly called evaporable water first takes place as the temperature increases. Then, and overlapping, chemically bound water (nonevaporable) is released progressively from the complex system of low crystalline order, calcium silicate hydrates, and other hydrates in the cement paste and from the calcium hydroxide crystals formed when the cement originally hydrated.

The dehydration of calcium hydroxide itself to form the oxide is a relatively simple process occurring above 400°C (752°F). Subsequently, the calcium oxide, after cooling and in the presence of moisture, will rehydrate to calcium hydroxide with a disruptive 14 % increase in volume. On the other hand, the dehydration of the silicate hydrates and other compounds present, through any intermediate stages, is more complicated and gradual and not fully understood. Dehydration is complete at 800°C (1472°F) and above. The stage reached at any given temperature is dependent also upon the rate of heating since all the reactions are not instantaneous. Most of these decompositions are irreversible so damage to concrete from this cause at sustained high temperatures is essentially permanent, and the practical end point is probably about 538°C (1000°F). After exposure to sustained temperatures of 649–816°C (1200–1500°F), concrete is friable, porous, and, after cooling, usually can be taken apart with the fingers. Many of these events are evident in thermogravimetric, dilatometric, and differential thermal analysis [15,21].

Zoldners' review of experimental thermal data [22] shows that the variation of thermal properties of the cement paste with temperature, in particular thermal volume change, is a very important component to the overall behavior of concrete because of incompatibility with the same properties in the aggregates. There appear to be two components to thermal volume change of paste prior to decomposition: (1) a true thermal expansion that is essentially constant and reversible, and (2) an apparent thermal expansion that is a hygrothermal contraction dependent on the internal transportation of moisture between various states in the capillaries and gel pores, resulting in shrinkage that is irreversible. Thermal expansion is additive to shrinkage due to dehydration. Many investigators have confirmed that cement paste expands up to 100°C (212°F), and then with further heating and loss of moisture, it contracts rapidly to more than cancel the initial expansion. Above 500°C (932°F) shrinkage again changes to expansion. Actual values of the apparent coefficient of contraction or expansion are time dependent as is dehydration, one of the causative mechanisms. Thermal conductivity of cement paste is also dehydration dependent: the lower the moisture content the lower the thermal conductivity. At 750°C (1382°F), where the paste is much drier and more porous, the thermal conductivity is only half the value measured at room temperature.

In addition to the effects of thermal incompatibilities, the decrease in evaporable and chemically bound water with progressive dehydration of the paste modifies the physical bonds existing initially in the concrete and promotes micro-cracking. The resulting influence in the mechanical properties of concrete with increasing temperature is clear, though separate data related to the paste component alone are sparse.

Influence of Aggregate Component

Unlike the situation with cements, the selection of one aggregate over another can have a great influence on the resistance of concrete to high temperature. Though not entirely justified by data, many building codes fire resistance calculation procedures group concretes into two to four classes of aggregate type as a basis for determining fire resistance ratings of concrete. Crushed firebrick and fused aluminum oxide (corundum) and other special aggregates used in refractory concretes perform best. Then in descending order of fire endurance come expanded slag, shale, slate, and clay; air-cooled slag; basic, finely grained igneous rock such as basalt; calcareous aggregate; and siliceous aggregate. Attention naturally centers on the reasons for the significantly different behavior of calcareous and siliceous aggregates that are the most widely used.

Carbonate aggregates are decomposed chemically by heat whereas siliceous aggregates generally are not. Between 660 and 979°C (1220 and 1795°F), calcium carbonate breaks down into calcium oxide with the release of carbon dioxide. Magnesium carbonate is likewise decomposed between 741 and 838°C (1365 and 1540°F). Both reactions are endothermic, thus absorbing heat and delaying temperature rise in the concrete. The calcined material is also less dense and hence a better insulator. Furthermore, the carbon dioxide, escaping from the surface of the concrete in considerable volume, forms an inert insulation layer thus further retarding temperature rise in the concrete. The work by Kodur et al. [10] supports the findings that carbonate aggregate increases the fire endurance of concrete columns compared to that of siliceous aggregate. Lin et al. [11,12] found that 305-mm by 305-mm (12-in. by 12-in.) columns with 38-mm (1 1/2-in.) cover made with 34–53 MPa (4950–7680 psi) concrete will have at least 3 h resistance. This research further identified that increasing the cross section size even in one direction and use of carbonate rather than siliceous aggregate significantly increases fire resistance.

Though siliceous aggregates may be chemically stable, they have deleterious properties at high temperatures. Most striking is the transformation of the quartz crystal from the α to β polymorph at 573°C (1063°F) with an increase in volume. At higher temperatures, other internal volume changes occur and the crystal form may be meta-stable. Furthermore, quartz has a much greater coefficient of thermal expansion up to 600°C (1112°F) than most other rock. The consequence in concrete is greater thermal incompatibility between the cement paste and aggregate and hence greater internal thermal stresses. Many researchers have ascribed a greater tendency to spalling for these reasons, in particular, the crystal form transformation, though, as later discussed, other mechanisms may dominate.

The desirable thermal properties of aggregate are low thermal conductivity that delays temperature rise, a thermal expansion as close to that of the cement paste as possible in order to minimize development of parasitic thermal stresses, and a high specific heat to absorb heat. Zoldners [22] reviewed studies at CANMET that give exhaustive data on rock thermal expansions up to 1000°C (1832°F) and confirm the dominance of mineral composition. For most rock, the rate of thermal elongation is many times greater above 500°C (932°F) than at room temperatures and abrupt increases or decreases may occur at specific temperatures, as notably happens with siliceous rocks. Mirkovich [23] found the greatest decrease in thermal conductivity occurs with those rock types that show the greatest thermal expansions, that is, quartz, sandstone, and granite, while anorthosite, basalt, and limestone show relatively little change. This connection between thermal conductivity and expansion is ascribed by Zoldners [22] to the fact that higher thermal expansion stresses cause more micro-cracking of crystals and loosening of grains resulting in increased rock porosity and hence decreased thermal conductivity. The "ideal" aggregate from a thermal conductivity point of view seems to require an amorphous microstructure and porous macrostructure, a specification best met by many lightweight aggregates.

Influence of Embedded Steel and Structural Systems

A prime objective in the design of fire-resistant structural concrete are to maintain structural integrity. Structural integrity requirements are primarily for life safety, but may also be required to provide property protection and for many buildings, to prevent collapse in accordance with the provisions of the building code and the specific requirements for the project. This requires keeping any embedded steel as cool as possible to avoid loss of tensile yield strength and to minimize deleterious amounts of differential thermal expansion between the concrete and steel.

Typical reinforcing steel used in reinforcing bars shows a linear decrease in yield strength with increase in temperature up to about 370°C (700°F) totaling about 15 %. After that, the decrease is more rapid and reaches 50 % by about 593°C (1100°F). Less than 20 % of the original yield strength remains at 760°C (1400°F). Prestressing wire and strand reach the 50 % loss of yield strength at a lower temperature, about 423°C (800°F), and some stress relaxation losses may remain after cooling. These temperatures, 593°C (1100°F) and 423°C (800°F), are taken as one of the end points in standard fire tests. The length of time before the steel reaches a critical temperature is when the moment capacity is reduced to the applied moment. In a fire this depends on the amount of cover to the steel and on the insulation properties of the concrete. The influence of cover is striking. For the same normalweight concrete exposed in a standard fire test for 1 h, steel with only 25-mm (1-in.) cover would reach over 427°C (800°F) whereas, with 50-mm (2-in.) cover, the steel temperature would be about 204°C (400°F). With 75-mm (3-in.) cover, the steel would probably be hardly warm to the touch. Similar orders of limitation on steel temperature with increased cover are applicable to other periods of fire exposure [9]. Steel that has remained encased in concrete will recover much of its lost strength on cooling and only exposed reinforcement that has become distorted and twisted will usually need replacing or supplementing. Fire protection requirements relating to structural steel encased in concrete are covered in Sheridan [4] and Harmathy and Allen [15]. Benjamin [3] observed the effect of both specimen area and cover noting that the temperature drop from the exposed surface is steep.

Both the coefficient of thermal expansion and the thermal conductivity of steel influence the performance of the surrounding concrete when exposed to high temperatures. The coefficient of thermal expansion of steel varies with temperature in a dissimilar manner to that of concrete at very high temperatures, but is generally close to that of concrete where compatibility is critical at lower temperatures. Changes in the coefficient of thermal expansion of steel at high temperatures are related to changes in the phase and crystal composition of the steel. The coefficient increases with temperature up to 649°C (1200°F), decreases to zero in the range up to 816°C (1500°F), and then increases again. The thermal conductivity of steel is much greater than that of concrete and sometimes after a fire a thin, whitish, porous layer may be evident surrounding reinforcing steel embedded in otherwise sound concrete. This tends to happen if part of the bar has become exposed to the heat source (as by loss of cover), and the steel temperature then rapidly becomes high enough to dehydrate the cement paste. This effect and the difference in coefficients of thermal expansion may adversely affect the local bond of concrete to steel at higher temperatures. Up to about 316°C (600°F), the bond strength between steel and concrete is the same as, or slightly higher than, at room temperature. Thereafter, it declines, and work by Diederichs and Schneider [24] and others shows that the bond strength at elevated temperatures may be an important structural consideration

The use of epoxy-coated reinforcing steel as a defense against corrosion raised the question of the effect of the organic coating on fire resistance, in particular on bond strength. Research by Sozen and Moehle [25] showed there are differences in the performance of bars with epoxy coatings as compared to black bars with regard to development and lap-splice lengths, but the work of Lin et al. [26] and Lin and Burg [27] indicate that the epoxy coating has little detrimental effect on the performance of concrete elements in fire exposures.

It should be emphasized that there is not one critical temperature or condition at which embedded steel (conventional reinforcement, prestressing strands, or structural steel) suddenly fails. The dead and live load intensity, structural indeterminacy, continuity, and member restraint all influence the outcome in terms of the distortion, deflection, and ultimate collapse of a member. Chana and Price [6] observed the ability of concrete elements to continue to support loads after the endpoint of the fire exposure criteria in a full-scale test of a concrete structure. This is further substantiated by the actual performance of concrete structures during and after fires, such as the 1973 fire in the Military Personnel Record Center in St. Louis, MO [28].

Effect of Experimental Conditions on Mechanical Properties

The observed strength of the same concrete when heated to a particular temperature, or after heating and cooling, depends on the circumstances and superimposed conditions. Variables include the following:

- whether the concrete is restrained or not during heating,
- whether free moisture is contained or not during heating,
- whether the concrete is subject to thermal cycling,
- whether it is tested while hot or after cooling, and
- whether it is quenched or allowed to cool slowly.

In addition, each concrete, depending on the behavior of the cement paste and aggregates, will have different strength and moduli values at different temperatures. Lin et al. [11,12] found that full restraint of concrete columns did not significantly decrease the fire resistance.

Compressive and flexural concrete strengths at elevated temperatures have been studied widely, tensile and shear strength less so. Bond strength between concrete and steel and cement paste and aggregates has been discussed in preceding sections. For purposes of a general understanding, only the more recent and significant studies have been referenced. Access to earlier work is provided by Uddin and Culver [1].

Effect on Compressive Strength

Because compressive strength is one of the prime qualities by which concrete is judged, it is probably the most investigated property at high temperature. In simply supported members, differences in the strength of concrete in the compressive zone have relatively minor consequences and do not affect fire endurance. However in the design of complex frames and continuous or restrained members, the compressive strength is important. Various techniques have been used to modify the compressive strength testing procedure used at normal temperatures, that is, ASTM Test Method for Compressive Strength of Cylindrical Concrete Specimens (C 39) to permit testing at high temperatures in universal testing machines. Usually, the specimen is heated by means of an electrical or gas furnace that jackets a concrete cylinder varying in size from 50 mm (2 in.) diameter and 100 mm (4 in.) length to 100 mm (4 in.) by 200 mm (8 in.). To maintain uniform temperature conditions, load is applied from the testing machine platens through cement-asbestos disks above and beneath steel cylinders abutting flat machined faces of the specimens. Where length changes as well as strength are to be measured, then extension rods or optical devices may be used to bring the gage points outside the furnace. Temperatures are measured and controlled by surface or embedded thermocouples.

Using such apparatus and 75 by 150-mm (3 by 6-in.) cylinders, Abrams [29] determined the compressive strength of concrete between 93 and 871°C (200 and 1600°F) for concretes in the strength range of 23–45 MPa (3300–6500 psi) that contain carbonate, siliceous, and light-weight (expanded shale) aggregates. The testing regimes included heating without load and testing while hot, heating at three stress levels and testing while hot, and testing after slowly cooling.

In summary, his results showed the following: (1) carbonate and sanded lightweight aggregate concrete retained more than 75 % of original strength up to 649°C (1200°F) when heated unstressed and tested hot—for siliceous aggregate concrete, the corresponding temperature was 427°C (800°F); (2) when loaded during heating, compressive strengths were 5–25 % higher but were not affected by the applied stress level; (3) residual strengths determined after cooling were a little lower than the corresponding hot compressive strength; and (4) the original strength of the concrete had little effect on the percent reductions in strength observed.

Turning to Malhotra [17], who tested 50 by 100-mm (2 by 4-in.) concrete cylinders made of 10 mm (3/8-in.) siliceous aggregate cylinders with maturities of from 7 to 42 days conditioning at 24°C (75°F) and 55 to 60 % relative humidity, provides the following observations on some additional factors: (1) aggregate/cement ratio had little bearing on changes in compressive strength up to 204°C (400°F); however, above this temperature lean mixes showed a proportionally smaller reduction in compressive strength than occurred with richer mixtures, and (2) water-cement (w/c) ratios in the range of 0.37–0.65 had little influence on the order of reduction of compressive strength of concrete up to 593°C (1100°F). These tests also confirmed those of Abrams [29] with respect to the benefit of the concrete being under compressive stress (in the order of design stress) when heated. Also recorded was a reduction of compressive strength of about 20 % in specimens tested after cooling.

If concrete specimens are quenched, as may happen to concrete that is hosed down with water during a fire, Zoldners [18] showed that for specimens exposure to temperatures of about 500°C (932°F), there is a larger reduction in residual compressive strength than when specimens were slowly cooled. Zoldners attributed this to thermal shock and noted (1) a harder outer shell in quenched cylinders due to rehydration of the cement, and (2) at higher temperatures the difference in strength between quenched and slowly cooled specimens was largely eliminated.

Zoldners's results include the effect of four different aggregate types on compressive strength at elevated temperatures [18]. They were a gravel comprised mainly of a mixture of metamorphic and granitic igneous rock, a relatively pure sandstone, a high-calcium fine-grained limestone, and an expanded slag. His conclusions provide three additional pieces of information in addition to generally agreeing with Abrams [29].

- The crystalline igneous and metamorphic rock gravel aggregate deteriorated more rapidly than did the limestone aggregate concrete; at 400°C (752°F), only 85 % of the initial strength remained as compared with over 95 % for the limestone.
- The sandstone aggregate concrete showed a significant strength gain up to about 300°C (572°F) after which deterioration was rapid.
- Above 525°C (977°F), the expanded slag aggregate concrete out-performed the others whereas below that temperature it was equal or inferior in compressive strength.

Research on the relationship of the reduced maximum aggregate size used in the small cylinder and beam specimens to the performance of concrete with typical aggregate size is lacking. The effects of the aggregate itself and the contact areas with cement paste may be important for accurately determining the performance of concrete elements. Further, as discussed by Zoldners [18], the specimen size is likely to be a significant influence.

Effect on Flexural Strength

The most common procedure for determining flexural strength appears to be a modification of three-point loading of small beams along the lines of ASTM Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78). The decreases observed in flexural strength are not necessarily of the same order and magnitude as those for compressive strength of concrete of the same mix proportions and containing the same constituents [18]. Generally, the decline of flexural strength is much greater than that of compressive strength. For example, Sullivan and Poucher [30] found the flexural strength of a siliceous aggregate concrete at 400°C (752°F) varied from 25 % to 0 % of the original strength. Zoldners [18] obtained similar results, but found only a 50 % flexural strength reduction with limestone aggregate concrete at 400°C (752°F) (a reduction level that he found to occur at over 600°C (1112°F) in the case of compression). Zoldners [18] also found that expanded shale only retained 16 % of its original flexural strength after exposure to 400°C (752°F). Again, quenching resulted in greater strength losses than occurred when specimens were tested at elevated temperatures and slowly cooled.

Effect on Modulus of Elasticity, Poisson's Ratio, and Bulk Modulus

The ratio of stress to strain is an important structural concrete design parameter. At high temperatures, the value of E, the modulus of elasticity, may be obtained dynamically as by Philleo [19], or by displacement measurements as by Sullivan and Poucher [30] on concrete specimens heated within a furnace. In general terms, there is a decrease in E in the order of 10–15 % between initial values at room temperature and those at 100°C (212°F). E then remains relatively constant up to 300°C (572°F) after which a progressive decline sets in so that by 538°C (1000°F) it is only half or less than half of that before heating. Sullivan and Poucher [30] noted in their tests, made up to 400°C (752°F) that for a given mortar or concrete (with siliceous aggregates), the value of E after cooling was essentially the same as that prevailing at the high test temperature, thus determining that the reduction in E with increasing temperature is a permanent one. Philleo [19] noted that the value of E depended on initial w/c ratio, curing regime, and age. Between unheated values for calcareous gravel aggregate concrete ranging from 2.21 to 5.06×10^{-8} MPa (3.20 to 7.34×10^{-6} psi) and final values at 760°C (1400°F) of from 0.62 to 1.59×10^{-8} MPa (0.91 to 2.30×10^{-6} psi), some benefit was found in low w/c ratios and air drying. Marechal [31] looked at the variation of E with temperature for expanded clay, crushed rock aggregate (mainly porphyry and quartzite), and gravel aggregate (siliceous and siliceous limestone) at one cement factor. He found the reduction in this modulus to be aggregate dependent, in the preceding order, up to 400°C (752°F).

Holm and Bremner [32] found that for individual concrete masonry units develop lower thermal expansions resulted in low residual deformations due to the lower rate of thermal expansion, lower modulus of elasticity, and prior exposure to elevated temperatures during manufacture. Bremner et al. [33] found that lightweight concrete masonry unit wall specimens experienced less bowing than normalweight concrete masonry unit wall specimens subjected to temperatures up to 900°C (1652°F).

Philleo [19] was able to compute Poisson's ratio because he measured both flexural and torsional frequencies of vibration. Because of sensitivity complications in the test procedure, the results were erratic, but a general tendency to decline with increasing temperature was apparent. Marechal [31] also calculated Poisson's ratio, but did so directly from the longitudinal axial load and measured transverse strain data and a similar decline was found after the evaporable water had been driven off. He made further calculations for the bulk modulus, *K,* which declined with temperature in a curve paralleling that for *E.*

Effect on Creep

Data on inelastic behavior of concrete at high temperature are essential for nuclear applications, since long-term behavior under complex mechanical thermal loadings is involved, and is important for fire resistance design. If inelastic behavior is not taken into account and if only data secured on small specimens are used, over-estimations of the affected parameters will occur. A first step in overcoming the continuing elusiveness of creep at high temperature was to devise apparatus for measuring it as reported by Cruz [34].

A study by Wang [35] of moderately high-strength concretes 38 and 46 MPa (5500 and 6700 psi) containing quartz gravel aggregate over the temperature range from 93 to 427°C (200 to 800°F) showed that the shape of creep-time curves was the same as at room temperature, but the creep rate was higher when the concrete was subject to both high temperature and a high stress-to-strength ratio. He also found that (1) the latter affects the creep rate more than the temperature; (2) there was a nonlinear relationship between creep and stress level; and (3) with a lower w/c ratio, creep was less. Sullivan and Poucher [30] attributed the fact that creep rates for concrete above 200°C (392°F) were higher than those for mortar, while the reverse was true below 200°C (392°F), to the development of microcracking between aggregate and paste. At 300°C (572°F), they measured creep rates in concrete at three times those at 125°C (257°F), and above 400°C (752°F), they observed that creep increased very rapidly at low stress levels as a plastic stage was reached. Dependence of creep on moisture content is stressed by Marechal [36]. Higher creep rates occur in saturated or sealed specimens for which a fully accepted explanation does not exist.

In addition to creep tests, Cruz [34] also carried out exploratory stress relaxation tests under constant strain over a 5-h period. Stress was reduced by 2, 32, and 74 % at 24, 316, and 649°C (75, 600, and 1200°F), respectively.

Investigations of concrete under transient high-temperature conditions, including time-dependent effects such as creep and relaxation, confirm that a very complex pattern of behavior probably exists [37].

Determining Thermal Properties

The basic thermal properties of concern to the behavior of concrete at elevated temperatures are: thermal conductivity (the ability of the material to conduct heat), a ratio of the flux of the heat to the temperature gradient, thermal diffusivity (the rate at which temperature changes can take place), and the two more widely understood parameters of specific heat and the coefficient of thermal expansion. Zoldners [22] provides experimental values but, as will be discussed, there are difficulties with many of the test procedures at elevated temperatures.

Thermal Conductivity, Diffusivity, and Specific Heat

Thermal conductivity and diffusivity of a mixture such as concrete are not additives of the properties of the constituents and there are difficulties in experimental determination at a given elevated temperature. These are due principally to accompanying moisture migration-desorption, though other physical-chemical changes also contribute. Thompson [38] discusses the shortcomings of steady-state methods such as ASTM Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus (C 177) or ASTM Test Method for Steady-State Thermal Performance of Building Assemblies by Means of the Guarded Hot Box (C 236) and indicates that meaningful determination would only be possible after moisture equilibrium is reached. Transient methods, such as the hot wire method, also present problems. Experimental errors are larger, since the hot wire does not present a surface of constant flux and the initial temperature rise in the wire at a location of high conductivity will be less than at a location of low conductivity, both of which exist in near proximity in a heterogeneous material such as concrete. However, Harmathy [39] offers two variable state methods that appear to overcome problems associated with other methods. One is based on fitting a temperature distribution curve used (assuming linear heat flow) to determine all thermal properties, and the other on the time-temperature rise response to heat pulses to determine diffusivity. Both are accompanied by the necessary mathematical interpretations.

Difficulties also plague direct measurement of true specific heat at elevated temperatures. Equilibrium at the test temperature is disturbed in making the determination by standard physical methods, and chemical dehydration reactions take place in the cement phase that may add or subtract heat. Such latent heat effects are overridden to some extent by the larger contribution of the aggregate fraction to the overall apparent specific heat of concrete, but must be taken into account if reasonably accurate results are to be attained in heat flow model studies. It is therefore not surprising that data from Harmathy [21], Harmathy and Allen [15], and other investigators show considerable dispersion in values for the specific heat of concrete. In general, specific heat appears to be insensitive to the aggregate used or to the mix proportions of the concrete. Values reported by Harmathy and Allen [15] for a variety of concretes over a temperature range from normal ambient to 650°C (1202°F) show a slight trend to higher values of specific heat with higher temperature. A high specific heat, including the contribution from moisture present, has the advantage of taking up more heat and delaying temperature rise in a fire.

Fortunately, thermal conductivity (*k*), thermal diffusivity (*h*), and specific heat (*c*) are simply related by the expression: $k = hcp$; where p is density.

Conductivity and diffusivity vary with the relatively small variation in specific heat with temperature. Density of the concrete is an important overall parameter when low density is due to air voids. This is most apparent between a dry lightweight concrete and a dry normalweight concrete. Moisture content has a significant role in heat conduction in concrete until dehydration is complete after which, and with decomposition of the constituents, the now dry, porous, normalweight concrete becomes a much better heat insulator than at lower temperatures. The larger the moisture content the larger the thermal conductivity according to data summarized by Zoldners [22]. For normalweight concrete, both thermal conductivity and diffusivity show considerable decrease at high temperatures. Harada et al. [40] cite a 50 % decrease at 800°C (1472°F) as compared with thermal conductivity at room temperature. For a lean concrete as compared with a rich one, a 20 % increase in thermal conductivity was noted assuming both were consolidated fully. From an examination of 16 concrete specimens made with Type I portland cement and a range of aggregates and processes used to manufacture concrete masonry units, Harmathy and Allen [15] confirm this order of reduction and find that all thermal diffusivities became approximately equal at about 600°C (1112°F); whereas at lower temperatures there was considerable difference between lightweight aggregate and normalweight aggregate concrete. The lightweight aggregate showed lower diffusivities and less reduction with increase in temperature than did concretes of normalweight aggregate. Their data on conductivity, while showing a similar trend, did not merge for the aggregate groups at the upper temperatures. These findings are supported by VanGeem et al. [41].

The three thermal properties so far discussed (conductivity, diffusivity, and specific heat) are important to over all behavior of concrete at elevated temperature because they essentially determine the inward progression of temperature rise into concrete and the rate and degree of temperature rise that may occur to steel or concrete in a fire. They are also important in heat flow calculations necessary in the design of structures for performance at elevated service temperatures as, for example, in nuclear reactors where significant temperature gradients are a feature of normal operations. On the microscale, they influence the thermal incompatibilities that occur between the cement paste and aggregates due to differential thermal volume changes.

Thermal Volume Change and Thermal Incompatibilities

The thermal expansion of the paste is highly dependent on moisture content while that of the aggregate is highly dependent on mineral composition. There is limited research referenced on the influence of moisture content and moisture retention of lightweight aggregates on the expansion of concrete. The observed thermal expansion in concrete is an additive of concrete moisture content and aggregate mineral composition. There are three aspects of volume change in concrete at elevated temperatures to consider: thermal volume changes; volume changes due to chemical reactions, for example, decomposition of calcium hydroxide or rehydration of the lime; and changes in physical form of the component materials, for example, the transition in quartz crystals. The term "thermal incompatibility" is used widely to describe those differential volume changes that result in differential strains and hence stresses between some types of aggregate particles and paste and between the constituent minerals of each. Unless relieved by creep or plasticity in the paste, these parasitic thermal stresses may be very high.

Looking at Philleo's data [19] for a calcareous aggregate concrete shows thermal expansion is linear up to about 260°C (500°F), then after a transition period there is a further linearity to about 427°C (800°F), but at a higher coefficient. Above 538°C (1000°F), the coefficient is again lower, probably due to a permanent dilation as a result of decomposition. Philleo's experimental technique seems fairly typical of those used to secure such data. The horizontal displacement between two wires hung vertically from gage points on a horizontal concrete beam is measured at the test temperatures, clear of the furnace containing the specimen.

Zoldners [22] summarizes data from other investigations that show that the thermal coefficient of expansion of concrete is in approximate proportion to the weighted value of those of the constituent aggregates once initial shrinkage of the cement paste due to dehydration has occurred. The coefficient of thermal expansion of concrete with increasing temperature thus tends to approach asymptotically that of the mineral type in the aggregate [40]. A point of particular interest in this data is that the expansion curve for limestone aggregate concrete is very close to that of mild steel over the temperature range up to 700°C (1292°F). Andesite, the "best" natural rock aggregate concrete Harada et al. [40] investigated, had about half the coefficient of thermal expansion of limestone or sand-stone concrete. Philleo [19] identifies decarbonation of calcareous aggregate as a factor.

Zoldners's own data [18] show a very rapid expansion above about 400°C (752°F) for a predominantly metamorphic and igneous gravel aggregate concrete and for a sand-stone aggregate concrete, as compared with limestone aggregate concrete and, in the case of an expanded slag or shale lightweight concrete, a slight overall contraction with an almost constant coefficient up to 900°C (1652°F). Holm and Bremner [32] reported similar findings for concrete masonry units. In all cases an initial shrinkage up to about 300°C (572°F) is noted. These data are for only one cement factor, one w/c ratio, and one aggregate-cement ratio. However, Philleo's work [19] did include three cement factors and corresponding w/c ratios. Above 427°C (800°F), these variables and initial curing (moist or air dried) had little effect; however, below 260°C (500°F), all these variables affected the coefficient values to a greater extent. Specimens having a higher w/c ratio and lower cement factor tended to have the higher coefficients.

Crispino [42] pursued an investigation to find the best combination of thermally compatible materials that would give good performance as a structural concrete for nuclear reactor pressure vessels at high temperature under thermal cycling. As compared with concretes containing limestone aggregate and a variety of portland and high-alumina cements, he found barytes aggregate with portland cement far superior.

The importance of thermal incompatibilities and the resulting thermal strains and stresses cannot be overemphasized. While the cement paste is shrinking as dehydration proceeds, the aggregate is expanding. After dehydration is complete, both are expanding, though at different rates. As a result, there is a clear, if not as yet fully explained, effect on the mechanical properties of concrete in which, as will be discussed, moisture effects may play a substantial role. Considerable hypothesizing continues toward a full explanation of the behavior of concrete at all temperatures with respect to thermal properties. Further reading is provided by Dougill [43].

Thermal Cycling

Since most investigations in the past have addressed fire resistance, it is not surprising that the effect of more than one thermal cycle has only really been addressed after interest arose in using concrete in nuclear reactor vessels. Both mechanical and thermal properties are affected [42].

The effect of thermal cycling on strength has been investigated by Campbell-Allen and Desai [44] and Campbell-Allen et al. [45], who compared the compressive strengths of limestone, expanded shale, and firebrick aggregate concretes up to 302°C (575°F) and noted a progressive deterioration with increasing number of thermal cycles though most of the loss occurred in the first few cycles. Similar effects are observed with flexural strengths and modulus of elasticity attributable to incompatible dimensional changes between cement paste and aggregate and to observed microcracking [30,44,45]. The interrelationship of thermal cycling, moisture content, and thermal volume change is also important in nuclear applications of concrete. Polivka et al. [46] examined concrete that allowed moisture a controlled escape or sealed it in, over durations up to 14 days of thermal exposure cycling up to 143°C (300°F). They showed the following:

- For a constant moisture condition (sealed) of the concrete, its thermal coefficient of expansion decreased during thermal cycling to 143°C (300°F). A similar decrease was observed for concretes that successively dried out during thermal exposure.
- After the first thermal cycle (21 to 143 to 21°C) (70 to 300 to 70°F) of the concrete in a moist condition, a significant residual expansion was observed, and this expansion successively increased with the number of cycles. This was also true for concrete specimens that were permitted to lose moisture during thermal exposure. A larger permanent expansion was observed for concretes of high moisture contents than for those of lower moisture contents.

In examining the significance of data from thermal cycling and sustained high-temperature tests, the nature of the thermal regime must be considered since many of the reactions influencing the measured thermal and mechanical properties including the internal transport or the expulsion of water, are dependent on time or the rate of temperature increase.

Review of Behavior Mechanisms and Influence of Moisture Content

The mechanical and thermal performance of concrete at elevated temperatures is determined by complex, interrelated physical-chemical behavior and strongly influenced by moisture content. The stresses from thermal loadings due to overall expansion or contraction, when added to those arising from mechanical loadings and restraint, determine the imposed total stress levels in the concrete. However, thermal stresses on a smaller scale between particles determine the resisting strength, compressive or tensile, of the concrete and the effect on other mechanical properties such as the various moduli. Thermal strain incompatibilities and the resulting parasitic thermal stresses are thus critical to performance of concrete at high temperature, especially where thermal cycling occurs.

Thus, investigators have explained their observations on compressive strength changes with temperature in general terms of the changes in physical properties of the cement and aggregate and the thermal incompatibilities between these components with temperature. From petrographic examination, Abrams [29] draws attention to micro-cracking and deterioration in the aggregate-to-paste bond. The advantage in compressive strength where the concrete is preloaded in compression is ascribed to the suppression of thermal micro-cracking in the cement paste, and the increase in compressive strength at moderately high temperatures has been attributed by Zoldners [18] to "a heat-stimulated cement hydration and densification of the cement gel due to the loss of absorbed water."

From his pulse velocity measurements, Zoldners [18] attributed the greater reductions in flexural strength as compared with compressive strength at the same temperature to micro-cracking. Whereas small defects would have little effect on compressive strength, they would immediately show up as a reduction in flexural strength.

Since the reductions in modulus of elasticity, Poisson's ratio, and bulk modulus, *K*, with increase in temperature follow the general pattern for reduction in compressive strength, these have usually likewise been put down to thermal incompatibilities, micro-cracking, and relaxation of bonds.

Lankard et al. [47] reviewed existing data secured on specimens at elevated temperatures from which moisture could escape, made their own tests on both sealed and unsealed specimens up to 260°C (500°F), and provided this summary:

- First, and probably most important, the influence of heat exposure per se on the structural properties of concrete is critically dependent on the moisture content of the concrete at the time of heating and is quite different for concrete in which the moisture is free to evaporate as compared to concrete sealed against moisture loss.
- For concrete that is heated slowly at atmospheric pressure, the primary factor influencing changes in the structural properties is the loss of free water. Partial loss of chemically combined water (dehydration of hydrated cement phases) occurs above 121°C (250°F) and primarily affects the flexural strength.
- For concrete slowly heated at saturated steam pressures, the primary factor influencing changes in the structural properties is the hydrothermal reactions that can occur in the cement phase and that convert the original highly cementitious calcium silicate hydrates into crystalline, lime-rich calcium silicate hydrates producing lower strength.
- Deterioration of structural properties under both moisture conditions (sealed and unsealed) can be expected from any test variable that can produce large temperature differentials in the concrete on heating (for example, quench heating and cooling, use of very large specimens, etc.).

While their data strongly support the role of moisture content in influencing mechanical properties of concrete at moderately high temperatures, they do provide somewhat of a challenge to the views expressed by others on the role of micro-cracking.

For unsealed specimens, they noted the micro-crack system was no worse after careful heating, possibly not as bad as that in the original concrete. This observation helps to explain that compressive strength decline is not significant below 260°C (500°F) (and in some cases may even increase) provided all the free water is removed on heating. It also adds to the usual explanation of the development of flaws and stress concentrations around flaws to account for the reduction in tensile strength being greater than that for compressive strength. In this case, they considered that the benefit of free water removal is offset by the loss of chemically bound water that reduces the critical stress requirements for tensile failure.

The removal of free water also appears to account for the observed independence of the reduction in modulus of elasticity with temperature at moderately high temperatures. However, once dehydration proceeds to a significant loss of other forms of water and to decomposition of the cement hydration products, and once aggregate deterioration and thermal incompatibilities increase with increasing temperature, then these latter mechanisms appear to account fully for the observed behavior. But it does appear that the presence or lack of free water is the controlling parameter at lower temperatures. Polivka et al. [46] established that at 143°C (300°F) a maximum reduction in strength and stiffness, in the order of 75 and 55 %, respectively, occurred at an intermediate moisture content corresponding to about a 50 % loss of the free water in the concrete.

Most of the data so far discussed on the thermal and mechanical properties of concrete have been secured by testing small unsealed specimens. The justifications for this are that most tests at normal temperature are made on small specimens and, in a fire, moisture is usually free to be progressively driven off from the concrete, sometimes accompanied by spalling in which moisture expulsion plays a significant role. However, nuclear reactor shields and primary containment vessels present a different set of conditions in which the concrete essentially is sealed against moisture loss. The prestressed concrete sections tend to be massive and concrete may be hot and sealed by a steel liner on one side, even where it is cool and open on the other. In hot sealed areas, the exposure conditions may be quite akin to autoclaving mature concrete and, with chemical and mineralogical changes in the component phases of the concrete and restriction on the mobility of moisture, there is a greater reduction with temperature in the physical properties of concrete than occurs when the moisture is freely driven off.

In “saturated steam” conditions, Lankard et al. [47] found a continual and much greater decline in compressive strength and modulus of elasticity up to 260°C (500°F). Compressive strength at the upper temperature was only 50 % of original while the modulus of elasticity fell to only 31 % of the original value. In observations of the composition of the hydrated cement phases under these sealed heated conditions, they determined from X-ray analysis the presence of new, more highly crystalline, probably lime-rich calcium silicate hydrates, some of which, if they occur in autoclaved products, are known to lead to lower strengths.

The amount of free water in concrete also has a major influence on thermal properties. The contraction of cement paste is due to the expulsion of free water and, while water is a relatively poor direct conductor of heat as compared with aggregate, it is a many times better conductor of heat than is the air that would be occupying voids, capillaries, or pores if the water was not present.

Because it influences almost every parameter of concrete behavior, including spalling, control and uniformity of moisture content in the concrete prior to fire tests has been of long standing concern. This led to investigations and proposals to modify ASTM E 119 by preconditioning the specimens [48]. Natural drying has been replaced with a specified conditioning regime of relative humidity and temperature and moisture content at the start of the test. While it is still not possible to cover every circumstance of possible moisture condition in a standard test, the present requirements are an improvement and save time and money. Carlson and Abrams, in discussion on Harmathy [49], suggest that the actual fire test should be conducted on a specimen conditioned in a manner such as to produce a microstructure within component material which compares to that of similar material after several years of service. There may be a need for further research and testing of concrete elements, especially with regard to the moisture content and retention involved with lightweight aggregates and the more dense high strength concrete using silica fume or similar materials for increased strength, both of which are discussed later, as work by Shirley et al. [50] found that the fire endurance of high-strength concrete slabs with and without silica fume and normal strength concrete slabs did not significantly differ and none of the test specimens exhibited spalling on the exposed surface nor any explosive behavior.

Many of the disparities in the results of physical tests for strength and other properties in the referenced investigations are probably explained by lack of control of the moisture parameter and the temperature gradient in the concrete. However, this apparent lack of control is the real situation when mature or young concrete is exposed to fire and sometimes it spalls and sometimes it does not.

Spalling and Cracking

Spalling of concrete surfaces, particularly at an arris, is an evident feature of most fire-damaged concrete. The worst consequence of this process, when it occurs on the fire exposed surfaces of concrete elements is that it opens up fresh surfaces to the heat source, thus making for more rapid progression of heat into the concrete and increasing the probability of embedded steel being adversely affected, especially if it becomes exposed. Spalling of concrete elements may contribute to prematurely reaching a temperature rise endpoint of ASTM E 119 on the unexposed side due to the loss of section and corresponding reduction in thermal resistance of the concrete element. Most investigations addressed two possible causes of such spalling: thermal incompatibilities and moisture entrapment. For many years, direct thermal causes, in particular such things as the 2.4 % increase in volume of quartz at 573°C (1063°F), were considered a main contributing factor. However, the quartz volume change may not be as significant as earlier thought. Experienced investigators such as Harmathy and Allen state they have never identified spalling due to this cause [15]. It seems that by the temperature necessary for quartz expansion to occur the plasticity of the paste is great enough to accommodate the expansion, though sudden popouts of siliceous aggregate particles at or near the surface will likely occur. Moisture entrapment now is identified by Meyer [51] and Harmathy [49] as predominant, at least in the earlier stages of spalling deterioration. A commonly held view is that the disruptive pressure causing spalling is the result of rapid heating with sufficient moisture present so that, even after the evaporable water escapes, closely bound water in capillaries and gel pores will produce saturated or superheated steam with explosive force. However, this does not appear to be an adequate explanation, and it is worthwhile to examine a more plausible explanation in some detail.

Harmathy [49] (including his work with Shorter) developed an explanation called "moisture clog spalling" along the following lines. As moisture is desorped in a layer adjacent to the heated surface, the vapor migrates toward the colder region and is reabsorbed. As the thickness of the dry layer increases, a completely saturated layer builds up at some distance in from the exposed face and later a sharply defined front forms between the two layers. As the temperature of the exposed surface increases, a very steep temperature gradient develops across the dry layer resulting in a high rate of heat flow, intensified desorption at the frontal plane, and vapor pressure buildup. Because the concrete to the rear is saturated, the vapor can only leave through the dry layer, expanding and meeting increasing flow resistance as it goes. If the permeability of the material is low, the pressure build up at the front continues until a tensile spall occurs separating the dry layers from the rest. However, in discussion of Harmathy [49], Carlson and Abrams advise that when a specimen is dried to an equilibrium relative humidity of 70 %, the condensation and re-evaporation process is such that dangerous steam pressures are not likely to develop. They observed liquid water being discharged from fine cracks and surface blemishes into the hot furnace. Escape of water was also observed in column tests by Kodur et al. [10]. Carlson and Abrams observed that thermal spalling is related to restraint of thermal expansion citing that: for thin slabs within a heavy restraining frame the spalling, if one can call it spalling, has been sufficiently violent to fail the specimen in a single occurrence. Shirley et al. [50] did not observe any spalling on the exposed surface nor any explosive behavior in slabs made of high-strength and normal-strength concrete.

In establishing an overall picture of the effects of moisture on the fire resistance of concrete, Harmathy [49] stressed two very important practical points. First, high moisture contents are conducive to spalling but if spalling did not take place (because, if resistance of the pores to moisture flow was not too high, the existing large vapor pressure differential would move the "moisture clog region" towards the colder region and the pressure buildup would level off), then the moisture in the concrete is most beneficial. Second, the absorption of heat associated with desorption of moisture checks the rise in temperature in the concrete.

Data on the thermal properties of cement paste, aggregates, and concrete emphasize the thermal contraction and expansion strain incompatibilities that develop with increasing temperature, within and between the constituents, and between the hotter and cooler parts of the concrete itself. The resulting thermal stresses may be sufficient to induce cracking. Thermal cracking may range from crazing on the surface and micro-cracking to deeper and substantial spalling or cracks of structural significance at locations where overall structural deformations or thermal stresses exceed the tensile capacity of the concrete. Planes of weakness may also develop at the level of the reinforcing steel, because of thermal shock, if the surface of the concrete is quenched with water.

Selvaggio and Carlson [52], in fire tests of prestressed concrete beams, observed some cases of longitudinal tensile splitting through the full depth of the flanges and webs when the temperature at the level of the steel tendons was as low as 149°C (300°F). By analysis, they established that the differential thermal expansion between concrete and embedded steel can be a source of tangential tensile (bursting) stresses of considerable magnitude. Two concrete factors were considered to contribute to lack of resistance to these stresses, (1) a high modulus of elasticity and (2) reduced tensile and compressive strength of concrete when simultaneously exposed to tensile and compressive stresses (from the longitudinal prestress), together with factors originating from the steel arrangement and moisture expulsion.

Sometimes it appears as though additional spalling occurs as the concrete cools down. While the normal contractions are opposed by a volume increase if moisture is present to rehydrate the calcium hydroxide, these processes are not rapid and most likely the apparent spalling is simply the falling away of already loosened pieces of concrete.

Investigation and Repair of Fire Damage

Once the structure is safe to enter after the fire, a first step is to try to obtain an estimate of the temperature reached and its duration and hence a first estimate of the likely remaining properties of the concrete. Many circumstantial clues may be found by sifting through the debris and identifying the remains of common objects that have known melting points. The appearance of the concrete can also provide important evidence, as described by Bessey [53,54]. Permanent color changes may accompany dehydration of the cement paste. As temperature increases, the color of siliceous or limestone aggregate concrete changes through pink or red to gray at about 600°C (1112°F) to buff at about 900°C (1652°F). Harmathy [55] proposes thermogravimetric and dilatometric testing of samples secured within a day or two of the fire as a means of determining a temperature history inwardly into the concrete. The approach is based on the fact that the continuum of reactions in the cement paste remains frozen at the temperature level reached (for at least a few days) and, by comparison with a reference sample of unaffected concrete or standard data, this temperature marker can be identified. In 1980, Placid [56] proposed a test based on thermo-luminescence that requires only small samples and is sensitive to the thermal exposure experienced rather than just the maximum temperature reached. More recently, Riley [57] has explored the potential of the petrographic analysis of thin sections to assess damage.

Abrams and Erlin [58] provide a method based on examining the microstructure and micro-hardness of prestressing steel for estimating the temperature it reached in a fire and hence it's remaining tensile strength. Though the effect of the temperature on normal reinforcing steel is not so critical, similar metallographic methods may be useful.

Once the likely temperature regime has been established, the available data on the engineering properties of the concrete and steel, at either the elevated temperature reached or after cooling, can be incorporated in the appropriate structural analysis to determine the mode of failure, if the structure should be demolished or can be repaired.

Confirmation of the residual engineering properties of the concrete and steel after cooling can be determined by securing samples for standard laboratory testing. Standard methods for in situ nondestructive testing such as ultrasonic pulse velocity measurements can be used to help delineate the damaged areas and compare the properties of the affected concrete with that untouched by the fire. Where doubt still exists after the structural analysis or repairs have been completed, confirmatory full-scale load tests can be made.

Smith [59] described the investigative, material, and structural evaluation techniques and restoration methods available to examine concrete damaged in construction fires.

Twenty-five years later he evaluated the performance of the remedial work undertaken and reviewed developments in investigative techniques over the intervening years [60]. Numerous accounts of investigations and repair of fire damage to mature concrete have been published. The account by Fruchtbaum [61] in 1941 remains a classic, and Ref 54 provides an excellent general review.

Lightweight Aggregate Concrete

The thermogravimetric, dilatometric, and thermal studies made by Harmathy and Allen [15] on concrete used in masonry units confirm the reasons why concretes made from lightweight aggregates have superior heat resistance to normalweight concretes. Lightweight aggregates (manmade expanded shale, clay, slate, slag, perlite, or mica and natural pumice or scoria) have significantly lower thermal diffusivities (up to 600°C (1112°F)) and thermal conductivities (at all temperatures). The capacity of the aggregate to retain moisture and its chemically stable vesicular and glassy (amorphous) composition are also advantageous. Abrams [29], Holm and Bremner [32], among others, point out that the compatibility in relative stiffnesses between the cement paste and lightweight aggregates is also beneficial. They further noted a good bond strength between paste and aggregate.

Zoldners [18] (with Wilson in subsequent work) looked at the residual engineering properties of lightweight concretes after exposure to temperatures of 300, 500, 700, and 1000°C (572, 932, 1292, and 1832°F) and after cycling 5 or 25 times from 100 to 300°C (212 to 572°F). They concluded from ultrasonic pulse velocity, compressive, and flexural strength determinations made after cooling that all the lightweight (expanded shale and slag) concrete stood up to the effects of high temperature better than did semi-lightweight concrete. They also found that, although there was no advantage in using Type IS (blended portland blast-furnace slag) cement as compared with a normal Type I cement, the addition of micro-fillers (fly ash, silica flour, and calcined shale dust) as 20 or 40 % cement replacements did improve the heat resistance of the concretes up to 500°C (932°F). In all cases where micro-fillers were used, the improvement in residual flexural strength was in the range of 10–20 % though similar improvements were not always noted in compressive strength. The pulse velocity measurements, taken to be an indicator of porosity and micro-cracking in the matrix, generally supported the conclusions drawn from the strength data. Residual strengths for all lightweight concretes were reduced to about 80 % by heating to 300°C (572°F) and to about 45 % by heating to 700°C (1292°F) as compared with the unheated reference concretes. After heating to 1000°C (1832°F), residual strengths were negligible. Since failure is largely because of the deterioration of the cement paste matrix, by using high alumina cement rather than portland or blended cements, insulating lightweight refractory concretes good up to about 950°C (1742°F) can be made. Very lightweight (cellular, vermiculite, or perlite insulating) concretes, 480 kg/m^3 (30 pcf), as used in roof or other building applications, enhance fire endurance [9].

Heavyweight Aggregate Concrete

Heavyweight concrete is used for radiation shielding in nuclear reactors. This shielding is usually in the shape of a hollow cylinder or sphere. Under moderately high operating temperatures, thermal gradients induce moisture migrations, and complex triaxial stresses can occur. In summarizing the results of compressive strength of portland cement concrete containing limonite, serpentine, magnetite, and steel punchings, and combinations thereof, Desov et al. [62] reported reductions of up to 40 % in hot-tested specimens after heating to 350°C (662°F) for between 3 and 7 h. As with normalweight concrete, these effects were ascribed to differences in thermal expansion between the cement paste and aggregates, and to the dehydration of the paste.

Very High Strength Concrete

Most of the data and their significance so far discussed have been from tests on concrete in the normal strength range used in general constructions. Very high strength concretes ranging in strength from 80 MPa (12 000 psi) to as high as 170 MPa (25 000 psi) have been used, or proposed for use, in tall buildings or off-shore structures where enhanced mechanical properties lead to cost effectiveness. It has been observed in laboratory investigations that very high strength concretes, especially if saturated, have a tendency to spall explosively when heated rapidly [6,16,63]. These concretes are more “brittle” with a denser cement paste matrix obtained by use of high cement factors, silica fume, fly ash, and super-plasticizers. Since the cement paste matrix will carry higher loads than in normal-strength concretes, maintaining a more homogeneous stress distribution with the aggregate, the decomposition of the cement paste with temperature rise is a critical factor in fire resistance. Coupled with the slower drying out of moisture due to the less porous microstructure, this increases the risk of damage from fire at an early age. Otherwise the mechanical properties appear to decline with increasing temperature in a similar trend to normal strength concretes. VanGeem et al. [41] found that the thermal conductivity and thermal diffusivity of high strength concrete made with normalweight aggregate were similar to those for conventional strength normalweight aggregate concrete.

Currently most applications of very high strength concrete are limited to concrete columns. Research by Shirley et al. [50] addressed slabs. In this work, high-strength concrete slabs with and without silica fume and a normal strength concrete slab were tested for 4 h. None of the five specimens exhibited spalling on the exposed surface nor any explosive behavior.

Refractory Concrete

Concretes used to withstand prolonged high temperatures, for example, in monolithic linings for kilns, can be made with high alumina cements and common refractory materials, such as crushed firebrick, as aggregate [64]. This type of concrete will be stable and retain considerable strength at dry heat temperatures of up to 1300°C (2372°F) because ceramic bonds have replaced the hydraulic bonds that were lost during desiccation. By using aggregates such as fused aluminum oxide with a cement that is essentially calcium aluminate, service temperatures for a refractory concrete can be as high as 1800°C (3272°F). The report of ACI Committee 547 [65] provides a definitive discussion of refractory concrete properties for those having deeper interest.

Elevated Temperature Coupled with Air Blast

At the time of the introduction of jet aircraft into service, there was considerable concern that the hot blast might be damaging to concrete in engine test cells, warm-up aprons, and runway ends. Immediately at the jet exhaust, temperatures of 677°C (1250°F) occur and this may be as high as 1927°C (3500°F) where afterburners are used. The velocity of the attendant hot blast has been estimated at 1067 m/s (3500 ft/s) and impingement on the concrete was particularly exaggerated by the downward inclination and low slung engines on many early jet aircraft. Tail and higher wing mountings in more recent planes have largely removed the problem from the practical sphere and a 1953 paper by Bishop [66] remains definitive.

The launching of rockets from pads or underground silos for space exploration or military purposes presents another aspect of the hot blast problem and is one for which all the answers have not yet reached the public domain. However, it is known that the solutions tried include sacrificial concrete and protective shields including ceramic ablative shields. However, the conditions though extreme, involving temperatures of about 2760°C (5000°F) and velocities over 2438 m/s (8000 ft/s), last for only a few seconds and the choice between sacrificing the concrete or trying to shield it is one of cost and convenience.

Concluding Remarks

Despite potential deficiencies in performance at elevated temperatures arising from dehydration and thermal incompatibilities, concrete has a long-standing and justified reputation as a fire-resistant material, notwithstanding a long-recognized need for better means of testing and specifying endurance [1,67]. Development of temperature regime-structural behavior models and greater understanding of thermal and mechanical properties are leading to significant improvements in specification and design against the extremes of fire and for moderately high temperature applications such as nuclear reactors. While striking advances have been made, full physical-chemical, thermodynamic explanations that tie together all the aspects of the very complex visco-elastic, moisture-dependent behavior pattern of concrete at elevated temperatures are still awaited.

As discussed by Abrams and Orels [48] there may be benefits to additional work correlating the many variables in concrete elements including mass of the specimen, shape of the specimen, design of the concrete mix, type of aggregate, age of specimen, conditioning and drying procedures and environment, physio-chemical changes. Work by Kodur et al. [10] suggests that this should be expanded to include size, spacing, and configuration of reinforcing steel.

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Air Content and Density of Hardened Concrete

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Preface

IN PREPARATION OF THIS CHAPTER THE CONTENTS of the 4th edition were drawn upon, as the 4th edition had drawn upon Samuel Helms's clear and concise work in the previous three editions of this ASTM Special Technical Publication. The discussion of air content is more extensive here, however, largely in response to the continuing interest and research in air-entrained concrete and freeze-thaw durability. It has also become evident that the further in time that the industry has come from the landmark contributions of the early workers in air-void systems and frost resistance in concrete, the less well understood are the fundamental principles laid down in the late 1940s and early 1950s. Following the pattern set by Helms, the present edition therefore reviews much of this key literature to explain the origins of today's state of the art. Finally, this edition appears when there is a proliferation of the use of the ASTM Practice for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete (C 457) microscopical analysis procedure, which was once exclusively performed by a small cadre of experts. While expansion of the use of the test has produced more data, it has in some cases produced more confusion where less experienced operators have used nonstandard equipment and procedures. The chapter has therefore been written in part to point out pitfalls and sources of error in performing and interpreting the test.

Part I: Air Content

Introduction

The air content within a given volume of concrete is the cumulative volume of a large number of air voids of multiple sizes ranging from microscopic bubbles to larger, irregularly shaped air pockets. These voids have their origins in the air initially trapped among the dry constituents, dissolved air in the water, and the air introduced as a result the kneading and folding action of mixing and trapped in the mix while depositing the concrete in the forms [1].

The presence of these air voids initially suspended in the mix water among the solid particulate constituents of the concrete influences the workability, consistency, bleeding tendency and yield of the fresh concrete, and the density, strength, and frost resistance of the hardened concrete. The most significant of these effects is the influence of air voids in mitigating the damaging effects of freezing and thawing of absorbed water. The magnitude and utility of these effects depends on not only the total volume of these air voids, but on the entire size-distribution of voids and on their dispersion throughout the hardened concrete. The beneficial consequences of air voids are obtained by using air-entraining admixtures and appropriate concrete production and construction procedures to encourage the retention of the smallest voids, and, by using effective placing and consolidation procedures to reduce the number and volume of the largest voids.

While the concrete is still plastic, however, the air voids have an opportunity to move, become larger or smaller, coalesce, or change shape. Some voids can be removed from the concrete entirely. The total air content and other air-void characteristics therefore depend on the stage in the mixing, transport, placement, and consolidation processes at which the measurement was taken. Once the concrete has hardened, however, permanent void spaces remain, preserving the size and shape of those air bubbles present at the time of setting. All air voids remaining within a given concrete mass, regardless of their size, shape, or origin, are often referred to as the "air-void system." In order to evaluate the characteristics of the air-void system, it is presently necessary to obtain a sample of the hardened concrete, perform a statistical analysis on a fraction of the exposed voids observed through a microscope, and to estimate the relevant characteristics to various degrees of accuracy and precision. The significance of such sampling, testing, and analysis, and the interpretation of the results obtained are the subjects of this chapter.

Influence of Air Content on the Behavior and Performance of Concrete

Introduction

Incorporating air voids into concrete influences the behavior of the material in both the fresh and hardened states. The mag-

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nitude and degree of utility of these influences depends on the total volume, sizes, and dispersion of the air voids, and on the material properties of the concrete.

Workability

A large number of microscopically small voids, well-dispersed throughout the cement paste, will generally increase workability at any given water content. This is because sufficiently small air bubbles can separate solid particles in the mix [2], acting as air-cushions to reduce inter-particle friction. In proportioning concrete mixes, one can take advantage of this increased workability by reducing the water content by 6–12 %, depending on aggregate gradation and other constituents, while maintaining slump [3–5].

Cohesion

When air-entraining admixtures are used to stabilize the smaller air voids, a mutual attraction develops between microscopic bubbles and the grains of portland cement. This attraction not only “anchors” the air bubbles to inhibit their loss from the fresh concrete due to buoyancy, but imparts a beneficial cohesion to the mix that reduces segregation, settlement, and bleeding [1]. In some mixes, this can also cause air-entrained concrete mixes to become “sticky” with increased adhesion to construction equipment and increased drag on finishing tools [4,5].

Density and Yield

By displacing heavier components in the concrete mixture, the air voids reduce the density of the mix. As described in the chapter by Daniel and Part II of this chapter, the density test, ASTM Test Method for Density, Yield, and Air Content (Gravimetric) of Concrete (C 138), can therefore be a useful means of measuring air content in fresh concrete.

Strength Reduction

By occupying space between the cement grains, creating a more porous cement paste, and reducing the density of the hardened concrete, air voids will reduce the strength of concrete. Thus, at equal water-cement ratios, the impact of air content decreases compressive strength, with various rules of thumb equating each 1 % increase in air content to a 3–5 % reduction in 28-day compressive strength [1]. More significant strength reductions have been reported [6], especially when the air bubbles cluster at the paste-aggregate interface [7,8]. Except in these latter cases, part of the potential strength loss can be compensated for with the water reduction due to air entrainment [9].

Finishing and Blistering

Blisters or shallow delaminations (2–3 mm thick) can develop on the finished surface of slabs due to the generally inadvisable combination of a steel-troweled finish and air-entrained concrete [4,10,11]. Although the mechanism of blister formation is not clear, there is an alarming coincidence of blisters or shallow delaminations formed on the surface of air-entrained concrete slabs when a steel-trowel finish is applied. This problem is apparently more severe the earlier the troweling is applied. While there are many examples of successful hard-troweled floors with air-entrained concrete, the appropriate timing of finishing relative to bleeding, surface-stiffening, and ambient conditions is apparently more difficult as air content gets higher. It is therefore difficult to achieve a steel-troweled finish on concrete with air content higher than about 3 %. With a few specific exceptions, non-slip, high friction floor surfaces are generally desirable where the concrete is routinely exposed to freezing conditions, making steel-troweled floors and high air contents typically mutually exclusive [12].

Frost Resistance

While water reduction, reduced bleeding, and resistance to segregation are useful characteristics of air-entrained concrete, the primary reason for accepting side-effects such as strength loss, variable yield, and finishing complications is the increased frost-resistance afforded to the hardened cementitious paste. This is accomplished through several mechanisms as described in the next section.

Nature of Concrete and the Mechanism of Freeze-Thaw Damage

Mechanism of Freeze-Thaw Damage

Concrete is fundamentally a porous material composed of coarse and fine aggregate particles of varying porosity, held together by a hardened cementitious paste whose porosity depends on the original water-cement ratio, the effectiveness of consolidation, and on subsequent curing conditions. Water can be absorbed into the pores in the aggregate particles and into the “capillary pores” of the hardened cementitious paste [13–15].

These capillary pores can be saturated to various degrees, as influenced by residual, non-evaporated mix water or the subsequent absorption of water from the environment. At sufficiently low temperatures, this water freezes and expands in volume by 9 %. When absorption of water has filled the capillaries to the point where the remaining empty pore space cannot accommodate expansion of the ice, the volume of the hardened concrete itself will be forced to expand with accompanying tensile or bursting stresses. Depending on the rate at which the freezing takes place and whether salt water or deicing agents are present, the expansive pressure can originate in the actual expansion of ice, movement of unfrozen water escaping the advancing ice front [16], osmotic pressure caused by differential salt concentrations, or by differences between the thermal volume changes in ice and in paste [13–15,17–24]. Since these pressures are generated with the onset of freezing and ultimately relaxed upon complete thawing, multiple freeze-thaw cycles “fatigue” the concrete. Whether physical damage occurs depends on the level of stress and the frequency of these fatigue cycles, and on the strength of the concrete. Air voids are intentionally incorporated into the cementitious paste to reduce (but not eliminate) the stresses generated with each freeze-thaw cycle, and thus increase the number of cycles to failure.

Influence of Air Voids

Air voids in concrete intersect the network of capillary pores in the hardened cementitious paste. Since the largest of the capillary pores are typically smaller than the minimum diameter of an air void (0.010–0.020 mm or 0.0004–0.0008 in.) absorbed water remains in the smaller capillaries, moving towards the air voids only when under the pressure generated by rapid freezing. Under normal conditions, the air voids therefore remain empty and able to accept either ice or unfrozen water when a freezing cycle begins.

Upon rapid freezing, water and ice move towards the vacant air voids under a pressure gradient that is highest at the freezing site and lowest at the air void. The pressure difference required to move water or ice increases with distance of travel [16,25,26]. This pressure is minimized by a well-dispersed system of air voids intersecting the capillary network at many points, providing a minimum travel distance from any point in the paste to the nearest air void.[2]

Just how far the water or ice can travel without generating damaging pressures depends on degree of saturation, rate of freezing, porosity, permeability, degree of hydration of the hardened cementitious paste, viscosity of the water, and the tensile strength of the concrete [16,17,25,26]. For any given cement paste and set of environmental conditions, one can theoretically compute a critical distance (Powers's term was "critical thickness") beyond which movement of ice or water will generate damaging pressures. Damage results when absorbed water in a portion of the concrete must move further than this critical distance to arrive at the nearest air void. Powers calculated this theoretical distance as 0.25 mm (0.010 in.) "or thereabouts" for commonly encountered pastes and for freezing conditions that are markedly more severe than typically encountered in nature [15,26,27].

As a consequence of the critical distance, any given air void can provide frost protection to only the hardened cementitious paste falling within a sphere of influence radiating outwards from the periphery of the void. The zone of protected paste occupies a "shell" surrounding the air void with a thickness approximately equal to the critical distance. (Among the mathematical details of the development of the "protected paste shell" concept is a reduction in the critical distance due to the curvature of the bubble surface, and the limiting condition that the volume of freezable water in the shell must be no greater than the volume of the bubble [25,28]. Shell thickness is therefore not independent of bubble size, but tends to be so as the bubbles become larger. Fagerlund [29] further suggests that the smallest air voids, similar in size to the capillaries, may not be dry at the onset of freezing, thus reducing their effectiveness.)

Requirements for an Effective Air-Void System

To effectively provide frost resistance, the air-void system must have a total volume of empty air voids that equals or exceeds the volume of water or ice not accommodated by empty space in the capillary pore system. (Unsaturated concrete would need less air than saturated concrete, and concrete that is continuously dry would need no air at all.) Of equal importance, the air voids must be dispersed throughout the cement paste so that nearly all of the paste is within the protective shell of one or more air voids [16,25,26,28]. As will be discussed, the precise requirements for volume, dispersion, and spacing of the voids depend on both the concrete and on the environment. Meeting such requirements will not eliminate the stress that accompanies freezing but will maintain it at tolerable levels.

Principle of Achieving Dispersion and Acceptably Small Bubble Spacing

Achieving dispersion and an acceptably small spacing for a given air content requires that the air-void system be comprised of a large number of small bubbles. As the same volume of air is subdivided into smaller and smaller bubbles, the total bubble surface area increases geometrically, as does the cumulative volume of the protected paste shells around the bubbles.

Influence of Factors Other than Air Volume and Air-Void Dispersion

The volume of freezable water initially present in the pores, or the "degree of saturation" will depend on the initial porosity and age of the concrete, curing conditions, and environmental exposure. Similarly, the velocity at which the water or ice must move through the capillaries enroute to the air voids depends on the rate of freezing, another environmental variable. As pointed out earlier, rapid freezing conditions probably induce water movement, while slow-freeze conditions are more likely to induce the movement of ice [13,16,17,25,26]. Mattimore and Arni et al. [30,31] demonstrated reduced freeze-thaw damage at slower rates of freezing, while Flack [32] produced data showing rate of freezing effects to be more complicated.

It can also be shown that the pressure developed as water moves towards the air voids increases with a reduction in paste permeability and with an increase in fluid viscosity [13,16,17,25,26]. The additional component of osmotic pressure is influenced chiefly by the presence of deicing salts and other dissolved solids.

Details of the interaction of air-void geometry, material properties of the paste, and environmental conditions are described in detail by multiple researchers [13–17,23,25,26,28, 33–38].

Influence of Frost Resistance of Aggregates

Freezing and thawing damage (frost damage) to concrete can result from the mechanisms of paste destruction described, or from the expansion of absorbed water in the aggregates, or both. The air voids protect only the hardened cement paste and do not improve the inherent frost resistance of the aggregates. As Powers described it, concentrating on air voids "ignores 75 % of the problem" of the frost resistance of concrete (assuming 75 % of the volume of the material is composed of coarse and fine aggregates) [26]. Obtaining frost-resistant concrete therefore requires the selection of frost-resistant aggregates combined with the incorporation of appropriately sized and dispersed air voids in the paste. The selection or testing of frost-resistant aggregates is discussed in other chapters of this publication.

Origin and Geometric Characteristics of Air-Void System in Concrete

Introduction

With or without the use of an air-entraining admixture, air is unavoidably present in fresh concrete as a remnant of the air initially present in and among the dry mix ingredients [16], and subsequently incorporated by the kneading and folding action of mixing, and as a result of transporting and depositing the concrete in the forms [1,33]. While the regular action of controlled mixing can stimulate formation and entrapment of a fairly well-defined grading of predominantly small air voids, the more random activity of depositing the concrete in the forms tends to trap randomly sized voids, often much larger than those stabilized during the mixing process.

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[2] Under slower freezing conditions, water can move from coarser to finer pores because the vapor pressure over the surface of liquid water in small pores is greater than the vapor pressure of water over ice in the larger pores at the same temperature [5, 15, 19].

Air-Void Systems without Benefit of an Air-Entraining Admixture

In ordinary concrete mixed without the stabilizing effects of an air-entraining admixture, a large number of the smaller air bubbles initially formed in the mixing process are lost from the system, leaving primarily larger voids in the hardened concrete. The problem with this is not that the larger air voids do not provide frost resistance; since a protected paste shell exists around the periphery of all air voids in concrete regardless of their size or origin [5,17]. The problem is that unless the air content is extraordinarily high, the large voids offer too little volume of protected paste. (The volume of protected paste relative to the volume of the air voids themselves increases for smaller air voids.) Large voids are therefore not ineffective, they are simply less effective in providing frost resistance than a much greater number of smaller voids occupying the same total air volume.

Function of Air-Entraining Admixtures

Air-entraining admixtures stabilize the smaller air voids in the mix by promoting their formation, retention, and dispersion. These admixtures, related to soaps, detergents, and the general class of chemicals known as "surface active agents" stabilize smaller bubbles by a combination of surface tension effects and precipitates at the bubble wall [5,39]. These admixtures also induce electrical charges on the outside of the bubbles that not only causes mutual repulsion and thus impedes coalescence, but also anchors the bubbles to cement grains and other charged particles. This latter mechanism provides some resistance to bubble loss due to buoyancy [1,15,40–43]. As pointed out by Whiting and Stark [1], however, "entrained air is produced by the mechanical stirring, kneading, and infolding actions of the concrete mixer"; the air-entraining admixture merely stabilizes these bubbles once they are formed.

Air-Void System Resulting from the Use of an Air-Entraining Admixture

As reported by Powers [26], "Comparison of sections of concrete made of the same materials, with and without an air-entraining agent, indicates that the voids present when an entraining agent is not used are also present when the agent is used." The consequence of using an air-entraining admixture is therefore to augment the coarser or "natural" air voids with a large number of the smaller voids stabilized during the mixing process. The resulting air-void system is therefore a "composite" [26] of the smaller voids stabilized and trapped in the concrete during mixing combined with the much larger voids trapped in the concrete as a result of handling and placing.

The term "entrained air" as conventionally used is intended to apply to those voids trapped during mixing and stabilized by the air-entraining admixture. In confusing contrast, the term "entrapped air" is generally intended to apply to those voids trapped in the concrete during handling and placement. Given that these larger, so-called "entrapped" air voids reduce the density and strength of the concrete to a greater degree than is justified by their limited contribution to frost resistance [13,44], it is normally advantageous to remove them from the fresh concrete by appropriate consolidation techniques.

The Composite Air-Void Gradation

While not implied in the literature, a common industry misinterpretation of the terms entrained and entrapped is that air

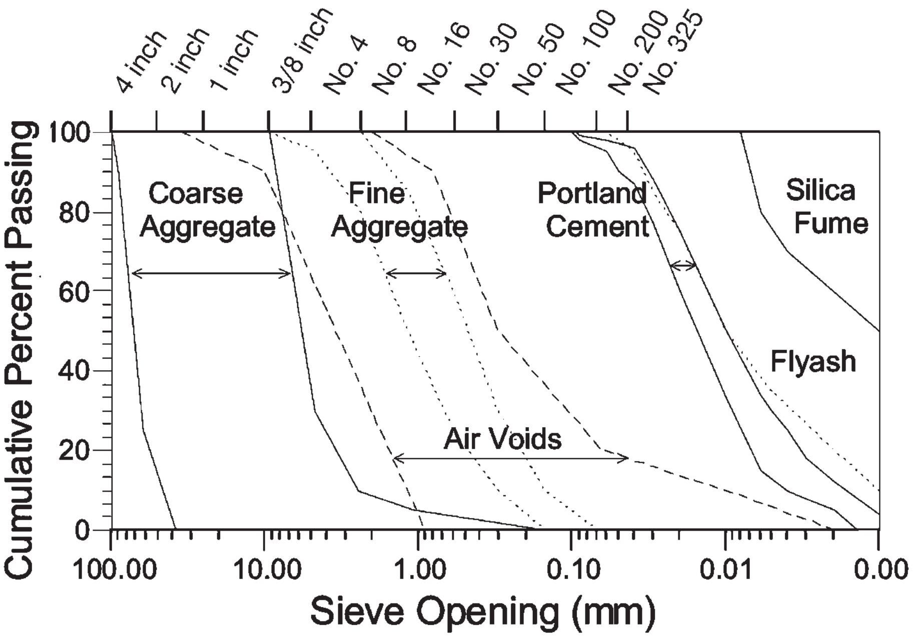

Fig. 1—Example grading curves of concrete constituents. Vertical axis represents the mass or volume "finer than" the size indicated on the horizontal axis. Mass or volume percent is shown as a fraction of the total mass or volume of that particular constituent. Coarse and fine aggregate grading bands are from ASTM C 33. Cement gradings are examples of coarse and fine grinds. Fly ash and silica fume examples are taken from Ref 45. In the coarser of the two air-void gradings, 90 % of the total air volume is contained in voids larger than 1 mm in diameter. In the fine air-void grading, 90 % of the total air content is contained in voids smaller than 1 mm in diameter. An air-entraining admixture is normally required to stabilize the air bubbles in such a mix.

voids in concrete are of two sizes only: large "entrapped" voids and small "entrained" voids. It is observed that the voids actually occupy a broad grading ranging from 10 μm to several or hundreds of millimetres–a size range from smallest to largest of more than a factor of 1000. As shown in Fig. 1, the air voids occupy the broadest range of sizes of all the constituents of concrete.

Despite this continuous grading of air voids, the simplest way to describe the size distribution of bubbles, voids, or aggregates is to report the number or volume of constituent parts larger than, and smaller than, some predetermined characteristic size. For air voids, ASTM Terminology Relating to Concrete and Concrete Aggregates (C 125) sets a size criterion of 1 mm (0.04 in.) and defines "entrapped" air voids as those above this nominal limit, and "entrained" air as those below. Underlying this definition by size may be the observation that retention of a significant number of air bubbles much less than 1 mm in diameter is unlikely without the stabilizing influence of an air-entraining admixture. Alternatively, air voids of greater than 1 mm in diameter or so can be present with or without the influence of an air-entraining admixture [26].

The Shape of Air Voids

While the smaller air voids in concrete are generally observed to be spherical in shape or nearly so, the larger voids are often seen to be non-spherical and irregularly formed. The observation that void shape becomes increasingly irregular with void size is observed in many other physical systems [46], and is not necessarily related to the use of an air-entraining admixture or to the effectiveness of the air voids in providing frost protection.

Caveat Concerning Terminology

As described earlier, the terms "entrained air void" and "entrapped air void" as defined in ASTM C 125 have specific meaning only in regard to the size and shape of the voids. These same terms can be misleading when used to imply the origin, evolution, or effectiveness of the voids. As has been discussed, "air-entraining admixtures" do not strictly "entrain" air into fresh concrete. Further, the smaller voids classified as the entrained air voids are in fact the result of air having been "entrapped" in the mix and stabilized via the admixture. The term "entrapped" when used correctly to denote void size is analogous to the term "coarse" when describing an aggregate gradation. Finally, large or "entrapped" voids are the natural consequence of batching, mixing, and placing the concrete. Large or irregularly shaped voids are not altogether without benefit to frost resistance, since a protected paste shell exists around their periphery. Such large voids are relatively inefficient in regard to proving frost resistance, however, since their protected paste volume as a fraction of the void volume itself is many times less than for the smaller, more efficient voids. The advantage of the more efficient, smaller voids was stated by Powers [16], "For a given degree of protection, the smaller the air-filled cavities the smaller the total volume of air required."

Quantitative Description of Air-Void System in Hardened Concrete—The Air-Void System Parameters

Measurements of Air Content

The air content of hardened concrete is most commonly expressed as a percentage of the combined volume of all constituents of the concrete including the total volume of air. Klieger [9,47] found that it can be more informative to express the air volume as a percentage of the mortar volume (that is, mortar volume = concrete volume minus the volume of coarse aggregate), observing that frost resistance consistently resulted from 9 ± 1 % air content in the mortar. Given that air voids contribute to the frost-resistance of the hardened cement paste only, it can be useful to express the air content as a percentage of the paste (volume of cementitious materials, water, and air). When calculating the spacing factor of the air-void system (as will be discussed), the air content must be expressed as a fraction of the "air-free" paste, by dividing the total air volume by the volume of cement and water only.

Measures of Air-Void Size and Size Distribution

As pointed out earlier, the air-void grading can range from 10 μm to more than 20 mm–a spread of more than three orders of magnitude. While Willis and Lord [48,49] developed mathematical techniques for estimating the complete grading curve for air voids in concrete, characterization of void size is currently done much more simply and approximately. The size of the air voids is typically defined by a statistical parameter known as the "specific surface," based on the ratio of total air void surface area to total air volume.

The Specific Surface

The specific surface (α) of the air-void system is analogous in some ways to the "fineness" of cement, which is expressed as the estimated total cement surface area per unit mass of the cement (300 m^2 of cement surface per kilogram of cement, for example), ASTM Test Method for Fineness of Portland Cement by the Turbidimeter (C 115) and ASTM Test Method for Fineness of Portland Cement by Air Permeability Apparatus (C 204). Higher fineness values indicate finer, smaller-grained cement since smaller particles have a greater surface area per unit mass. This same approach is used to describe the average fineness of the air voids, where the specific surface is defined as the cumulative surface area of the voids divided by their cumulative volume (rather than cumulative mass). The specific surface is expressed as surface area per unit volume resulting in units of mm^2/mm^3 or 1/mm ($in.^2/in.^3$) or (1/in.); higher values correspond to a finer air-void system. It is observed, for example, that the air-void system typically produced in concrete incorporating an air-entraining admixture will result in a specific surface of 25–45 mm^2/mm^3 (or about 600–1100 $in.^2/in.^3$) [26,34] (see also ASTM C 457).

Limitations of the Specific Surface Alone as an Index to Void Size

The specific surface of air voids and the fineness of cement are two examples of the industry's need to characterize the behavior of multi-size systems with a single, numerical size-index. The "fineness modulus" of sand (see ASTM C 125) is another example. In each of these cases where a single number is used to represent an entire grading, critical information is lost concerning the range and distribution of sizes included. While single-number indices may imply an "average" fineness of sorts, they provide no information about the actual number of particles or air voids with a fineness near the average value. Such indices do not uniquely define the size distribution, since multiple, broadly different size distributions could be described by the same index value.

Powers recognized this limitation of specific surface, citing its utility only for air-void distributions without "extremely coarse voids" [26]. When air-void systems vary considerably in the breadth or overall shape of their size distributions, the specific surface loses much of its value as a comparative index, as do any other indices calculated from the specific surface. Among air-void systems with similar distributions of void sizes, however, the specific surface can be a useful indicator of the relative void fineness [15,50].

Air-Void Dispersion and Spacing

As discussed earlier, only those saturated portions of cement paste within the "critical distance" of an air void are assumed to be able to tolerate the pressure generated during freezing. While both the air content (expressed as the volume of air per unit volume of air-free paste) and the air-void size (as characterized by the specific surface) will jointly be used to estimate the number of air voids within a volume of paste, it remains to assess the dispersion or spacing of those voids and so determine whether substantial portions of the paste are sufficiently close to one or more air voids.

Characteristics of Actual Geometric Arrangement

As observed through the microscope, actual air voids in concrete are randomly dispersed in regard to both size and location [33,50,51] (see ASTM C 457). Mather [52] observed an inhomogeneity in bubble size and dispersion such that "Many samples show variations from area to area, and one cannot escape the conclusion that different parts of such concrete would behave differently with respect to resistance to freezing and thawing." This inhomogeneity has been observed in multiple studies [53–56].

Spacing Factor

Mathematically rigorous approaches are available for determining the relative proportion of the hardened cementitious paste within the beneficial zone of influence of one or more air voids [28,37,57–61]. A less accurate but far simpler approach has been adopted by the industry, however, in which one computes a theoretical maximum distance from any point in the paste to the nearest air void. Under the assumption that a majority of the maximum paste-to-void spacings in the paste are less than this computed value, the so-called "spacing factor," generally designated by $\bar{L}$, can serve as an index to the effectiveness of the air-void system in contributing to frost resistance.

The Simplified Model of Air-Void Spacing: Powers's Spacing Factor

Powers developed the concept of the spacing factor as a simplified approach to the complex mathematics of the actual distribution of air-void spacing in concrete [16,25,26]. The relevant equations for determining the spacing factor are given in ASTM C 457. To those not familiar with the derivation of these equations, their apparent complexity may imply a level of mathematical rigor beyond the intent of the developer, as the relationships bypass the complexity of the real air-void system and substitute a very simplified model of reality.

The basis for the utility of the spacing factor is that it takes into account the combined influence of the "total" air content, total air-free paste content, and a characteristic void size on the spatial distribution of the air voids. To compute the spacing factor in accordance with the ASTM equations, one needs to first determine the air content, paste content, and specific surface of the air voids. No measurements are required or performed relative to observed distances between voids or from points in the paste to the nearest air void.

After having obtained the relevant input data, the first simplifying step is to replace the multi-sized voids in the actual system with a system of single-size voids. The void size in the simplified system is chosen to have the same total volume and same total void surface area as in the actual concrete (see Fig. 2). As pointed out by Willis [48] and by Powers [25] the "number" of air voids in the actual versus simplified systems may be significantly different.

Next, the random dispersion of air voids in the actual cement paste is replaced in the model with a geometrically regular pattern of single-size voids arranged in a uniform, three-dimensional grid. As described by Walker [44], "Consider a hypothetical set of air voids, all one size, arranged in paste in a cubic, three-dimensional array. Every void is equidistant from six other voids, and imaginary lines connecting the voids are mutually perpendicular," as shown in Fig. 2. The dimensions of the grid system are determined so that the volume of the air

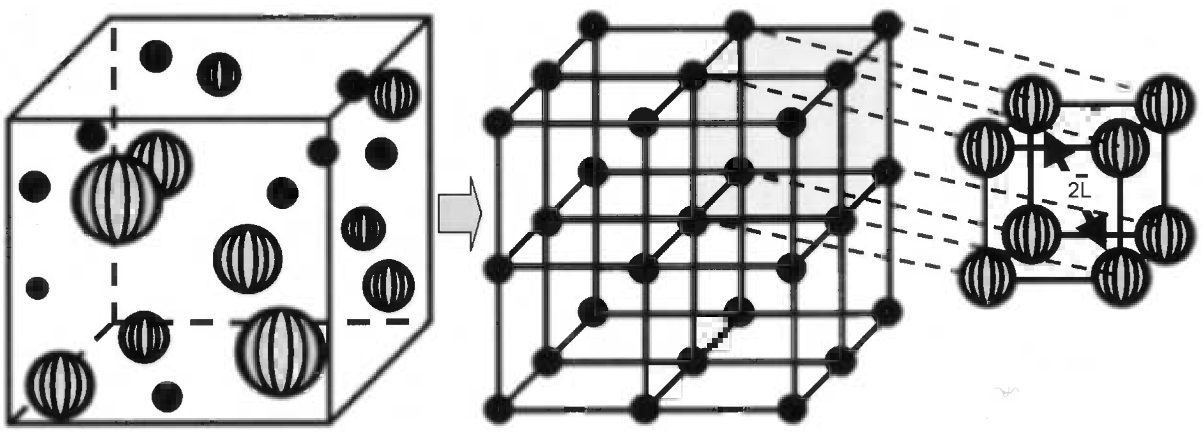

Fig. 2—(After Ref 15.) Air voids arranged in a simple cubic lattice. Calculation of the spacing factor (*L*) is based on the assumptions that all air voids are (1) the same size, and (2) arranged in a simple cubic lattice where each void is equidistant from its nearest neighbor. The ratio of the volume of the unit cube to the volume of the air voids is set to be equal to the ratio of the paste volume to the total air volume in the paste. The spacing factor (*L*) is the distance along the interior diagonal from the center of the unit cube to the periphery of the nearest void, representing the farthest that water or ice would have to travel through paste to get to the nearest air void in the hypothetical air-void system.

voids relative to volume of "grid space" between them is equivalent to the measured volume of air relative to the measured volume of hardened cement paste. The spacing factor is then determined as half of the greatest distance between any two adjacent air voids. These assumptions and the simplified geometry just described are embodied in the standard equation for spacing factor (ASTM C 457-98, Eq 13) [15,21,62].

When it has been determined that air content in the actual system is more than about 23 % of the air-free paste volume (normally occurring in only high air content/low paste concretes), an even simpler model is used to determine the spacing factor (ASTM C 457-98, Eq 12). In this case, one simply assumes that the total paste volume is evenly distributed over the combined surface area of the air voids as a uniformly thick coating. The thickness of this hypothetical paste layer is assumed to be equivalent to the spacing factor [25,26]. This simplified model likewise ignores the complexity of the actual void sizes and random dispersion.

Approximate Nature of the Spacing Factor

Due to the nature of the simplifying assumptions in constructing this model, Powers himself pointed out that the method "does not give actual spacing" [26]. He later wrote [5], "The factor so obtained is obviously not the actual average spacing in a system of graded sizes, nor is it necessarily related to the actual spacing in the same way." ASTM C 457 reports that the spacing factor is "related to the maximum distance in the cement paste from the periphery of an air void." Philleo [58] summarized two primary limitations of the Powers spacing factor, "first, being derived from total void volume and surface area, it [Powers's spacing factor] is strictly applicable only to concretes with similar void size distributions and, second, even if the average spacing is determined accurately, the performance of a particular concrete might be determined by a much larger spacing factor that occurs less than half the time. Thus two concretes with the same calculated spacing factor might not behave the same." Fagerlund [29] reached a similar conclusion, stating that "The traditional way of calculating the air requirement, by use of Powers spacing factor, is an approximation that can only be used for normal situations as regards moisture load, frost load, and air-pore system. It cannot be applied universally."

Methods of Evaluating the Geometry of the Air-Void System in Hardened Concrete

Introduction

As previously discussed the frost resistance of concrete depends on environmental conditions, the frost-resistance of the aggregates, material properties of the hardened cement paste, and the air-void system. Once the concrete has hardened, however, frost resistance is normally estimated on the basis of actual measurements of the air-void system, coupled with assumptions concerning material properties and environmental conditions. The current procedure for evaluating the air-void system of hardened concrete is ASTM C 457.

ASTM C 457 Microscopical Analysis

ASTM C 457 was originally proposed by Brown and Pierson [63] on the basis of earlier developments in geological sciences, with modifications for use on concrete. The mathematical foundation for the technique was laid down by Powers [16,25], Willis [48], and Lord and Willis [49], and key modifications were made by Mielenz et al. [36] based on work by Chayes [64].

A sample of hardened concrete is sawn to expose a plane surface that is then ground to provide an "extremely smooth and plane section" suitable for microscopical inspection (ASTM C 457). The operator causes the prepared sample to move in a systematic pattern under the crosshairs of a stereo microscope, and makes a series of measurements or counts on a random sample comprised of only those air voids that come into a narrow field of view. Statistical estimates of air content, paste content, air-void size distribution, and spatial dispersion of voids are computed on the basis of these measurements.

Sampling

Samples submitted for microscopical analysis are often cores extracted from concrete in service, or may be hardened samples cast specifically for the purpose. ASTM C 457 recommends at least three samples per determination when the tests are being performed for "referee purposes or to determine the compliance of hardened concrete with requirements of specifications for the air-void system." Actual performance of the test, however, requires only one sample, provided the minimum area requirements of ASTM C 457 can be met by the single specimen. Although often only one sample is used, the accuracy of the method is greatly improved when the average results of at least three samples are reported [15,50], as will be discussed. Further, the orientation of cores relative to the exposed concrete surface is important. Sandberg and Collis [56] have pointed out that the most common field sample is a core extracted from a concrete surface, whereby the axis of the core is perpendicular to the exposed concrete surface. Characteristics of the air-void system determined are therefore an average over the depth of the core, strongly influenced by the air-void system remote from that surface. The air-void system of greatest interest when evaluating the frost resistance of the exposed surface, however, will be confined to the top 5 mm or so of the core. Unfortunately, it is difficult to extract a specimen that offers a statistically relevant area of concrete within this critical surface zone.

Specimen Preparation

When observing the specimen through the microscope, the operator must be able to clearly distinguish among the various constituents, with the air voids appearing as sharply defined depressions in the surface. (These depressions are made more visible under the microscope by side-lighting with a harsh, low-angle light.) The less distinct the edges of these depressions the less accurate are the measurements, and sharpness of the void edges depends on the quality of the surface preparation prior to microscopical evaluation.

The current procedure recommends grinding the surface with successively smaller abrasive grits, although alternative methods have been used with success [51,65–67]. The end result should be an "extremely smooth" semi-polished surface, although Brown and Pierson [63] and Mather [68] recommended stopping short of "polishing." This vital issue is discussed by St John et al. [69], who observed that the higher the level of magnification used, the more optically flat must be the specimen surface. They recommend that observation of a "finely ground" surface, such as required by ASTM C 457, "is best restricted to low-power stereo microscopy using magnification not exceeding ×100."

Conduct of the Test

ASTM C 457 describes two alternative methods: (1) the Rosiwal linear traverse technique (ASTM C 457 Procedure A) and (2) the modified point-count method (ASTM C 457, Procedure B).

The nature of the measurements differs somewhat between the two methods as will be described, but the ultimate results are essentially equivalent. Both methods are statistical survey procedures in which inferences about the air-void system of the sample as a whole are made on the basis of measurements of only a fraction of the air voids visible on the specimen surface, which are in turn only a fraction of the total number of air voids present in the sample. For example, a typical 100 mm (4 in.) diameter core taken from concrete in which an air-entraining admixture was used may contain something on the order of 20 million air voids. Of these only about 1000 will be evaluated through the microscope.

The inspected surface is a two-dimensional plane cut and ground from the three-dimensional concrete. Three-dimensional air voids intersected by this plane are represented by depressions of various shape and depth in the plane of the sample. Irregularly shaped air voids in the concrete appear as depressions with an irregular outline, while those voids that were spherical or nearly so are represented by depressions that are clearly circular. The diameters of these circles are only indirectly related to the diameters of the original voids, however, as the plane of the surface could have randomly cut the original void at any point [48,49]. Only a coincidental and unlikely cut at mid-depth of a void would leave a circle with the diameter of the original void. A small circular depression on the specimen surface could therefore be the two-dimensional artifact of a small void cut near its center, or of a much larger void sliced near its periphery.

It can be demonstrated mathematically, however, that small circles are more likely to have originated in small rather than large spheres, which permits the use of statistical theory combined with a sufficiently large number of observations to project an estimate of the original bubble sizes [33,48,49, 59–61,63,65,70]. Using these principles, one could painstakingly measure the diameters of the circles on the plane of the specimen and from these measurements project an estimate of the distribution of the original bubble sizes. As will be discussed, a simpler operation is performed in lieu of measuring the diameters of the depressions. (Note: Since the ASTM C 457 equations are not based on void diameters, mathematical corrections would be required for microscopists who choose a nonstandard procedure based on measuring void diameters in lieu of chords to compute air-void system parameters [61].)

Linear Traverse Method

The linear traverse method requires that a series of "preferably parallel and equally spaced" lines be traced across the surface of the specimen as shown in Fig. 3 [63]. (In practice, this is done by moving the specimen through the microscope in a regular pattern.) These traverse lines randomly intercept a fraction of the depressions on the plane surface, and the number and length of these intercepts are recorded as the test continues. No attempt is made to direct the lines through the diameters of the circles; the intercepts are therefore random "chord lengths" or "chord intercepts." A typical test may consist of total traverse length of 1400–4000 mm per specimen distributed over about 50–1600 cm^2 (7–250 in.^{2}) of sample surface depending on paste content as suggested by aggregate size (see ASTM C 457), and can take 2–6 h of microscopical observation to complete.

The total air content is estimated from the cumulative length of chord intercepts across air voids, divided by the total length of the traverse, multiplied by 100 to convert to percentage measure (Eq 4 of ASTM C 457-98). Paste or aggregate content is similarly estimated by dividing the cumulative chord intercept across the constituents of interest by the total traverse length.

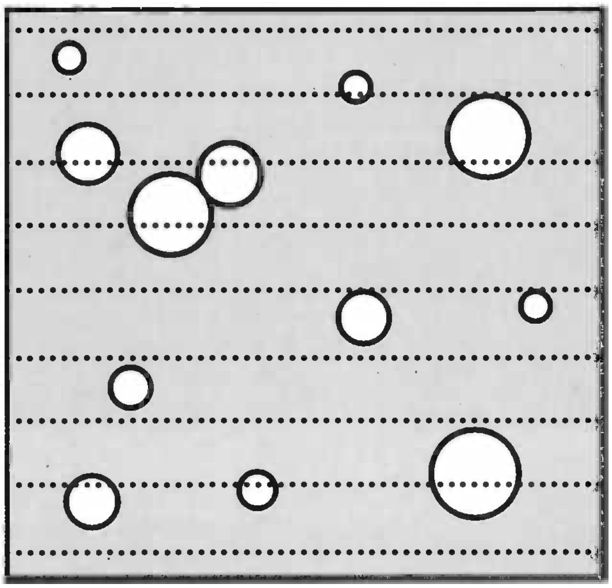

Fig. 3—(After Ref 15.) Schematic diagram of linear traverse procedure. The microscope moves in parallel, closely spaced paths across the prepared concrete surface. Chord intercepts are measured that do not coincide with diameters of the air voids. Note also that the size of the two-dimensional void depressions does not necessarily indicate the spherical diameter of the voids.

As previously discussed, it is possible to estimate the distribution of air-void sizes from the distribution of diameters of the depressions on the plane of the specimen. This can also be done, although with less certainty, from the distribution of the chord intercepts. Neither of these steps are generally taken, however, as void-size distributions are usually neglected in favor of determining the specific surface, α. It has been shown that α can be estimated from the average of the lengths of the individual chord intercepts [48,49,65].

The cumulative length of chord intercepts divided by the number of chords intercepted produces the average chord intercept, $\bar{l}$, (ASTM C 457-98, Eq 6). As demonstrated by Willis [48] the average chord intercept or average chord length is statistically related to the specific surface of the air-void distribution via the expression

$$\alpha = 4/\bar{l} \text{ (ASTM 457-98, Eq 8)}$$

where α is the expected value or "most-probable" estimate of the specific surface of the population of voids with an average chord length of $\bar{l}$. Having obtained values for the air content, paste content, and specific surface, one computes the spacing factor in accordance with Eqs 12 or 13 of ASTM C 457-98.

One additional air-void system parameter in common use is the number of voids encountered per unit length of traverse, or the "void frequency," generally designated by n. The greater this number, the more voids per unit volume, making the void frequency a useful index. Since the number of voids encountered is equivalent to the number of chords intercepted, the void frequency is equal to the number of chords divided by the

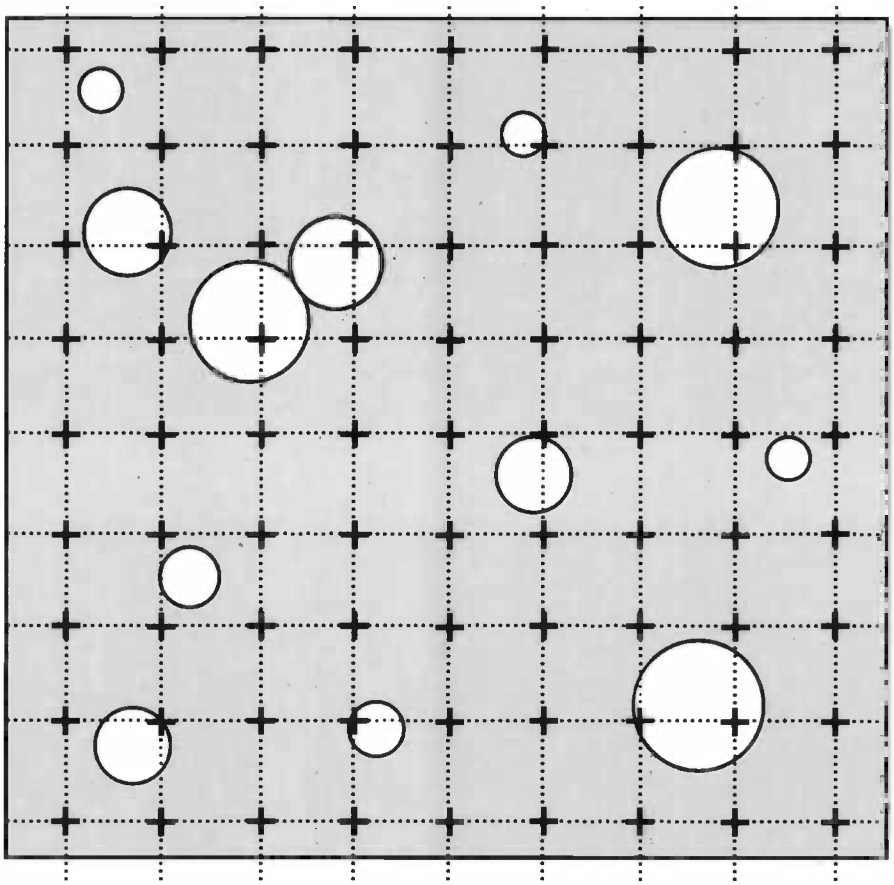

Fig. 4—(After Ref 15.) Schematic diagram of modified point count procedure. The microscope moves in a grid pattern over the prepared concrete surface. Air content is determined by the number of points identified as falling on air voids divided by the total number of points counted. Note that points falling on the edge between air and paste, for example, can complicate the procedure.

total length of traverse. ASTM C 457, Eq 7, relates void frequency and air content to average chord length.

Modified Point-Count Method

The alternative to the linear traverse method is the modified point-count technique, in which a series of traverse lines are superimposed on the sample surface in a manner similar to that just described in Fig. 3. Rather than measuring the individual and cumulative chord lengths, however, regularly spaced stops are made and the constituent directly under the cross hairs is identified at each stop [36]. (Regularly spaced stops along parallel, equally spaced traverse lines form a rectangular grid of points, Fig. 4.) The air content is estimated as the number of points falling on an air void divided by the total number of points in the grid. The volume proportions of all other constituents can be estimated similarly. After completing each traverse line in this point-to-point manner, the operator then reverses the direction of travel over the same line, counting the total number of air voids intersecting the traverse. A typical test may consist of 1000–2400 points counted per specimen, distributed over about 50–1600 cm^2 (7–250 $in.^2$) of sample surface depending on aggregate size (see ASTM C 457), and can take 2–6 h of microscopical observation to complete.

The computed air content multiplied by the total length of traverse provides an estimated total chord length, theoretically equivalent to that determined by the linear traverse method. This "cumulative chord length" (even though no chords were recorded) is then divided by the total number of voids encountered on the traverse to determine an "average chord length," also theoretically equivalent to that determined by linear traverse. Average chord length may also be determined from void frequency. This average chord length is then used as explained earlier to estimate specific surface and subsequently the spacing factor.

Precision and Bias

The discussion of precision and bias that accompanies ASTM C 457 includes the results of two studies on precision [51]. In tests sponsored by the ASTM subcommittee responsible for the test method and using one set of prepared specimens, it was estimated that in 95 % of all cases the expected difference between two independent measurements of air content on a single specimen would be less than or equal to 0.82 % air if the two tests were performed in the same laboratory. The expected difference would be less than or equal to 1.16 % air if tested in two different laboratories. In Sommer's independent tests [51], the expected difference between two measurements of air content on the same specimen would be less than 1.61 % air within the same laboratory, and less than 2.01 % air if performed in different laboratories. Variations in estimated air content would be greater than these reported values if based on analyses of different samples from the same batch.

ASTM C 457-90 reports Sommer's precision data for spacing factor, showing that at the 95 % confidence level two subsequent measurements of spacing factor within the same laboratory could vary by as much as 22.6 % of the value of the spacing factor. If the studies were done in different laboratories, the variation could be as much as 56.9 %.

Langan and Ward [71] conducted a study in which two prepared specimens were sent to various labs for evaluation by various operators within those labs. Results reported for air content ranged between 6.14–9.45 % air for one of the specimens, and between 2.3–2.89 % on the other. For the specimen with the higher air content, the specific surface ranged from 21.9 to 27.6 mm^2/mm^3 (556 to 701 $\text{in.}^2/\text{in.}^3$), and values for spacing factor ranged from 0.079 to 0.170 mm (0.003 to 0.007 in.). For their specimen with the lower air content, the specific surface ranged from 21.9 to 46.5 mm^2/mm^3 (556 to 1180 $\text{in.}^2/\text{in.}^3$), and computed values for spacing factor ranged from 0.135 to 0.284 mm (0.005 to 0.011 in.).

Among the multiple studies of the variability and uncertainty inherent in the ASTM C 457 procedures [36,50,63,71–77], Pleau and Pigeon [77] have presented perhaps the most comprehensive. Among other results they report uncertainty of 27, 24, and 20 % for air content, specific surface, and spacing factor, respectively, for analysis of a single sample. These uncertainties are reduced by about 1/3 when the results are the average of three samples [15]. None of these studies have replicated the typical industrial conditions of random sampling of nonuniform concrete combined with variable surface preparation and local variations in procedures, operators, and equipment. ASTM C 457 therefore advises that "The variability of the test method would be higher in actual practice for specimens sampled and prepared from in-place concrete since additional variation due to sample selection and surface preparation in different laboratories would increase the coefficient of variation."

Sources of Variability and Uncertainty in Test Results

The discussion on precision and bias has demonstrated that the results of the microscopical analysis procedure are not only variable from one sample to another, one laboratory to another, and one operator to another, but being statistical estimates the results are fundamentally uncertain as well. It would therefore be expected that the same operator would obtain somewhat different results for each analysis of the same specimen. Specific sources of this variability and uncertainty are detailed subsequently.

Inherent Statistical Uncertainty

The linear traverse and modified point-count procedures are based on random statistical sampling of a small fraction of the air voids within a small fraction of the concrete so that test results are statistical estimates based on a limited number of observations. (If 1000 voids were observed in one core extracted from one cubic yard of concrete, the voids observed would represent only 0.01 % of the voids in the 1 CY sample.) The uncertainty in these statistical estimates depends on the inherent variability of the concrete, the area of concrete sampled, the area of cement paste available, the number of voids measured, and on the breadth of the distribution of void sizes [50,76,77].

Multiple studies have been conducted on these sources of uncertainty, independent from the variability caused by operational factors [36,49–51,63,71,76,77]. While these references should be consulted for details, it is observed that the uncertainty in air content is primarily a function of the length of the traverse or the number of points counted [36,63,71,76]. The longer the traverse or the greater number of points the more accurate will be the estimate of air content.

To increase the probability of having traversed sufficient concrete surface to have a representative estimate of air and paste content, ASTM C 457 requires a minimum length of traverse for Procedure A, and a minimum number of points for Procedure B. Both these minimum values increase with increasing nominal or observed maximum aggregate size, recognizing that mixes with larger aggregates normally have a reduced paste content [36,63]. Note that whether a sufficiently large number of voids has been intercepted to permit a reasonable estimate of void size depends not only on the traverse length but on the void frequency. For concrete with a low air content and a correspondingly small number of air voids, fewer total voids will have been intercepted even when the ASTM C 457 minimum traverse length or number of points has been completed. This condition tends to increase the uncertainty in the estimates of specific surface and spacing factor. It is of interest to note that a microscopical analysis of hardened concrete is most often called-for when a low air content is suspected; yet the lower the air content the less certain are the results of the analysis. Snyder et al. [76] have shown that when using the linear traverse method this problem may be minimized by running the traverse beyond the ASTM minimum traverse length as necessary until about 1000 voids have been intercepted.

Fundamental statistical uncertainty in estimating the specific surface depends on both the uncertainty in the air content and the uncertainty in establishing the average chord length or void frequency, while the uncertainty in estimating the spacing factor depends on the combined uncertainties in air content, paste content, and average chord length [71,76]. The fundamental uncertainty in computing percent composition on the basis of point-count is discussed by St John et al. and Van der Plas and Tobi [69,78].

Procedural Sources of Uncertainty and Variability

Sampling

If the concrete placement being evaluated were homogeneous throughout, the accuracy of the microscopical analysis would still depend on the number of samples examined. Since the results of the test are statistical estimates of the true values, the error inherent in the reported values "decreases exponentially" with the number of samples [50]. For example, at the 95 % confidence level, the error in estimating the specific surface can drop from 25 % to about 15 % by examining three rather than one specimen, assuming that the concrete is homogeneous. In actual structures, the characteristics of the air-void system vary with location due to variations in batching, mixing, placement, consolidation, and finishing [53,79–81], making the issue of sampling substantially more influential and complex.

No guidance is provided by ASTM C 457 for applying the results from a single sample to the balance of the volume of concrete in question. In this context, however, Simon et al. [53] have shown broad variations in air content, specific surface, and spacing factor among multiple samples taken from within 125–250 mm (5–10 in.) of one another within the same concrete slabs. Such variations can be intensified by the localized effects of consolidation [53,80].

Surface Preparation

Faulty or incomplete specimen preparation has been cited as a source of error by many researchers [33–36,50,51,63,65,82]. Roberts and Gaynor [82] have reported that the effects of specimen preparation alone can skew the results of the ASTM C 457 test by as much as 3 % on air content. Sommer reports similar errors stemming from surface preparation [51].

Level of Magnification of the Microscope

At higher levels of magnification the operator can see and measure smaller voids. While such small voids have little influence on total air content, they have significant impact on specific surface, and therefore on spacing factor [83]. While ASTM C 457 requires a minimum magnification of ×50, Bruere [84] reported that in comparison to tests performed at ×120, "Low magnifications (×40 and ×60) gave erroneous results since very small bubbles could not be seen clearly." Sommer reported that spacing factor was always decreased by magnification of more than ×50, and a magnification of ×100 instead of ×50 can be assumed to reduce an $\bar{L}$ of 0.25 mm (0.010 in.) by at least 10 % [51]. Langan and Ward reported the identical effect [71]. In light of such observations, Pleau et al. [50] suggested a minimum magnification of ×100 and a maximum of ×125. Recall also the earlier statements that magnifications higher than about ×100 require a more sophisticated surface preparation than is normally performed for ASTM C 457 samples [69].

Operator Subjectivity

The most sensitive and sophisticated piece of equipment required in the microscopical analysis of air-void systems in concrete is a human brain, with human eyes as input devices and the human frame as a support system. Each of these is severely taxed by the tedious and time-consuming process described. Despite the mechanically rote aspects of the test (which can be streamlined with modern equipment), it is the human operator who makes the decisions about where to stop and start the individual measurements of chord length, and whether the constituent under the crosshairs is paste, coarse aggregate, fine aggregate, or an air void. In many cases these decisions are not easy, such as when the crosshairs or line of traverse is nearly tangent to a void, when poor surface preparation makes boundaries unclear, or when the presence of aggregates or pozzolans mimic the appearance of an air void. Detailed discussions are found in Ref 50 and in the text and notes in ASTM C 457.

Operator bias can be particularly important in the modified point-count procedure when the crosshairs fall directly on the edge of an air void. Counting this as an "air" or "non-air" point is a subjective choice that can significantly affect the test results. There have been multiple suggestions for keeping this error from accumulating [50]. It can be meaningful to simply keep track of the questionable points and report their number along with the number of clearly identifiable points.

Given the subjective nature of these decisions and the need for informed judgment, it is clear that operators must be well-trained and well-experienced. In comparing test results obtained with experienced operators, Rodway [85] reported "erratic" results from the inexperienced operators using modified point-count procedure, interpreting this as a confirmation of Langan and Ward's previous findings relative to operator variability [71]. Mielenz et al. [36] reported that "the results of the linear traverse are adequately reproducible provided the operator is trained properly." In one test series, Pleau et al. [50] documented operator subjectivity as the cause of variability in computed spacing factor, with an inexperienced operator reporting about 0.145 mm (0.006 in.) and three experienced operators averaging about 0.210 mm (0.008 in.) on the same specimen. While emphasizing the "paramount importance" of training new operators, Pleau et al. conclude that "an intrinsic error due to the operator's subjectivity will always remain."

Arbitrary Deletion of Large Voids

ASTM C 457 states that "no provision is made for distinguishing among entrapped air voids, entrained air voids, and water voids. Any such distinction is arbitrary, because the various types of voids intergrade in size, shape, and other characteristics" (ASTM C 457-98, paragraph 5.3). Some testing agencies nevertheless distinguish at least between the coarse, so-called "entrapped" air voids and the finer, so-called "entrained" air voids [44,51,86,87]. Other agencies have determined such practices to produce misleading results [72]. One reason for discounting the larger voids is to avoid their possible impact on skewing the specific surface as discussed earlier [5,15]. To delete large voids, however, is to artificially modify the recorded air-void size distribution and calculated specific surface, and to delete the contribution made by the large voids to air content and protected paste volume. The effect of discounting the larger chords is consistently to decrease the reported value of air content and to increase the reported value of specific surface. At a minimum, if coarser voids are arbitrarily deleted from the air void analysis, such that A in the ratio p/A reflects only the finer voids, the paste volume p must likewise exclude the volume of the coarse voids as well. A common error is to ignore the coarse voids altogether, consign their volume to the paste, and in the process not only discount their small contribution to frost resistance but also ignoring the fact that their volume does not require the protection of the finer voids included in the analysis. Thus for calculation purposes ignored air voids should be counted as if they were aggregates, not as paste.

Calculations

Several potential errors in the air-void system calculations are worth noting. First, the equation for spacing factor requires a value for the paste content, and the accuracy of the computed value will depend, in part, on the accuracy of the paste content (see Refs 69 and 78 for paste content errors using point-count). In some cases the computed value for the spacing factor is either so great or so small that appropriate conclusions may be drawn without much concern for the accuracy of the estimated paste content. In less obvious cases, an accurate estimate of paste content is essential, and may be determined from either the linear traverse or the point-count technique. (However, Pleau et al. [50] have observed that microscopical examination always underestimates paste content.) Some laboratories use the paste content determined from the mix design or reported batch weights or, in some cases, will merely assume a value for paste content.

A closely related source of error is the common mistake of using a value for paste content that includes the volume of the air itself. The value required in computing spacing factor is the fractional volume of "air-free" paste.

Image Analysis Techniques

There has been a great deal of research and development into automated and semi-automated image analysis techniques for air void systems in hardened concrete, towards improving accuracy and precision and reducing the time and effort required to perform the microscopical analysis. Image-based methods preceded the current techniques [65,88] and have been subsequently attempted to various levels of effectiveness [15,58,86,89–99]. While multiple semi-automated systems are presently marketed, only those in which a human operator discerns the air voids in accordance with ASTM C 457 meet the requirements of that method. As more image analysis systems are coming into the marketplace, it is only a matter of time until one or more of these become an accepted standard test method, interchangeable with, or perhaps as a replacement for, the human operator-based C 457 method. While any given analysis system may not be standardized, and thus might not be a valid means of determining compliance with some specifications, such systems can nevertheless provide important information for evaluating mixtures, or the effects of admixtures or construction operations on air-entrained concrete. Any method than provides fast, accurate, and reliable information can prevent serious problems in the field, and may in fact reduce the need for the more time-consuming standard C 457 analysis.

One consequence of the development of automated techniques is a more thorough appreciation of the difficulties faced by the skilled C 457 operator. The current test requires that the operator make a large number of subtle distinctions and judgment calls based on color, texture, shadow, and the appearance of the feature in question compared to its background (about 1000 such judgments per analysis). The challenge of programming a computer to make the same judgments and distinctions is a measure of the power of the human eye coupled with the human brain, and an indication of the value of a skilled, experienced operator. A related challenge is how to evaluate any given image analysis system. Ostensibly this is a simple matter of comparing automated results to standard C 457 results. This is complicated, however, by the variability of the C 457 test and the range of results that can be obtained over multiple runs on the same specimen by the same operator, and the broader range of results from the same specimen by different operators.

Depending on the direction taken by the developing technology and the industry response, it is likely that the critical issues of sampling and surface preparation will remain. It may be that more rapid turn-around on test results or a reduced cost per test will permit a larger number of samples to be examined or each sample examined more thoroughly, with a resulting decrease in overall uncertainty and variability.

Comparing Air-Void System Parameters in Fresh and Hardened Concrete

Total air content is the only air-void system parameter that can be directly compared between the fresh and hardened concrete using conventional methods. This is because of the fundamental inability of any of the standard test methods including ASTM Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231), ASTM Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173), and ASTM C 138 (density) to provide information about void size or dispersion in fresh concrete. These standard test methods indicate the total air content only, regardless of the size or distribution of the air voids. (The Air Void Analyzer (AVA) is a relatively new technique for estimating air bubble size in fresh concrete, but it is not yet a standard test method [100]. Discussion of this useful technology is beyond the scope of this chapter.)

When differences exist between the mixing, handling, placing, or consolidation of the concrete sampled for the determination of air content in the fresh state, and the concrete sampled for later microscopic determination of air content, differences in the two test results are expected. This is because the air voids can change in size, number, shape, and volume within the fresh concrete, and can be removed from the system entirely under the influence of mixing, vibration, pressure, and temperature [1,79,101–105]. In fact, the reason for vibrating the concrete is to remove air from the concrete, and not all of the air removable by the vibrator became trapped after mixing and discharge in the process of placing the concrete in the forms [36,53,80,106,107].

The procedure for the ASTM C 231 pressure method therefore alerts the user that "The air content of hardened concrete may be either higher or lower than that determined by this method." (Examples of the variation between fresh and hardened air contents are shown in Ref 108.) The ASTM C 231 procedure goes on to explain that the magnitude and direction of the difference between fresh and hardened air contents will depend upon "the methods and amount of consolidation effort applied to the concrete from which the hardened concrete specimen is taken; uniformity and stability of the air bubbles in the fresh and hardened concrete; accuracy of the microscopic examination, if used; time of comparison; environmental exposure; stage in the delivery, placement and consolidation processes at which the air content of the unhardened concrete is determined, i.e., before or after the concrete goes through a pump and other factors." In 1968 T. C. Powers reported, "the air content of fresh concrete may not be the same as after hardening and that losses of air from air-entrained concrete during handling have desirable consequences" [5]. While the latter comment may be an over-generalization in some cases, it certainly moderates the alarm that can accompany the observation that hardened air content is less than fresh air content.

Interpretation of Test Results

Interpreting the Results of Tests on Hardened Concrete

General Comments

A more detailed discussion of criteria for obtaining frost resistance is included in the chapter in this publication by Nmai. It is useful to keep the following principles in mind, however, when interpreting the air-void system parameters obtained using the procedures already described:

- The test results themselves are variable and subject to uncertainty, complicating any inference concerning frost resistance.
- The criteria against which the results are to be compared can be equally uncertain. Natesaiyer and Hover [28,37] have shown that while present criteria (to be discussed) can identify concrete that is almost certainly frost-resistant and concrete that is almost certainly nonfrost-resistant, there exists a broad, marginal zone or "gray area" in which frost resistance is difficult to judge.
- Some of the difficulty in interpreting frost resistance on the basis of air-void system parameters that are neither "clearly acceptable" nor "clearly unacceptable" is due to the fact that frost resistance depends also on the mix proportions and material properties of the concrete, and on environmental exposure.
- The incorporation of a beneficial air-void system in concrete does not eliminate the pressure caused by freezing,

but merely reduces it to tolerable levels. In a practical sense, risk of frost damage is minimized, not eliminated entirely, by the incorporation of air [85].

- The frost resistance of concrete in-service depends on its properties in-place. Measures of air content in the fresh concrete, or air-void system parameters determined on concrete samples not handled, placed, consolidated, or finished in a representative manner may lead to incorrect conclusions about frost resistance.

Air Content

Klieger recognized that the necessary air content was dependent on mix proportions [9,47]. In two independent studies, he demonstrated that when the coarse aggregate volume (which is not protected by air voids in concrete) is ignored, the optimum air content for frost resistance was consistently 9 % ± 1 % of the volume of the mortar. Klieger's observations are reflected in the mix design recommendations of ACI 211.1 [3], in which the suggested air content increases as the nominal maximum coarse aggregate size decreases. This is not because smaller coarse aggregate particles are less frost-resistant (the reverse is generally true) [109]. It is because the ACI 211 mix design method will correctly result in higher mortar content in mixes using smaller coarse aggregates. This conclusion was independently reached by Siggelokow [110]. As Saucier et al. pointed out, however, while a particular value of air content is necessary to provide frost resistance for a given concrete, obtaining such air content is not by itself sufficient because of the need to obtain an appropriately sized and distributed air-void system [105].

Specific Surface

While frost-resistant concrete is generally characterized by values of specific surface greater than 25 mm^2/mm^3 (or about 600 $in.^2/in.^3$), Neville [111] reports frost resistance for certain concrete at values as low as about 16 mm^2/mm^3 (400 $in.^2/in.^3$). Specific surface is merely an indicator of bubble size, however, providing no information about volume or dispersion. One could have an air-void system composed of acceptably small bubbles as indicted by specific surface, but there may be too few of these bubbles or they may be nonuniformly spaced so as to leave large gaps of unprotected paste.

Void Frequency

For frost-resistant concrete, the number of voids encountered per unit length of traverse is generally expected to be "one to one and a half times the numerical value of air content" [112,113]. Thus, if the air content is 5 %, one would expect 5–7.5 voids per inch. This rule of thumb arises from the algebraic relationships among the air content, void frequency, and specific surface; when void frequency is about equal to the air content, specific surface is about 16 mm^2/mm^3 (400 $in.^2/in.^3$). When void frequency is 1.5 times air content, the specific surface is about 25 mm^2/mm^3 (600 $in.^2/in.^3$). Void frequency and specific surface are therefore not independent variables and only one of the two can be specified.

Spacing Factor

Various agencies and organizations, such as ACI Committee 212 on concrete admixtures [114] and ACI Committee 201 on durability [115] have recommended a spacing factor of 0.008 in. (0.200 mm) as being indicative of frost-resistant concrete [112,116]. The Canadian Standards Association [117] specifies that "The concrete will be considered to have a satisfactory air-void system when the average of all tests shows a spacing factor not exceeding 230 μm (0.0091 in.), with no single test greater than 260 μm (0.0102 in.), and air content greater than or equal to 3.0 % in the hardened concrete. For concrete with a water-to-cementing materials ratio of 0:36 or less, the average spacing factor shall not exceed 250 μm (0.0098 in.), with no single value greater than 300 μm (0.118 in.)." It is further noted that "Considering that the ASTM C 457 test is subject to large variations, it is recommended that the target spacing factor be less than 170 μm to have reasonable assurance that the 230 μm requirements of this clause will be met." (Conversions to inches are not part of CSA Standard, and are shown within quotations for convenience.)

While it is likely that concrete with a spacing factor in such ranges will be frost resistant, it is not equally likely that concrete with a larger spacing factor will necessarily be nonfrost resistant. Pigeon and Pleau [15] and Pigeon and Gagné [118] have demonstrated that when deicer salt scaling is not an issue, frost resistance can be obtained in many cases at spacing factors in the area of 0.500 mm (0.020 in.) when a severe but natural freezing rate is used. Spacing factor values on the order of 0.200 mm (0.008 in.) are required at faster freezing rates, longer sub-freezing periods, or when resistance to deicer salt scaling is required. Pinto and Hover [119] also observed more stringent requirements for scaling resistance, while Fagerlund [29] reports that when it comes to scaling resistance "it is not possible to find any well-defined value of L_{CR}." Fagerlund went on to write that "The values of the critical spacing factor are by no means indisputable," and that in comparison to criteria such as 0.008 in. (0.200 mm) "quite other values have been found in other experiments." Philleo [120] reported that "Data suggest that at water-cement ratio below 0.50 by mass the required spacing factor is a function of water-cement ratio. Thus, the required spacing factor becomes larger as the water-cement ratio decreases." Interpretation of frost resistance on the basis of spacing factor is not without difficulty, therefore, in spite of the fact that of the indices available, spacing factor is the most commonly used.

Multiple studies correlating freeze-thaw durability with computed spacing factor have shown a scattered but general trend of increased durability with decreasing spacing factor [28,37,72,113,118,121,122]. Ivey and Torrans [121] concluded that for conventional concretes "the transition between durable and nondurable concrete seems to be somewhere between an *L* of 0.008 and 0.010 in. [0.20 to 0.25 mm]." This general assessment appears to remain valid, and coincides with Powers's original proposal that "for typical concretes and environmental conditions spacing factors not exceeding 0.250 mm (0.010 in.) or thereabouts" were generally indicative of frost-resistant concrete [25]. In this proposal, Powers also recognized that the spacing factor required for frost resistance depended on material and environmental factors.

Part II: Density[3] (Unit Weight)

Introduction

The term "density" as used here refers to the weight per unit volume of hardened concrete. The term density when used to

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[3] As noted in the 2001a edition of ASTM C 138, "Unit Weight was the previous terminology used to describe the property determined by this test method, which is mass per unit volume."

describe the weight of fresh concrete per unit volume, as determined by ASTM C 138, is discussed in the chapter in this publication by Roberts. While the densities of fresh and subsequently hardened samples of a particular mix are expected to be related, the values are not expected to be identical. The relationship between the fresh and hardened densities for a particular concrete mixture will depend on the mix proportions and characteristics of the aggregate, the degree of consolidation, sampling, volume changes, age, and curing.

Significance of Density as a Characteristic of Concrete

Density

In some cases, the density of hardened concrete, per se, is critical to the performance of the structure or facility. Examples include setting a maximum density requirement when lightweight aggregate concrete is used to limit structure self-weight, or setting a minimum density requirement when self weight is to be maximized for structural stability or for nuclear shielding. As described by ASTM Test Method for Density of Structural Lightweight Concrete (C 567), "The measured or calculated equilibrium density of structural lightweight concrete determines whether specified density requirements have been met."

Uniformity or Consistency of Materials, Construction Operations, and Testing

Consistent densities normally indicate consistency in all phases of concreting operations. This is because the density of hardened concrete is a function of the densities of the initial ingredients, mix proportions, initial and final water content, air content, degree of consolidation, degree of hydration, volume changes, and subsequent gain or loss of water, among other factors. Dependence on these factors makes density an effective indicator of the uniformity of raw materials, mixing, batching, placing, sampling, and testing. A significant change in density signals a change somewhere in the process.

For example, if the density is seen to vary among samples of hardened concrete that had been cast at the point of concrete delivery, variability in the constituent materials or proportions could be indicated. On the other hand, in this situation variable density could mean nonuniform batching or mixing, or nonuniform casting of test specimens. Routine weighing of standard compressive strength specimens before testing is recommended to quickly approximate density and to indicate sample uniformity.

Alternatively, density tests performed on samples of hardened concrete extracted from the structure can be useful for indicating segregation, nonuniform consolidation, or other problems. Because this useful information is so readily obtained, routine density testing for all cores extracted in the field is often useful, regardless of the primary purpose for obtaining those cores.

Voids Content

In dried, hardened concrete all of the internal voids are air-filled, including capillary pores in the hardened cement paste, voids in the aggregate particles, bleed water channels, water gain voids, microcracks, and air voids intentionally or unintentionally incorporated into the mixture. The volume of some, but not all, of these voids can be determined by the weight of absorbed water when a dried specimen is immersed for a period of time. Those voids that communicate directly with the exterior surface of the sample or are connected to the surface via capillary channels or via cracks formed by drying or shrinkage, absorb water upon immersion. These are called the "permeable pores," and represent the pore space measured by drying followed by immersion. Other voids, which include a portion of the capillary void system, some of the aggregate pores, and a fraction of the system of air voids, are termed "impermeable pores." These spaces do not fill with water upon immersion and cannot be measured by these techniques. Because one cannot discriminate among the various types of voids present when determining voids content by absorption methods, the final result is termed "total permeable voids." For a given set of raw materials of fixed densities, the lower the density of the mixture the greater will be the voids content. Helms [123] has documented an empirical relationship between oven-dry density and voids content.

The total volume of permeable voids of a sample is related to porosity, a basic characteristic of concrete that influences many of its properties. Numerous studies, beginning with those of Feret and continuing more recently to Popovics, have explored these relationships [124–129]. In fact, Feret [124] established a clear relationship between the strength of mortars and the voids/cement ratio (volume of voids in the sample divided by the volume of the cement) well before the more currently recognized relationship was established between strength and water/cement ratio. Data published by the U.S. Bureau of Reclamation confirmed that the relationship between voids and strength applied equally well to a wide variety of concrete mixes [130]. Helms [123] has reported a similar relationship even when lightweight aggregate concretes were tested.

Permeability

While the porosity of concrete can be related to unit weight and voids content, permeability depends not only on the total pore or flow channel volume, but also on the connectivity, tortuosity, and hydraulic characteristics of the channels. Nevertheless, correlations have been demonstrated between certain types of permeability tests and density of hardened concrete [131–133]. Such relationships, even if only empirical in nature, suggest a linkage between the density and durability, since the durability of concrete can be related to the degree to which water, water vapor, oxygen, and carbon dioxide can permeate the concrete.

Degree of Consolidation

Analogous to routine measurements in soil mechanics and bituminous materials, one can estimate the degree of consolidation from density measurements of hardened concrete. Degree of consolidation is generally expressed as the ratio between the observed density and some value taken for the "maximum" or "optimum" density. Olsen [134] defined degree of consolidation as the density of the hardened concrete divided by the density of the fresh concrete. In Whiting's work [131], the maximum density was that obtained from hardened samples that had been consolidated on a vibrating table. (It is interesting to note that within Whiting's laboratory testing program the standard deviation on measured density was approximately 40 kg/m^3 (2.5 lb/ft^3), which may approximate a minimum uncertainty for density tests.) Whiting went on to correlate the degree of consolidation (or "relative unit weight") to compressive strength, bond strength, and rapid chloride permeability.[4]

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[4] Note that density measurements can be misleading when evaluating the effectiveness of consolidation by means of an immersion vibrator due to the tendency to displace aggregates leaving a high mortar content at the point of vibrator insertion.

Bisaillon and Malhotra [133] also demonstrated the benefits of improved consolidation in reducing the hydraulic permeability of concrete.

Thermal, Acoustic, and Nuclear Shielding Properties

Density is a key parameter in defining the ease of transmission of energy through the concrete, and when such properties are of interest it may be more appropriate to measure density than compressive strength. Valore [135] and Brewer [136] separately demonstrated the relationship between density of hardened concrete and its ability to transmit heat. Both the modulus of elasticity of concrete and the ultrasonic pulse velocity can be shown to be dependent on density. For that reason, it is often necessary to determine the density of concrete in order to interpret the results of dynamic modulus or the pulse velocity tests.

Although the topic of heavyweight, nuclear-shielding concrete is beyond the scope of this chapter, the key issue in attenuating the transmission of atomic particles is to put as many atomic nuclei in the path of the radiation as possible. This means that, in general, the denser the mass, the better is its shielding ability. The density test is a simple means of determining whether the required density has been achieved.

Inferring Batch Weights and Composition

Just as the density test can lead to estimates of the voids content, some information about the composition of the balance of the sample is theoretically possible as well. The effects of composition on density will be discussed.

Typical Values

Figure 5 [123,137,138] displays the approximate range of densities and air contents represented by aggregates, concrete, and

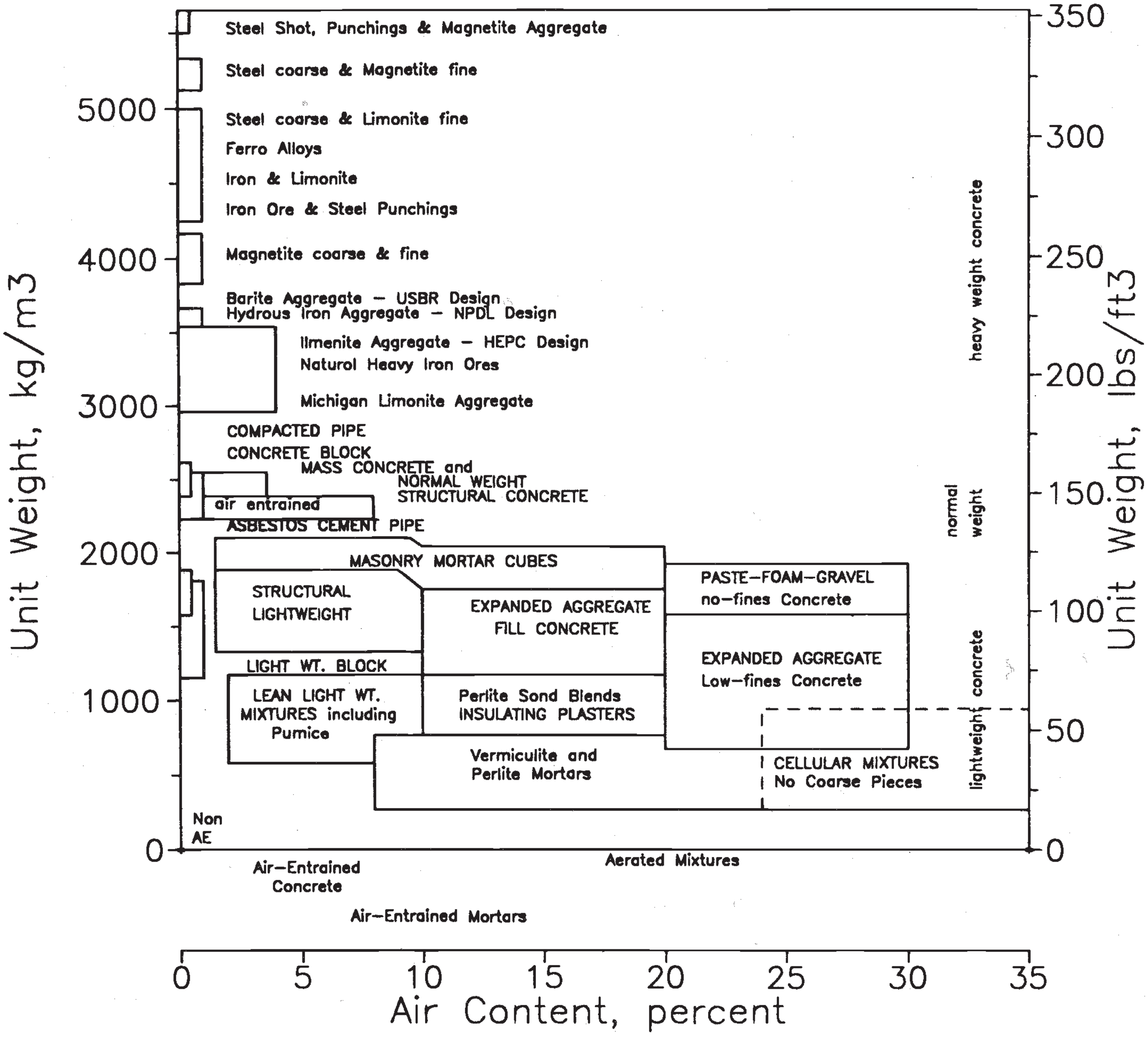

Fig. 5—Density block diagram (after Helms [123]).

cementitious materials. The range is bounded at the high-density end with steel shot, steel punchings, and magnetite aggregate used for radiation shielding concretes and counterweights, with densities above 5000 kg/m^3 (above 310 lb/ft^3). The low-density end of the range is occupied by cellular concretes with densities less than 1000 kg/m^3 (60 lb/ft^3) and with air contents above 25 %.

Litvin and Fiorato [139] have independently prepared the "Lightweight Aggregate Spectrum" that also graphically depicts concretes with compressive strengths ranging from 0.7 to 40 MPa (100 to 6000 psi) with concrete densities from 240 to 2000 kg/m^3 (15 to 120 lb/ft^3).

Methods of Determining the Density of Hardened Concrete

General

The measurement of density of hardened concrete is based on procedures that are simple and direct, using samples such as molded specimens, drilled cores, or portions taken from hardened structures and trimmed to cylinders or prisms of sufficient size to be representative. Hardened samples are dried or otherwise conditioned, and weight and volume measurements are taken. The specimens are then immersed in water so that the permeable pores are filled. Weight measurements are repeated after immersion and from these data one determines the volume of the permeable pores and weight/volume relationships for the dry and saturated specimens. It is reasonable to require density by displacement measurement to be reported to the nearest 1.6 kg/m^3 (0.1 lb/ft^3) and within an accuracy of 0.1 %. Reference is made to the detailed provisions of ASTM C 567 and ASTM Test Method for Density, Absorption, and Voids in Hardened Concrete (C 642). (Exceptions to the general simplicity of density measurements are the nuclear methods described in ASTM Test Method for Density of Unhardened and Hardened Concrete in Place by Nuclear Methods (C 1040).)

More advanced, yet nonstandard techniques for determining the density of hardened concrete include mercury pycnometry [140], although such methods are more appropriate for very small samples of hardened concrete, mortar, or paste (that is, generally less than 20 cm^3).

Procedural Factors Influencing the Results

In principle, the density of hardened concrete is obtained just as the density, specific gravity, absorption, etc. are obtained for aggregates, and the same concerns apply to proper moisture conditioning. This is made more difficult, however, when dealing with concrete samples that are many times larger than aggregate particles, and therefore come to moisture equilibrium more slowly. Other concretes are more absorptive than normal-weight aggregates and therefore can take on water at a faster rate.

The moisture condition to be used for weight determination depends on the purpose of the measurement and the in-service condition of the concrete in question. For example, when specimens are obtained by extracting cores from a structure, care is required to retain the "as-is" moisture condition. Since the density of lightweight aggregate concrete in-service is a critical issue, ASTM C 567 makes special note of establishing in-service and "equilibrium" moisture conditions in the sample.

In both ASTM C 567 and C 642, the volume of the sample is determined by the "displacement method," in which the difference between weighing in air and weighing in water is attributed to the buoyant effect of the water, which in turn is related to the density of the water and the displaced volume. In informal testing, the volume of the specimen is frequently calculated from the physical dimensions of the sample. This latter approach can be useful only when the shape of the specimen is highly regular and the dimensions accurately obtained. Even with standard concrete cylinders, the unevenness of uncapped ends and the tendency to out-of-roundness can introduce significant errors.

The issue of sample size is briefly addressed in ASTM C 642, pointing out the need to have a representative sample. This is more difficult than it may at first appear, particularly given the variations in degree of compaction that exist vertically and horizontally within a given concrete member. Such variations are well documented in work by Simon et al. [53] and by Kagaya et al. [141].

Influence of Composition on Density

Effect of Aggregate Density

Since aggregate can occupy 60–80 % of the volume of most concrete mixes and the specific gravity of normal-weight coarse and fine aggregate is approximately one-third greater than that of the hardened cement paste, the aggregate has considerable influence on density of hardened concrete. The data shown in Table 1 were collected by the U.S. Bureau of Reclamation [142] and show a 6–7 % variation in density for mixes using aggregates of various specific gravities but identical proportions.

TABLE 1—Observed Average Density (adapted from Ref 142)

Maximum Size of Aggregate, mm (in.)	Average Values			Density, kg/m^3 (lb/ft^3) Specific Gravity of Aggregate, SSD				
	Air Content, %	Water, kg/m^3 (lb/yd^3)	Cement, kg/m^3 (lb/yd^3)	2.55	2.60	2.65	2.70	2.75
19 (3/4)	6.0	168 (283)	336 (566)	2200 (137)	2230 (139)	2260 (141)	2290 (143)	2330 (145)
38 (11/2)	4.5	145 (245)	291 (490)	2260 (141)	2290 (143)	2340 (146)	2370 (148)	2405 (150)
76 (3)	3.5	121 (204)	242 (408)	2310 (144)	2357 (147)	2390 (149)	2440 (152)	2470 (154)
152 (6)	3.0	97 (164)	167 (282)	2360 (147)	2389 (149)	2440 (152)	2470 (154)	2520 (157)

TABLE 2—Effect of Paste Content and Water/Cement Ratio on Density

Water/Cement Ratio	Specific Gravity of Paste	Density of Paste, kg/m^3	Density of Paste, lb/ft^3
0.30	1.90	1900	119
0.40	1.68	1680	105
0.50	1.48	1480	92
0.60	1.34	1340	84
0.70	1.22	1220	76

Effect of Paste Content

Cook [140] reported the dry specific gravity of hardened cement pastes at various water/cement ratios after 56 days of continuous wet cure. The results are shown in Table 2, in which it is clear that the density of hardened cement paste decreases as water/cement ratio increases. Since the densities of normal aggregates usually range from about 2560 to 2800 kg/m^3 (160 to 175 lb/ft^3), it is clear that a reduction of paste volume by substitution of aggregate will increase the density. Further data from the Bureau of Reclamation [143] demonstrates this, in which mixes with a 43 % paste content had a density of 2206 kg/m^3, while a similar mix with a 22 % paste content had a density of 2533 kg/m^3 (137 and 155 lb/ft^3, respectively).

Effect of Air Content

Density is reduced with an increase in the relative volume of pore space in the concrete sample, regardless of the origin of the pores. Therefore, all other factors remaining constant, density will decrease with an increase in air volume, regardless of the size, shape, or distribution of the air voids. The density test cannot discern air-void size, nor can it differentiate between those voids constituting the desirable air-void system in the hardened concrete and spaces such as capillary pores, bleed water channels, aggregate voids, etc. For these reasons, one has to exercise judgment in using measurements of the density of hardened concrete to evaluate air content.

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Analyses for Cement and Other Materials in Hardened Concrete

William G. Hime[1]

Preface

THE CHEMICAL ANALYSIS OF CONCRETE FOR PORTland cement content was covered by H. F. Kriege in the first edition of *ASTM STP 169.* Dr. L. John Minnick provided an excellent discussion of methods of analysis for portland cement content in *ASTM 169A.* The present writer extended the discussions in *ASTM 169B* and *ASTM 169C* to include analytical methods for other concrete components, and broadened the title from the "Cement Content" of the first two editions. This chapter in *ASTM 169D* extends analytical developments to the twenty-first century.

Introduction

In the period of time since *ASTM 169C,* there have been few advances in analytical procedures for cement and concrete components other than in instrumentation. Indeed, "wet chemistry," the use of gravimetric and volumetric techniques, is no longer used in many laboratories, nor often taught in college. Nevertheless, wet chemistry is the standard for many procedures used to analyze concrete.

Minnick [1], in *ASTM STP 169A*, and Hime [2,3], in *ASTM STP 169B* and *169C,* discussed the analysis of concrete for portland cement content, and the latter also provided methods for other concrete components in those publications and in Ref 4. Unfortunately there are no accepted procedures for many components in concrete made with blended hydraulic cements or with supplementary cementitious materials. Unless the original components of such concretes are available, estimation of component concentrations is often best done by an experienced petrographer, as discussed by Erlin in Chapter 22. Indeed, a petrographer is often a necessity to direct the chemical analyst in the recognition of potential interfering concrete components. The detection of supplementary cementitious materials (e.g., fly ash and slag) in concrete is also usually a task requiring a petrographer. The situation is even more complicated when components such as silica fume are present. Neither the chemist nor the petrographer will often detect silica fume and thus a cement content analysis by a silica procedure may provide erroneously high results.

The presence of organic components is not generally revealed by optical procedures. At times, the properties of the concrete may suggest such substances: delayed set suggests an organic sugar derivative, while a hydrophobic nature suggests a stearate compound or a polymeric coating. Infrared spectroscopy is a powerful technique for identifying such materials, as noted below.

Methods of analysis of grouts and mortars generally are the same as, or are similar to those used for portland cement concrete, and thus are discussed herein.

Chemical Analysis Procedures for Cement Content

Analyses for portland cement are usually based on two of its features: a calcium oxide content, regardless of manufacturer, of about 63 %, and an acid-soluble silica content of about 21 %. Thus, if the aggregate is non-calcareous, analysis for calcium usually provides an excellent result. If the aggregate is limestone, analyses for acid-soluble silica are generally applicable, as is a procedure using maleic acid dissolution of an uncarbonated fraction of the concrete sample. However, if a siliceous aggregate is alkali reactive, neither the silica nor the maleic acid procedure is applicable.

The three procedures are well presented in ASTM C 1084. Although that designation primarily provides wet chemical procedures, the analyses for calcium or for soluble silica can be done by instrumental techniques such as atomic absorption spectroscopy. For such analyses, several standard samples, as available from NIST and other organizations, should be used to calibrate the instrument.

Analyses for the sulfate content of the concrete are sometimes made to determine cement content. Although portland cement contains sulfate and most, but not all, aggregates do not, such analyses may suffer greatly because present-day portland cements have sulfate contents ranging from about 1.5 % to over 5 %. This wide variance, and the huge multiplication factor to convert to cement content, can produce enormous errors.

The percentage of cement found in the analytical sample can be converted to pounds per cubic yard on two bases. One uses the unit weight determined on a saturated-surface-dry basis, and correlates well with the batched concrete. The other uses a dry unit weight and correlates with the in-place concrete.

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Petrographic Analysis Procedures for Cement Content

Experienced petrographers often estimate cement content based on: point-count or linear traverse measurements of paste content, "measured" or estimated water-cement ratio, and, if fly ash or slag is present, visual estimates of their volume. The accuracy of such estimates depends on the complexity of the concrete system, its age and exposure to carbon dioxide or other aggressive agents, and curing. Further, conversion of volume percentages to weight percentages are necessary, but densities of such components as fly ash may vary greatly. Standards greatly increase reliability, provided that they duplicate components and exposure, and therefore are particularly applicable for early-age quality control of concrete production.

ASTM C 856, Standard Practice for Petrographic Examination of Hardened Concrete, and ASTM C 457, Standard Test Method for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete, are useful adjuncts to the determination of cement content, but provide no specific procedure for such analysis. Chapter 22 of this book discusses petrographic methods of analysis that augment the procedures in C 856.

Procedure Details

Sample Selection

Proper sample selection is of extreme importance in attaining meaningful cement content data, but is often not achieved. Although ASTM C 85 (replaced by ASTM C 1084) required three 10-lb concrete pieces, experience indicates that a smaller sample, carefully chosen, can be sufficient and will present fewer laboratory problems. As indicated in ASTM C 1084, a single core may be satisfactory for the cement content determination. It should have a diameter of at least four times the maximum aggregate size and be taken through the entire vertical depth of a concrete slab. For thicker concrete members, the selection must consider that concrete composition may vary with depth or with structural feature. In such cases, several cores analyzed separately will disclose the degree of uniformity of cement distribution.

A second core or sample, or a larger core, is generally also required to perform a determination of unit weight to allow petrographic examination, or to allow horizontal sampling for components such as chloride, so that concentrations can be determined as a function of depth.

If a concrete cylinder is being analyzed, the sides will be paste rich. To best represent the concrete, the entire cylinder should be pulverized before sampling.

Sample Preparation

If a second core or sample has not been obtained, it is desirable to cut off (longitudinally) a portion of the sample for unit weight determination, petrographic examination, or such further analyses as might be suggested by the problem or the cement content results. The major portion should be crushed to quarter-inch size (or as directed), quartered, and a representative portion pulverized as required. Since very fine particles interfere with many of the analysis procedures, the sample should be pulverized for short intervals, with sieving after each so that most particles just pass the designated sieve.

Significant amounts of iron may be introduced by some pulverization and grinding equipment. A magnet may be passed through the sample to remove the iron before analysis if a petrographer confirms that such a process does not remove aggregate components such as magnetite.

ASTM C 1084 Analysis

Soluble Silica Procedure

ASTM C 1084 requires analyses by a prescribed procedure. In contrast to the earlier ASTM C 85, ice-cold hydrochloric acid is employed to dissolve the cement, thus minimizing dissolution of silicates in aggregates, fly ash, or slag. It is useful to examine the acid-insoluble residue so that the presence of fly ash may be revealed.

Calcium Oxide Procedure

A wet chemical procedure for calcium is lengthy and difficult. Alternatively, any determination method may be employed if the method can be shown to provide accurate analyses of NIST or other Standard Cements. Atomic absorption spectroscopy or X-ray emission spectroscopy is employed by many laboratories to determine calcium rapidly.

Maleic Acid Procedure

Maleic acid determinations of cement content were first suggested by Tabikh [5], with modifications by Clemena [6], Pistilli [7], and Marusin [8]. These vary primarily in the concentration of the maleic acid solution and the length of digestion time. All are designed to minimize solution of both calcareous and siliceous aggregates, but to allow nearly complete dissolution of the portland cement component. For a particular aggregate, one of the versions of the test may be better than the others, but most laboratories do not have the luxury of allowing a test variance for each sample.

It is imperative that the analyst minimize carbonation of the sample because carbonation converts soluble cement hydration products to insoluble calcium carbonate. If highly fractured concrete is the subject of the analysis, either a petrographer should confirm that carbonation is minimal or another procedure should be employed. ASTM C 1084 now includes a maleic acid procedure. If the portland cement incorporates limestone as a processing aid (up to 5 % is allowed by ASTM C 150), the maleic acid procedure will provide a lower cement content than will the calcium and silica procedures.

Interferences

ASTM C 1084 discusses interferences to those procedures.

Cement Type Analysis

At times an investigation requires a determination of the type of cement used in the concrete: for example, concern may have been expressed that a concrete structure has not been made with the specified Type V cement. Dissolution of the cement paste by the maleic acid procedure provides an avenue to a successful approach if the aggregate is insoluble and fly ash is not a component of the concrete. The maleic acid is then destroyed by evaporating with nitric acid, and aluminum and iron determined by chemical or instrumental methods. From these data the tricalcium aluminate (C_3A) content may be determined and compared to ASTM Specification for Portland Cement (C 150) limits.

Similar methods may be used to determine if the cement was "low-alkali," but interference by the aggregate may occur if it contains alkalies (for example, a feldspar), and it will occur if fly ash is present. Of course, no study of the concrete will reveal if the cement satisfied other chemical or any physical requirements.

Calculations and Report

Perhaps the greatest error in the cement content analysis is the assumption that the result represents the cement content as delivered. Two factors can cause serious divergence between determined and delivered cement factor. First, because the calculation is in terms of pounds per cubic yard, poor consolidation of the concrete leads to a lowered cement content. And second, even under good placement practice, cement concentration may vary with height of placement (for example, in columns and walls), or with location in a heavily reinforced structure. To partially account for such occurrences, ASTM C 1084 provides two methods for density determination: oven-dry and saturated surface-dry. The latter relates better to as-delivered concrete.

Analysis of Masonry Mortars, Grouts, Stucco, and Other Portland-Cement-Based Materials

The cement content methods detailed earlier have been employed by various laboratories to analyze mortars and other portland-cement-based materials, usually on the mistaken belief that they are made without coarse aggregate. Such materials often contain, in addition to portland cement and sand, hydrated lime, finely ground limestone, or other substances. Fine limestone, for example, is the major component of most masonry cement. Therefore, analyses based on calcium will overestimate portland cement content, analyses based only on soluble silica underestimate total cementitious material, and analyses based on the maleic acid procedure usually cannot differentiate between the cementitious components.

Portland cement products should generally be analyzed only in conjunction with microscopic studies by an experienced petrographer who meets the requirements of ASTM C 856. With the guidance of the petrographer, successful analyses are often possible by determining portland cement through the soluble silica data, and hydrated lime or calcium carbonate from the calcium data after subtracting off the portland cement contribution. Such analyses are in error to the extent that the aggregate contains soluble calcium or silica. A procedure was outlined by Erlin and Hime [9], and is the basis for ASTM C 1324.

The methods described herein are usually applicable to such materials as those used for patching, dry packing, floor leveling, and the like.

Determination of Other Concrete Constituents

The analysis of concrete for cement content alone usually provides insufficient information to adequately determine cause for distress. In many cases, determinations of the presence of cement additives, concrete admixtures, and of water content will provide more useful information. These determinations are outlined in the following paragraphs.

Additives and Admixtures

Organic additives to cement and admixtures to concrete are often present in concentrations of a few thousandths of a percent. In general, appropriate extraction and spectroscopic analysis procedures allow determination of most of the commercially available products.

Spectroscopic procedures applicable to the active organic components of most commercial products were described by Connolly et al. [10] and, earlier, by Hime et al. [11]. Unfortunately, there have been few publications in this area since *ASTM STP 169B*. In addition to the methods of Connolly and Hime, methods have been presented by other investigators for specific compounds or admixture types:

Lignosulfonates–Kroome [12], Reul [13], Rixom [14]
Sugars–Shima and Mishi [15], Rixom [14]
Polycarboxylic Acids–Frederick and Ellis [16], Rixom [14]
Triethanolamine–Connolly and Hime [17], Reul [13], Rixom [14]
Vinsol Resin–Kroome [12], Reul [13]
Retarders–Halstead and Chaiken [18], Reul [13], Rixom [14]
Waterproofers–Reul [13], Rixom [14]

Procedures for supplementary cementitious materials such as fly ash, silica fume, and slag are almost completely proprietary to a producer or an analytical laboratory. If the particular fly ash or slag used in a concrete is available for analysis, some element not present in significant amounts in the cement or aggregate may be found, thus allowing elemental analyses for it in the concrete. Among the elements found useful in this connection for some fly ashes and slags are barium, manganese, and titanium. Petrographic examination usually will allow approximation of the fly ash or slag concentration, generally as related to the cement content. No generally accepted procedure for silica fume content has been developed. Indeed, successful methods for detection of silica fume in concrete have not been reported except when lumps of silica fume are present. In that case, optical petrography has proven successful.

For the determination of chloride in concrete, published procedures employ either water extraction or acid extraction. The former relates best to present-time corrosion potential, but the latter is more conservative. In either case, currently used procedures generally pulverize the concrete so that any chloride originally embedded in the aggregate is determined, thus often over-estimating potential for corrosion. ASTM procedures for acid-soluble chloride in cement in C 114 and ASTM Test Methods for Acid-Soluble Chloride in Mortar and Concrete (C 1152) are widely used, but most governmental agencies use the AASHTO method (T 260), Sampling and Testing for Chloride Ion in Concrete and Concrete Raw Materials. The method provides for water-soluble and acid-soluble chloride analyses of portland cement, concrete, mortar, and aggregate.

For nitrite, a component of a commercial corrosion inhibitor, a colorimetric procedure such as that of Jeknavorian et al. [26] is recommended.

The air void content of hardened concrete is determined by the procedures of ASTM C 457. Details are discussed by Hover in Chapter 26 of this book.

Aggregates

The aggregate content of concrete can be determined by the linear traverse or point count procedures of ASTM C 457, which follow those of Polivka et al. [19] and Axon [20]. Aggregate content can usually be determined as the residue from the maleic acid cement content procedure. With some siliceous aggregates, the aggregate content is determined by an insoluble residue procedure such as in ASTM C 114.

Water

The determination of the water content of hardened concrete is readily made by ignition to 1000°C and correction for carbon dioxide by any usual procedure. In contrast, there is no generally applicable method for the much more useful determination of the amount of water that was present in the fresh concrete. For non-air-entrained concrete, the procedures of Blackman [21] and Brown [22] can be employed, but are difficult to perform and may not be accurate. An estimation procedure by Axon [20] employs microscopic techniques. Many petrographers estimate water-cement ratio by petrographic techniques and experience. However, water "bleed," settlement, and other factors may lead to significant error. Substitution of cement content data determined by chemical methods in that water-cement ratio value allows at least an estimate of water content.

Instrumental Methods of Analysis

The rapid development of instrumental methods of analysis during the last half century, and the remarkable improvement of the capabilities of such instruments during the last decade or two, now allow analyses not possible before and greatly improved rapidity, sensitivity, and accuracy. As used in studies of concrete, such methods of analysis are detailed in Ref 4. A few comments on their particular advantage in certain analyses are given below.

Infrared spectroscopy (IR) allows analyses for organic components, generally as extracted from the concrete by selective solvents, and for some inorganic substances. Fourier transform infrared spectroscopy (FTIR) permits very rapid analyses or use of a very small sample. An attenuated total reflectance (ATR) attachment allows surface analyses, often without special sample preparation.

Scanning electron microscopy (SEM), when coupled with an X-ray fluorescence analyzer ("EDS" or "EDX"), is a powerful technique for determining the structure and components of such materials as concrete, or indeed of most nonorganic substances. Elements of an atomic number as low as three can be detected. Furthermore, the volume analyzed by usual EDS techniques can be as small as a trillionth of a cubic inch. The technique is thus enormously powerful. However, because the volume is so low, misinterpretation of the significance of findings has been an occasional result.

Thermal methods of analysis include differential scanning calorimetry (DSC), differential thermal analysis (DTA), and thermogravimetric analysis (TGA), and various modifications of these. These procedures usually require little sample preparation except grinding, and allow single-scan detection and quantification of many inorganic components of concrete, including calcium silicate hydrates, gypsum, plaster (calcium sulfate hemihydrate), calcium and magnesium hydroxide, ettringite, and various carbonates.

A disadvantage of some of these procedures is that the temperature of occurrence of a thermal event depends upon the particular instrument and sample holder. Ettringite thermally decomposes between about 52°C and 100°C, depending upon relative humidity and pressure, but when using DTA, for example, the temperature recorded may be 150°C or higher.

X-ray diffraction (XRD) allows rapid detection of crystalline components of portland cement or concrete. Usual instrumentation and techniques do not allow detection of components present in amounts less than a few percent. Furthermore, in a very finely ground sample even a major component may not be detectable. For example, modern day portland cements are often very finely ground, and the soft particles of gypsum reduced to a few crystal layers. Gypsum in several percent amounts may be clearly revealed by DTA, but not detected by XRD.

X-ray fluorescence (XRF) allows analysis for all except the lightest elements, depending upon the instrument. Computerization that corrects for interferences allows quantification to levels often matching those attained by most "wet chemical" procedures.

General Reference Works

Methods of analysis for cement content, as presented in the two previous editions of this book, provide considerable information about the effect of aggregate on the calcium and silica procedures, and other useful historical background. General methods for analysis of cement and concrete were presented by Hime in Refs 2, 3, 4, and 23. Methods of analysis of concrete and other cement products are presented in books by the Society of Chemical Industry [24], and by Figg and Bowden [25]. The latter contains a bibliography of 193 references. Chemical and instrumental methods of analysis of concrete by many techniques are provided in the *Handbook of Analytical Techniques in Concrete Science and Technology* [4].

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Nondestructive Tests

V. Mohan Malhotra[1]

Preface

THE SUBJECT OF NONDESTRUCTIVE TESTING OF concrete was covered in *ASTM STP 169A, ASTM STP 169B,* and *ASTM STP 169C.* The chapter in *ASTM STP 169* was entitled "Dynamic Tests" and was authored by E. A. Whitehurst, whereas the chapter in *ASTM STP 169B* was entitled "Nondestructive Tests" and was authored by E. A. Whitehurst and V. M. Malhotra. The chapter in *ASTM STP 169C* was authored by V. M. Malhotra. This chapter updates the information presented in the previous publications.

Introduction

In the inspection and testing of concrete, the use of nondestructive testing (NDT) has been making slow but steady progress since the 1950s. The slow development of these testing methods for concrete is due to the fact that, unlike steel, concrete is a highly nonhomogenous composite material, and most concrete is produced in ready-mixed plants and delivered to the construction site. The in-place concrete is, by its very nature and construction methods, highly variable, and does not lend itself to testing by traditional NDT methods as easily as steel products.

Notwithstanding the preceding, there has been considerable progress in the development of NDT methods for testing concrete in recent years. A number of these methods have been standardized by ASTM International, the International Standards Organization (ISO), and the British Standards Institute (BSI).

For the purposes of this chapter, tests that are identified generally as nondestructive may be subdivided into two main types. The first type includes those identified as sonic and pulse velocity tests, which involve the determination of the resonant frequency and the measurement of the velocity of a compressional pulse traveling through the concrete. Also included in this category are stress wave tests for locating the flaws or discontinuities that may be present, or measuring the thickness of concrete. The second type includes those tests that are used to estimate strength properties and include the surface hardness, penetration, pullout, maturity, pulloff, and combined methods. Some of these methods are not truly nondestructive because they cause some surface damage that is generally insignificant.

Resonant and Pulse Velocity Methods

Resonant Frequency Methods

Natural frequency of vibration is a dynamic property of an elastic system and is primarily related to the dynamic modulus of elasticity and density in the case of a vibrating beam. Therefore, the natural frequency of vibration of a beam can be used to determine its dynamic modulus of elasticity. Although the relationship between the two is valid for homogenous solid media that are isotropic and perfectly elastic, they may be applied to concrete when the size of a specimen is large in relation to the size of its constituent materials.

For flexural vibrations of a long, thin, unrestrained rod, the following equation or its equivalent may be found in any complete textbook on sound [1]:

$$N = \frac{m^2 k}{2\Pi L^2}\sqrt{\frac{E}{\rho}} \tag{1}$$

and solving for E

$$E = \frac{4\Pi^2 L^4 N^2 \rho}{m^4 k^2} \tag{2}$$

where

E = dynamic modulus of elasticity,
ρ = density of the material,
L = length of the specimen,
N = fundamental flexural frequency,
k = radius of gyration of the section about an axis perpendicular to the plane of bending ($k = t/\sqrt{12}$ for rectangular cross section where t = thickness), and
m = a constant (4.73 for the fundamental mode of vibration).

The dynamic modulus of elasticity can also be computed from the fundamental longitudinal frequency of vibration of an unrestrained specimen, according to the following equation [2]:

$$E = 4L^2\rho N^2 \tag{3}$$

Equations 1 and 3 were obtained by solving the respective differential equations for the motion of a bar vibrating: (1) in flexure in the free-free mode, and (2) in the longitudinal mode.

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Thus, the resonant frequency of vibration of a concrete specimen directly relates to its dynamic modulus of elasticity. If the concrete undergoes degradation, the modulus of elasticity will be altered and so will the resonant frequency of the specimen. Therefore, monitoring the change in resonant frequency allows one to infer changes in the integrity of concrete.

The method of determining the dynamic elastic moduli of solid bodies from their resonant frequencies has been in use for the past 45 years. However, until recently, resonant frequency methods had been used almost exclusively in laboratory studies. In these studies, natural frequencies of vibration are determined on concrete prisms and cylinders to calculate the dynamic moduli of elasticity and rigidity, the Poisson's ratio, and to monitor the degradation of concrete during durability tests.

The resonant frequency method was first developed by Powers [3] in the United States in 1938. He determined the resonant frequency by matching the musical tone created by concrete specimens, usually 51 by 51 by 241-mm prisms, when tapped by a hammer with the tone created by one of a set of orchestra bells calibrated according to frequency. The error likely to occur in matching the frequency of the concrete specimens to the calibrated bells was of the order of 3 %. The shortcomings of this approach, such as the subjective nature of the test, are obvious. But this method laid the groundwork for the subsequent development of more sophisticated methods.

In 1939, Hornibrook [4] refined the method by using electronic equipment to measure resonant frequency. Other early investigations on the development of this method included those by Thomson [5] in 1940, by Obert and Duvall [2] in 1941, and by Stanton [6] in 1944. In all the tests that followed the work of Hornibrook, the specimens were excited by a vibrating force. Resonance was indicated by the attainment of vibrations having maximum amplitude as the driving frequency was changed. The resonant frequency was read accurately from the graduated scale of the variable driving audio oscillator. The equipment is usually known as a sonometer and the technique is known as forced resonance.

The forced resonance testing apparatus, as described in ASTM Test Method for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215), consists primarily of two sections–one that generates mechanical vibrations and another that senses these vibrations [7]. The principal part of the vibration generation section is an electronic audio-frequency oscillator that generates audio-frequency voltages. The oscillator output is amplified and fed to the driver unit for conversion into mechanical vibrations (see Fig. 1).

The mechanical vibrations of the specimen are sensed by a piezoelectric transducer. The transducer is contained in a separate unit and converts mechanical vibrations to electrical voltage of the same frequencies. These voltages are amplified for the operation of a panel-mounted meter that indicates the amplitude of the transducer output. As the frequency of the driver oscillator is varied, maximum deflection of the meter needle indicates when resonance is attained. Visible indications that driving frequency equals the fundamental frequency can be obtained easily through the use of an auxiliary cathode-ray oscilloscope. This is because a resonance condition can be established at driving frequencies that are fractions of the fundamental frequency.

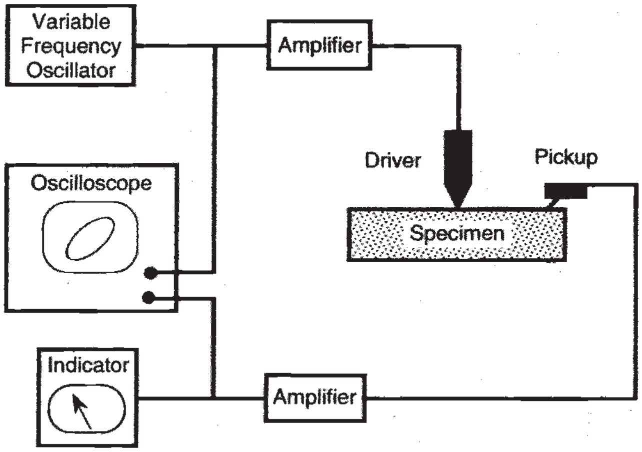

Fig. 1—Schematic of apparatus for forced resonance test.

Some skill and experience are needed to determine the fundamental resonant frequency using a meter-type indicator because several resonant frequencies may be obtained corresponding to different modes of vibration. Specimens having either very small or very large ratios of length to maximum transverse direction are frequently difficult to excite in the fundamental mode of transverse vibration. It has been suggested that the best results are obtained when this ratio is between three and five (ASTM C 215).

The supports for the specimen under test should be of a material having a fundamental frequency outside the requency range being investigated, and should permit the specimen to vibrate without significant restriction. Ideally, the specimens should be held at the nodal points, but a sheet of soft sponge rubber is quite satisfactory and is preferred if the specimens are being used for freezing and thawing studies.

The fundamental transverse vibration of a specimen has two nodal points, at distances from each end of 0.224 times the length. The vibration amplitude is maximum at the ends, about three fifths of the maximum at the center, and zero at the nodal points. Therefore, movement of the pickup along the length of the specimen and observation of the meter reading will show whether the specimen is vibrating at its fundamental frequency. For fundamental longitudinal and torsional vibrations, there is a point of zero vibration (node) at the midpoint of the specimen and the maximum amplitude is at the ends.

Sometimes in resonance testing of prismatic concrete specimens, two resonant frequencies may appear that are close together. Kesler and Higuchi [8] believed this to be caused by a nonsymmetrical shape of the specimen that causes interference due to vibration of the specimen in some direction other than that intended. Proper choice of specimen size and shape should practically eliminate this problem; for example, in a specimen of rectangular cross section the problem can be eliminated by vibrating the specimen in the direction parallel to the short side.

In performing resonant frequency tests, it is helpful to have an estimate of the expected fundamental frequency. Approximate ranges of fundamental longitudinal and flexural resonant frequencies of standard concrete specimens have been given by Jones [9].

Calculation of Dynamic Moduli of Elasticity and Rigidity and Poisson's Ratio

The dynamic moduli of elasticity and rigidity (or shear modulus of elasticity) and the dynamic Poisson's ratio of the concrete can be calculated by equations given in ASTM C 215. These are modifications of theoretical equations applicable to specimens that are very long in relation to their cross section and were developed and verified by Pickett [10], and Spinner and Teftt [11]. The corrections to the theoretical equations involve Poisson's ratio and are considerably greater for transverse resonant frequency than for longitudinal resonant frequency. For example, a standard 102 by 102 by 510-mm prism requires a correction factor of about 27 % at fundamental transverse resonance, as compared with less than 0.5 % at fundamental longitudinal resonance [12]. The longitudinal and flexural modes of vibration give nearly the same value for the dynamic modulus of elasticity. The dynamic modulus of elasticity may range from 14.0 GPa for low-quality concretes at early ages to 48.0 GPa for good-quality concrete at later ages [13]. The dynamic modulus of rigidity is about 40 % of the modulus of elasticity [14]. It should be mentioned that more input energy is needed for longitudinal resonance and, therefore, the transverse resonance mode is used more often in laboratory investigations.

Other Methods of Resonant Frequency Testing

A new method for determining fundamental frequencies has been proposed by Gaidis and Rosenberg [15] as an alternative to the forced resonance method. In this method, the concrete specimen is struck with a small hammer. The impact causes the specimen to vibrate at its natural frequencies. Hence the technique is known as impact resonance and the specimen response is measured by a lightweight accelerometer mounted on the specimen (see Fig. 2).

The amplitude and frequency of the resonant vibrations are obtained using a spectrum analyzer that determines the component frequencies via the fast Fourier transform. The amplitude of the specimen response versus frequency is displayed on the screen of a frequency analyzer, and the frequencies of major peaks can be read directly.

In operation, the pick-up accelerometer is coupled to the end of the specimen with microcrystalline wax or a similar material, and the specimen is struck lightly with a hammer. The output of the accelerometer is recorded digitally by the waveform analyzer and the recorded signal is processed to obtain the frequency response. On the resulting amplitude versus frequency curve, a dot marker (cursor) may be moved to coincide with the peak, and the frequency value of the peak is displayed on the screen. The advantages of this method over the forced-resonance procedure are the greater speed of testing, the capability of testing specimens having a wide range of dimensions, and the ability to measure readily the longitudinal frequency. However, the initial high cost of equipment appears to be a disadvantage. This impact resonance procedure was adopted by ASTM in 1991 as an alternative to the existing procedure. The various modes of vibration are obtained by the proper location of the impact point and an accelerometer.

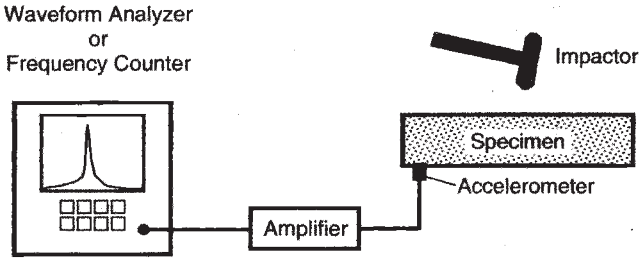

Fig. 2—Schematic of apparatus for impact resonance test.

Damping Properties of Concrete

Damping is the property of a material that causes free vibrations in a specimen to decrease in amplitude as a function of time. Several investigators, particularly Thomson [5], Obert and Duvall [2], Kesler and Higuchi [16], Shrivastava and Sen [17], and Swamy and Rigby [14], have shown that certain properties of concrete can be related to its damping ability.

There are several methods of determining the damping characteristics of a material, but two common methods used for concrete are [18]:

1. The determination of logarithmic decrement, δ, which is the natural logarithm of the ratio of any two successive amplitudes in the free vibration of the specimen.
2. Calculation of the damping constant, Q, from the amplitude versus resonance curve of the test specimen.

The measurement of the damping properties of concrete specimens has not been standardized by ASTM, but research continues toward gaining a better understanding of the significance of these measurements.

Factors Affecting Resonant Frequency and Dynamic Modulus of Elasticity

Several factors influence the resonant frequency measurements, the dynamic modulus of elasticity, or both. These include the influence of mixture proportions and properties of aggregates, specimen size effect, and the influence of curing conditions. These have been discussed in detail elsewhere [18].

Standardization of Resonant Frequency Methods

ASTM C 215 was published in 1947 and since then has been revised more than seven times. The last revision to this standard was in 2002.

The significance and use statement of the resonant frequency method as given in ASTM C 215 is as follows:

> 5.1 This test method is intended primarily for detecting significant changes in the dynamic modulus of elasticity of laboratory or field test specimens that are undergoing exposure to weathering or other types of potentially deteriorating influences.
>
> 5.2 The value of the dynamic modulus of elasticity obtained by this test method will, in general, be greater than the static modulus of elasticity obtained by using Test Method C 469. The difference depends, in part, on the strength level of the concrete.
>
> 5.3 The conditions of manufacture, the moisture content, and other characteristics of the test specimens (see section on Test Specimens) materially influence the results obtained.
>
> 5.4 Different computed values for the dynamic modulus of elasticity may result from widely different resonant frequencies of specimens of different sizes and shapes of the same concrete. Therefore, it is not advisable to compare results from specimens of different sizes or shapes.

Limitations and Usefulness of Resonant Frequency Methods

Although the basic equipment and testing procedures associated with the resonant frequency techniques have been standardized in various countries, and commercial testing equipment is easily available, the usefulness of the tests is seriously limited because of the following two points:

1. Generally, these tests are carried out on small-sized specimens in a laboratory rather than on structural members in the field because resonant frequency is affected considerably by boundary conditions and the properties of concrete. The size of specimens in these tests are usually 152 by 305-mm cylinders or 76 by 76 by 305-mm prisms.
2. The equations for the calculation of dynamic elastic modulus involve "shape factor" corrections. This necessarily limits the shape of the specimens to cylinders or prisms. Any deviation from the standard shapes can render the application of shape factor corrections rather complex.

Notwithstanding the preceding limitations, the resonance tests provide an excellent means for studying the deterioration of concrete specimens subjected to repeated cycles of freezing and thawing and to deterioration due to aggressive media. The use of resonance tests in the determination of damage by fire and the deterioration due to alkali-aggregate reaction have also been reported by Chefdeville [19] and Swamy and Al-Asali [20].

The resonant frequency test results are often used to calculate the dynamic modulus of elasticity of concrete but the values obtained are somewhat higher than those obtained with standard static tests carried out at lower rates of loading and to higher strain levels. The use of dynamic modulus of elasticity of concrete in design calculations is not recommended.

Various investigators have published correlations between the strength of concrete and its dynamic modulus of elasticity. Such correlations should not be used to predict strength properties of concrete unless similar relationships have been developed in the laboratory for the concrete under investigation [18].

Pulse Velocity Method

The application of pulse transmission techniques to the testing of concrete is believed to have had its origin with Obert [21]. Tests were made on concrete replacement pillars in mines and involved the use of two geophones, two high-gain amplifiers, and a camera with a moving film strip. Two holes, approximately 6.09 m (20 ft) apart vertically, were drilled into the pillars. The geophones were placed in the backs of the holes and the holes filled with cotton waste. A hammer blow was struck at the base of the pillar, and at the same time the camera lens was opened and the moving film strip exposed. After the film was developed, the transit time of the impulse in traveling from one geophone to the other was determined by measuring the distance between the two signals on the film, the speed of motion of the film having been controlled carefully. The velocity of the stress pulse could then be calculated.

Long and Kurtz [22] reported performing somewhat similar experiments with a seismograph in which the longitudinal velocity of the stress pulse created by a single impact was measured between arbitrarily placed geophones. They stated that only very limited experiments of this nature had been conducted but the method appeared to hold great promise providing the apparatus could be adapted to measure much shorter time intervals than could be measured with the seismograph.

Long et al. [23] undertook further investigations along these lines and in 1945 reported on the instrument and technique that resulted from their work. The apparatus consisted of two vibration pickups (in the form of phonograph cartridges), two amplifiers, two thyratron tube circuits, and a ballistic galvanometer circuit. The concrete was struck with a hammer approximately in line with the two pickups. The propagating pulse actuated the first pickup, the voltage from which energized the first thyratron and started a flow of current through the galvanometer. When the energy impulse reached the second pickup, the voltage from its amplifier ionized the second thyratron and cut off the flow of current. The deflection of the galvanometer was directly proportional to the time required for the wave to travel the distance between the two pickups.

In a discussion of this paper, the substitution of an electronic interval timer for the ballistic galvanometer was suggested. This device consists of a capacitor that begins to charge when the first thyratron is ionized and stops charging when the second is ionized and a vacuum-tube voltmeter measures the charge. The meter may be calibrated directly in units of time, thus eliminating the necessity for computations involving the magnitude of the current flowing through the galvanometer. This device was found to be more reliable than the ballistic galvanometer for field use.

Subsequent investigations in North America and Europe have resulted in the development of a number of other devices quite similar in most respects. These include the Micro-timer developed by the U.S. Bureau of Reclamation, and the Condenser Chronograph developed by the Danish National Institute of Building Research.

In 1946, the Hydro-Electric Power Commission of Ontario, Canada, in an effort to develop a technique for examining cracks in monolithic concrete structures, began a series of studies that resulted in the construction of an instrument known as the Soniscope. The device developed by Leslie and Cheesman [24] consists basically of a stress transmitter using piezoelectric crystals, a similar pulse receiver, and electronic circuits that actuate the pulse transmitter, provide visual presentation of transmitted and received signals on a cathode-ray tube, and accurately measure the time interval between the two.

The physical and electrical features of the Soniscope passed through several stages of improvement, and a number of these instruments have been built by various laboratories in the United States and Canada. The instrument was used extensively in Canada [24,25] and the United States [26].

During approximately the same time that the Soniscope was being developed in Canada and the United States, work of a similar nature was being conducted in England. These investigations resulted in the development of an instrument known as the Ultrasonic Concrete Tester. This instrument and its applications have been described at length by Jones [27,28]. The Ultrasonic Concrete Tester differed from the Soniscope primarily in the higher frequency of the transmitted pulse and the pulse repetition rate, which was about three times greater than that of the Soniscope. These changes improved the accuracy of measurement on very small specimens but limited the usefulness of the instrument for field testing, since the high frequencies suffer much greater attenuation in passing through concrete than do the lower ones. The maximum range of the Ultrasonic Concrete Tester was about 2 m, whereas that of the Soniscope in testing reasonably good concrete was 15 m or more.

Portable, battery-powered ultrasonic testing units have become available worldwide. One of the units available in the United States is called the V-Meter.[2]

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[2] This is the same unit that is manufactured in England and is known as PUNDIT.

The pulse travel time is displayed in three numerical digits that can be varied for three different ranges: (1) 0.1 to 99.9 μs with an accuracy of 0.1 μs, (2) 1 to 999 μs with an accuracy of 1 μs, and (3) 10 to 9990 μs with an accuracy of 10 μs.

Whereas the use of the resonant frequency tests has been restricted primarily to the evaluation of specimens undergoing natural or artificial weathering, pulse velocity techniques have been applied to concrete for many purposes and, in most areas of investigation, only limited agreement has been reached concerning the significance of test results. The quantity measured by all of these instruments is the travel time of stress pulses passing through the concrete under test. If the path length between transmitter and receiver is known, or can be determined, the velocity of the pulse can be computed. It is in the interpretation of the meaning of this velocity and in its use for determining various properties of concrete that agreement is incomplete. The technique is as applicable to in-place concrete as to laboratory-type specimens, and results appear to be unaffected by the size and shape of the concrete tested, within the limits of transmission of the instrument employed, provided care is taken when testing very small specimens. This, of course, is a highly desirable attribute and, in many respects, makes the pulse velocity techniques more useful than those involved in resonant frequency testing.

Because of the fundamental theoretical relationship between pulse velocity techniques and resonant frequency techniques, there is a strong inclination for users of the pulse technique to endeavor to compute the dynamic modulus of elasticity from the results of the tests. Theoretically, such values of modulus should be the same as those determined by resonant frequency tests upon the same specimens. It has been shown that on some occasions this is true and on others it is not [29]. Because of these unexplainable differences, most of those experienced in the use of pulse velocity techniques are inclined to leave their results in the form of velocity without attempting to calculate elastic moduli therefrom.

If the modulus of elasticity is to be computed from the pulse velocity, the relationship generally recommended is

$$E = V^2 \rho \frac{(1+\mu)(1-2\mu)}{(1-\mu)} \tag{4}$$

where

E = dynamic modulus of elasticity,
V = longitudinal pulse velocity,
ρ = mass density, and
μ = Poisson's ratio.

This equation relates modulus to pulse velocity and density in an infinite medium and presumably should apply only to mass concrete. However, the experience of most investigators has been that, even for very small laboratory specimens, this relationship gives better results than do those applying to either slabs or long slender members. Leslie and Cheesman [24] have suggested that the best results are obtained if, for concretes having unit weights in excess of approximately 2240 kg/m^3, the value of Poisson's ratio is assumed to be 0.24.

The use of pulse velocity techniques has been suggested for evaluating the strength of concrete, its uniformity, its setting characteristics, its modulus of elasticity, and the presence or absence of cracks within the concrete. There appears to be little question as to the suitability of such techniques to determine the presence and, to some extent, the magnitude of cracks in concrete, although it has been suggested that if the cracks are fully water-filled their locations may be more difficult to ascertain. In all of the other fields of investigation, independent investigators have reported widely different degrees of success through the use of these techniques [26].

A comprehensive report of the early experiences of users of pulse velocity techniques in the United States and Canada may be found in Ref 30. Sturrup et al. [31] have published data dealing with the experiences of Ontario Hydro, Toronto, in the use of pulse velocity for strength evaluation.

Experiences in the use of pulse velocity measurements for evaluating concrete quality have been reported in many other countries, notably Great Britain and Russia. In some instances, investigators in these countries have appeared to have greater confidence in the use of such techniques for acceptance testing than has been the case in the United States.

It is generally agreed that very high velocities of > 4570 m/s are indicative of very good concrete and that very low velocities of < 3050 m/s are indicative of poor concrete. It is further agreed that periodic, systematic changes in velocity are indicative of similar changes in the quality of the concrete. Beyond these areas of agreement, however, it appears that the investigator must have a rather intimate knowledge of the concrete involved before attempting to interpret velocity as a measure of strength or other properties of the concrete. This is particularly true if the aggregate involved is a lightweight aggregate.

Standardization of Pulse Velocity Method

ASTM Committee C9 initiated the development of a test method for pulse velocity in the late 1960s. A tentative standard was issued in 1968. A standard test method was issued in 1971 and the current edition was approved in 2002. See Fig. 3.

The significance and use statement of the test method, as given in the ASTM Test Method for Pulse Velocity Through Concrete (C 597), is as follows:

> 5.1 The pulse velocity, *V*, of longitudinal stress waves in a concrete mass is related to its elastic properties and density according to the following relationship:
>
> $$V = \sqrt{\frac{E(1-\mu)}{\rho(1+\mu)(1-2\mu)}} \tag{5}$$

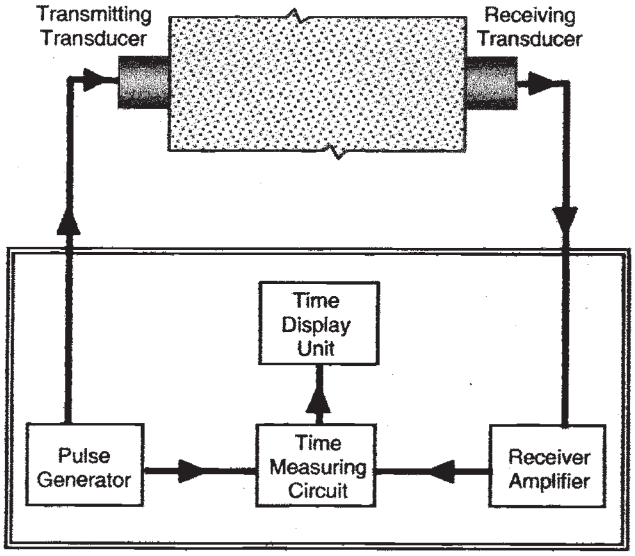

Fig. 3—Schematic of pulse velocity apparatus.

where

E = dynamic modulus of elasticity,
μ = dynamic Poisson's ratio, and
ρ = density

5.2 This test method is applicable to the assessment of the uniformity and relative quality of concrete, to indicate the presence of voids and cracks, and to evluate the effectiveness of crack repairs. It is also applicable to indicate changes in the properties of concrete, and in the survey of structures, to estimate the severity of deterioration or cracking. When used to monitor changes in condition over time, test locations are to be marked on the structure to ensure that tests are repeated at the same positions.

5.3 The degree of saturation of the concrete affects the pulse velocity, and this factor must be considered when evaluating test results (Note 1). In addition, the pulse velocity in saturated concrete is less sensitive to changes in its relative quality.

NOTE 1: The pulse velocity in saturated concrete may be up to 5 % higher than in dry concrete [32].

5.4 The pulse velocity is independent of the dimensions of the test object provided reflected waves from boundaries do not complicate the determination of the arrival time of the directly transmitted pulse. The least dimension of the test object must exceed the wavelength of the ultrasonic vibrations (Note 2).

NOTE 2: The wavelength of the vibrations equals the pulse velocity divided by the frequency of vibrations. For example, for a frequency of 54 kHz and a pulse velocity of 3500 m/s, the wavelength is 3500/54 000 = 0.065 m.

5.5 The accuracy of the measurement depends upon the ability of the operator to determine precisely the distance between the transducers and of the equipment to measure precisely the pulse transit time. The received signal strength and measured transit time are affected by the coupling of the transducers to the concrete surfaces. Sufficient coupling agent and pressure must be applied to the transducers to ensure stable transit times. The strength of the received signal is also affected by the travel path length and by the presence and degree of cracking or deterioration in the concrete tested.

NOTE 3: Proper coupling can be verified by viewing the shape and magnitude of the received waveform. The waveform should have a decaying sinusoidal shape. The shape can be viewed by means of outputs to an oscilloscope or digitized display inherent in the device.

5.6 The results obtained by the use of this test method are not to be considered as a means of measuring strength nor as an adequate test for establishing compliance of the modulus of elasticity of field concrete with that assumed in the design. The longitudinal resonance method in Test Method C 215 is recommended for determining the dynamic modulus of elasticity of test specimens obtained from field concrete because Poisson's ratio does not have to be known.

NOTE 4: When circumstances permit, a velocity-strength (or velocity-modulus) relationship may be established by the determination of pulse velocity and compressive strength (or modulus of elasticity) on a number of samples of a concrete. This relationship may serve as a basis for the estimation of strength (or modulus of elasticity) by further pulse-velocity tests on that concrete. Refer to ACI 228.1R [33] for guidance on the procedures for developing and using such a relationship.

5.7 The procedure is applicable in both field and laboratory testing regardless of size or shape of the specimen within the limitations of available pulse-generating sources.

NOTE 5: Presently available test equipment limits path length to approximately 50 mm minimum and 15 m maximum, depending, in part, upon the frequency and intensity of the generated signal. The upper limit of the path length depends partly on surface conditions and partly on the characteristics of the interior concrete under investigation. A preamplifier at the receiving transducer may be used to increase the maximum path length that can be tested. The maximum path length is obtained by using transducers of relatively low resonant frequencies (20 to 30 kHz) to minimize the attenuation of the signal in the concrete. (The resonant frequency of the transducer assembly determines the frequency of vibration in the concrete.) For the shorter path lengths where loss of signal is not the governing factor, it is preferable to use resonant frequencies of 50 kHz or higher to achieve more accurate transit-time measurements and hence greater sensitivity.

5.8 Since the pulse velocity in steel is up to double that in concrete, the pulse-velocity measured in the vicinity of the reinforcing steel will be higher than in plain concrete of the same composition. Where possible, avoid measurements close to steel parallel to the direction of pulse propagation.

Stress Wave Propagation Methods

In recent years, considerable research has been undertaken in Canada [34] by CANMET and in the United States [35,36] by NIST to develop methods to determine flaws and discontinuities in concrete. These methods are classified as stress wave propagation methods and the common feature of the various methods under development is that stress waves are introduced into concrete and their surface response is monitored using a receiving transducer connected to a digital data acquisition system capable of performing frequency analysis. One of the most attractive features of the stress wave propagation methods is that access to only one surface of concrete is required.

Two main types of stress wave methods being used are the echo method and the impulse response method. Carino has published a detailed review of the principles involved in the development of the methods [37].

Echo Method

In the echo method, a stress pulse is introduced into concrete by a transducer and its reflection by flaws or discontinuities is monitored by the same transducer that transmits the wave or by a separate transducer located near the transmitting transducer. In the former case, the technique is called pulse-echo and in the latter case the technique is known as pitch-catch. See Fig. 4.

The receiver output is displayed on an oscilloscope. If the compressional wave speed is known, time-domain analysis,

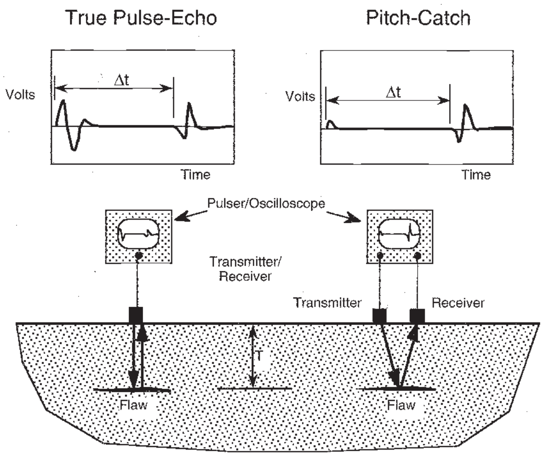

Fig. 4—Schematic of pulse-echo and pitch-catch techniques.

that is, measuring the travel time of the pulse to and from the flaw in the concrete, can be used to determine the depth of the flaw or discontinuity.

In the impact-echo method, instead of a transducer, mechanical impact is used to generate a lower frequency stress wave that can penetrate into concrete. The stress waves are reflected by the discontinuities, and the surface motion caused by the arrival of reflected waves is measured by a receiving transducer and recorded on a digital oscilloscope. Frequency analysis of the recorded signal permits measurement of the depth of the reflecting interface.

These methods are being used for thickness measurements, flaw detection, and integrity testing of piles. ASTM C 1383-98a entitled Standard Test Method for Measuring the P-wave Speed and the Thickness of Concrete Plates Using the Impact-Echo Method describes the use of this method.

Impulse Response Method

This method is somewhat similar to the impact-echo method. An instrumented hammer is used to generate a stress wave. The arrival of the reflected echos causes the concrete surface to vibrate, and the velocity of this vibration is measured by a transducer located near the point of impact. A dynamic signal analyzer is used to analyze the force-time history of the impact and the recorded velocity. From the analysis, information is obtained about the location of the reflecting interface and the dynamic stiffness of the test object. This method is primarily being used to test the integrity of the piles.

ASTM D 5882 entitled "Standard Test Method for Low Strain Integrity Testing of the Piles" issued in 2000 covers the use of this method. Impact-echo has also proven to be useful for other applications such as subgrade contact and crack depth.

Spectral Analysis of Surface Waves Method (SASW)

This method is based on the principle that when surface waves (R Waves) are created by an impact on a concrete surface, the various wavelength components of the generated surface waves penetrate to different depths in the concrete. The surface motion is monitored at two locations at a fixed distance apart, and from this information the speed of various wavelength components is extracted (Fig. 5). The data are analyzed using digital signal processing techniques and the stiffness profile of the concrete being tested can be developed. The technique was named spectral analysis of surface waves [37].

According to Carino, SAWS is the most complex of all the stress wave propogation methods and needs the services of high-strained technologists. To date the use of the SAWS method has been limited to the evaluation of concrete pavements.

Infrared-Thermographic Techniques

The use of infrared-techniques to measure delaminations in concrete bridge decks was first investigated by the Ontario Ministry of Transportation and Communications, Toronto, Canada, in the early 1970s. This technique is based on the principle that discontinuities in concrete such as delaminations in reinforced concrete caused by corrosion of reinforcement affect heat flow through concrete, and thus result in localized differences in surface temperature. By measuring these differences in surface temperature, engineers can determine the location of the delaminations. Following the original investigations in Canada, research was undertaken by various organizations in the United States, and the technique, enhanced with more sophisticated and computerized systems, was used to determine the delaminations in concrete pavements in various parts of the United States. According to Weil [38] one of the largest individual infrared thermographic inspections was carried out in 1987 at the Lambert St. Louis International Airport, and he describes the details as follows:

> This involved testing concrete taxiways. The concrete slabs ranged from 14 to 18 in. (360 to 460 mm) in thickness. The rules set up by the airport engineering department dictated that the testing had to be performed during low air traffic period (11:00 p.m. to 5:00 a.m.) and no loading gates could be blocked. The field inspection was completed in five working nights. Ap-proximately 2 000 000 ft^2 (186 000 m^2) of concrete was inspected with production rates approaching 1 000 000 ft^2 (93 000 m^2) per night. In addition to determining individual slab conditions, the use of an infrared thermography-based system with computer enhancements allowed the determination of damage caused by traffic patterns and underground erosion caused by soil migration and subsurface moisture problems.

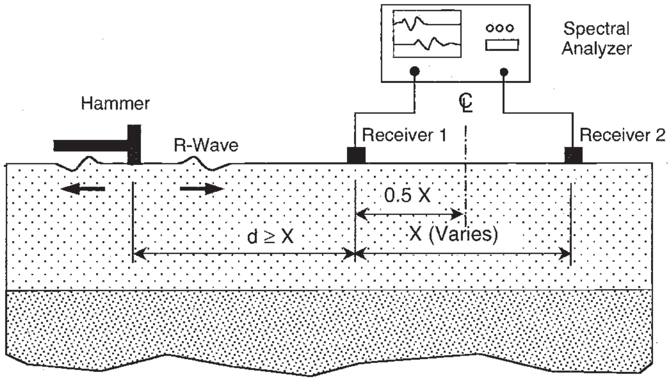

Fig. 5—Schematic of the SASW test method.

Testing Equipment

According to Weil (see previously cited reference), the equipment needed to perform infrared-thermographic investigations consists primarily of an infrared sensor, somewhat like a portable video camera, a real-time microprocessor coupled to a monitor, a data acquisition and analysis system, and image recording and retrieving devices. For pavements and bridge deck investigations, the equipment is mounted on a van and taken to the site under inspection.

Testing Procedure

The infrared-thermographic investigations on pavements and bridge decks are performed by establishing control areas of sound and poor concrete using chain dragging or coring.

Once these reference areas have been established on the pavements and images have been taken for future reference, the motor vehicle carrying the equipment described above is moved as rapidly as images can be taken. The speed of the vehicle can range anywhere up to 50 km/h. The acquired images are then subjected to detailed analysis to define areas of distress in the concrete.

Advantages and Limitations of the Infrared Thermographic Technique

The advantages of the infrared thermographic technique are obvious. It is nondestructive and is an area testing method, whereas most other nondestructive methods are "point testing." However, this method has serious limitations. The surface temperature of the area being inspected is affected by solar radiation, cloud cover, ambient temperature, wind speed, and surface moisture condition of concrete. This limits the available time during which inspections can be performed. Another limitation of the test method is that when this technique is used to determine voids in concrete, the test method cannot determine depth or thickness of the void. Almost all of the reported case histories deal with inspection of bridge decks and pavements indicating its limited use in the concrete building industry.

Standardization of Infrared-Thermographic Technique

ASTM D 4788 Test Method for Detecting Delaminations in Bridge Decks Using Infrared Thermography was issued in 1988, and describes in detail the test procedures associated with this technique.

Ground Penetrating Radar (GPR)

Ground Penetrating Radar[3] (GPR) is a nondestructive detection tool for determining delaminations in concrete pavements and bridge decks, and for estimating the thickness of concrete pavements. It is based on the principle that different solids have different scattering and reflection response to the transmission of electro-magnetic waves, and these response patterns can be used to determine various properties of fresh and hardened concrete, but primarily delaminations in concrete and its thickness.

Equipment

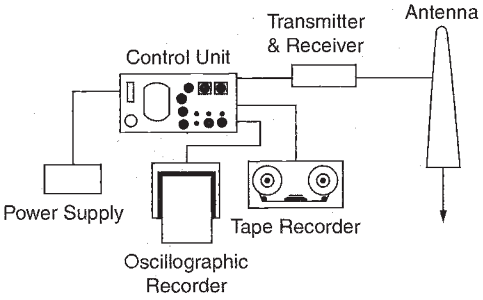

Fig. 6—Components of a typical GPR system.

A simple radar system consists of a control unit, an antenna that is used for both transmitting and receiving, an oscillographic recorder, and a power source (Fig. 6). Cleména [39] describes the operation of the system as follows:

> In operation, a circuit within the radar control unit generates a trigger pulse signal at a rate of 50 kHz, i.e., a pulse at every 20 s. Each trigger pulse, in turn, causes a solid-state impulse generator in the transmitting antenna to produce a pulse with a very fast rise time, which is then electrically discharged from the antenna in the transducer as a short burst of electromagnetic energy. The resulting pulse is then radiated into the material being examined.
>
> As the radiated pulses travel through the material, different reflections will occur at interfaces that represent changing dielectric properties. Each reflected electromagnetic pulse arrives back at the receiving antenna at a different time that is governed by the depth of the corresponding reflecting interface and the dielectric constant of the intervening material. A receiver circuit reconstructs the reflected pulses at an expanded time scale by a time-domain sampling technique. The resulting replicas of the received radar signals are amplified and further conditioned in the control unit before they are fed to an output.

Detecting Delaminations in Concrete

The principle underlying the use of GPR for determining the delaminations in concrete is best described by Cleména (see the previously cited reference) as follows:

> When a beam of microwave energy is directed at a reinforced concrete slab (Fig. 7), a portion of the energy is reflected from the surface of the concrete, and the remaining energy penetrates this interface. The surface reflection has a negative polarity since the dielectric constant of concrete, which has been reported to range from 6 when dry to about 12 when saturated, is considerably higher than that of air, which is 1. (It must be noted that the actual *in situ* relative dielectric constant of concrete and most materials will vary because it is affected to varying degrees by not only its water content but also by its conductivity, mineral composition, etc.)
>
> As the remaining microwave energy propagates into the concrete, a portion of the beam will be

---

[3] Radio detection and ranging.

> completely reflected and scattered as it strikes the top mat of reinforcement. This reflection will also have a negative polarity, since the dielectric constant of metal is infinite compared with that of the surrounding concrete. The remaining energy will continue deeper into the concrete slab until a portion of it strikes the second mat of reinforcement and the same reflection and scattering process occurs. Eventually, some portion of the original beam of microwave energy will reach the bottom of the concrete slab, and some of it will be reflected at the concrete/air interface to give a positive reflection signal. The remainder will penetrate through this interface and be lost from the receiving antenna.
>
> When a concrete slab is delaminated, usually at the level of the top mat of reinforcement, there is an additional reflection from the deteriorated section. This additional reflection, usually of negative polarity, serves as an indicator of the presence of a delamination in the concrete slab.

When inspecting small areas of concrete, the antenna is placed with its transmitting face parallel to and at a distance of 0.2 to 0.3 m from the surface of the concrete. In cases of very thick sections, the antenna may be placed directly above the concrete.

When inspecting large areas of concrete, the antenna is mounted on the front or rear of the inspection van which has the instrumentation for measuring the horizontal distance. The A tape recorder keeps a continuous record of the radar signals, and these are subjected to detailed analysis later.

ASTM D 6087 issued in 1997 deals with the detection of delaminations in concrete bridge decks covered with asphalt.

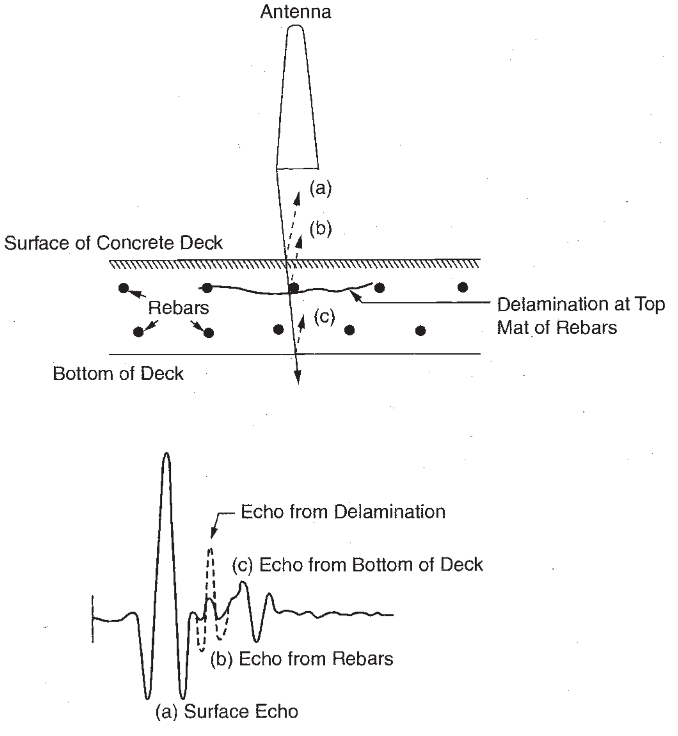

Fig. 7—Radar echoes from the cross section of a reinforced concrete deck. The presence of a delamination causes additional reflection of the incident energy.

Measurement of Thickness of Concrete Pavement

GPR has been used to determine the thickness of concrete pavements. ASTM D 4748 issued in 1987 describes in detail the test procedures.

According to Cleména, the principle of the test method is that when the dielectric constant of a concrete slab is uniform and known, the two-way transit time of microwave pulses through the concrete is directly proportional to the thickness of the slabs.

Surface Hardness Methods

The increase in the hardness of concrete with age and strength has led to the development of test methods to measure this property. These methods consist of the indentation type and those based on the rebound principle. The indentation methods consist principally of impacting the surface of concrete by means of a given mass having a given kinetic energy and measuring the width or depth, or both, of the resulting indentation. The methods based on the rebound principle consist of measuring the rebound of a spring-driven hammer mass after its impact with the concrete.

Rebound Method

In 1948, a Swiss engineer, Ernst Schmidt [40–42], developed a test hammer for measuring the hardness of concrete by the rebound principle. The Schmidt rebound hammer is principally a surface hardness tester with little apparent theoretical relationship between the strength of concrete and the rebound number of the hammer. However, within limits, empirical correlations have been established between strength properties and the rebound number. Further, Kolek [43] has attempted to establish a correlation between the hammer rebound number and the hardness as measured by the Brinell method.

Description

The Schmidt rebound hammer weighs about 1.8 kg and is suitable for use both in a laboratory and in the field. The main components include the outer body, the plunger, the hammer mass, and the main spring. Other features include a latching mechanism that locks the hammer mass to the plunger rod and a sliding rider to measure the rebound of the hammer mass. The rebound distance is measured on an arbitrary scale marked from 10 to 100. The rebound distance is recorded as a "rebound number" corresponding to the position of the rider on the scale.

Method of Testing

To prepare the instrument for a test, the plunger is released from its locked position by pushing the plunger against the concrete and slowly moving the body away from the concrete. This causes the plunger to extend from the body and the latch engages the hammer mass to the plunger rod. The plunger is then held perpendicular to the concrete surface and slowly the body is pushed towards the test object. As the body is pushed, the main spring connecting the hammer mass to the body is stretched. When the body is pushed to the limit, the latch is automatically released, and the energy stored in the spring propels the hammer mass toward the plunger tip. The mass impacts the shoulder of the plunger rod and rebounds. During rebound, the slide indicator travels with the hammer mass and records the rebound distance. A button on the side of the body is pushed to lock the plunger in the retracted position, and the rebound number is read from the scale.

The test can be conducted horizontally, vertically upward or downward, or at any intermediate angle. Due to different effects of gravity on the rebound as the test angle is changed, the rebound number will be different for the same concrete and require separate calibration or correlation charts [44,45].

Correlation with Strength

Each hammer is furnished with correlation curves developed by the manufacturer using standard cube specimens. However, the use of these curves is not recommended because material and testing conditions may not be similar to those in effect when the calibration of the instrument was performed.

A typical curve established by Zoldners [46] for limestone aggregate concrete is shown in Fig. 8. This curve was based on tests performed at 28 days using different concrete mixtures.

Although the rebound hammer provides a quick, inexpensive means of checking the uniformity of concrete, it has serious limitations and these must be recognized. The results of the Schmidt rebound hammer test are affected by:

1. smoothness of test surface;
2. size, shape, and rigidity of specimens;
3. age of test specimens;
4. surface and internal moisture conditions of the concrete;
5. type of coarse aggregate;
6. type of cement (portland, high alumina, super sulfated);
7. type of mold; and
8. carbonation of the concrete surface.

These limitations are discussed in detail elsewhere (Ref 18, Chapter 1).

To gain a basic understanding of the complex phenomena involved in the rebound test, Akashi and Amasaki [47] have studied the stress waves in the plunger of a rebound hammer at the time of impact. Using a specially designed plunger instrumented with strain gages, the authors showed that the impact of the hammer mass produces a large compressive wave, σi, and a large reflected stress wave, σr, at the center of the plunger. The ratio, $\sigma r/\sigma i$, of the amplitudes of these waves and the time, *T*, between their appearance was found to depend upon the surface hardness of concrete. The rebound number was found to be approximately proportional to the ratio of the two stresses and was not significantly affected by the moisture condition of the concrete [47].

Carette and Malhotra [48] have investigated the within-test variability of the rebound hammer test at test ages of one to three days and have studied the ability of the test to determine early-age strength development of concrete for formwork removal purposes. The rebound tests were performed at one, two, and three days on plain concrete slabs, 300 by 1270 by 1220-mm in size. Also, companion cylinders and cores taken from the slabs were tested in compression.

From the analyses of the test data, the authors concluded that because of the large within-test variation, the rebound hammer test was not a satisfactory method for reliable estimates of strength development of concrete at early ages.

According to Kolek [43] and Malhotra [44,45] there is a general correlation between compressive strength of concrete

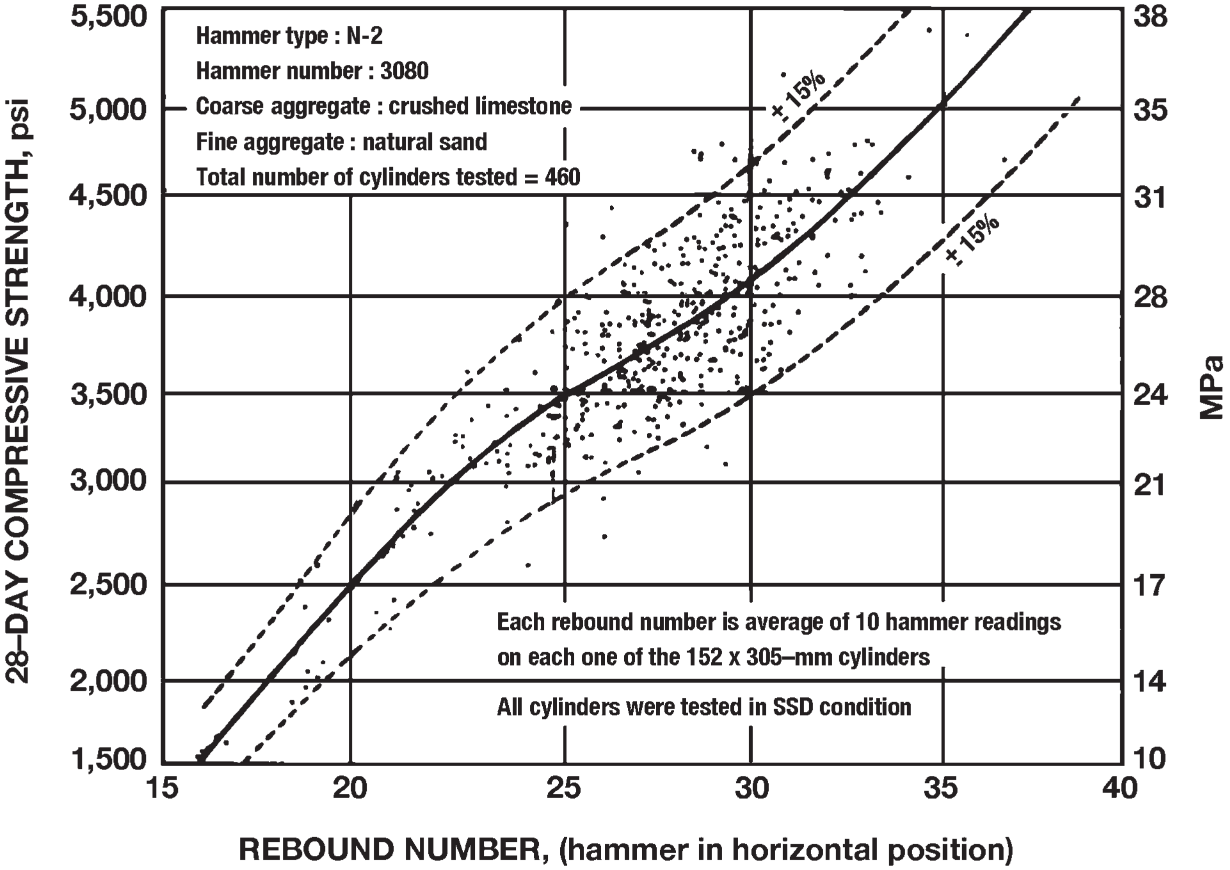

Fig. 8—Relationship between 28-day cylinder compressive strength and rebound number for limestone aggregate concrete obtained with Type N2 hammer (from Ref 46).

and the hammer rebound number. However, there is a wide degree of disagreement among various researchers concerning the accuracy of the estimation of strength from the rebound readings and the empirical relationship. Coefficients of variation for estimated compressive strength for a wide variety of specimens averaged 18.8 % and exceeded 30 % for some groups of specimens. The large deviations in strength can be narrowed down considerably by developing a proper correlation curve for the hammer, which allows for various variables discussed earlier. By consensus, the accuracy of estimation of compressive strength of test specimens cast, cured, and tested under laboratory conditions by a properly calibrated hammer lies between ± 15 and ± 20 %. However, the probable accuracy of estimation of concrete strength in a structure is ± 25 %.

Greene [49] and Klieger et al. [50] have established correlation relationships between the flexural strength of concrete and the hammer rebound number. They have found that the relationships are similar to those obtained for compressive strength, except that the scatter of the results is greater. Further, they found that the rebound numbers for tests conducted on the top of a finished surface of a beam were 5 to 15 % lower than those conducted on the sides of the same beam.

Limitations

The limitations of the Schmidt hammer should be recognized and taken into account when using the hammer. It cannot be over-stressed that the hammer must not be regarded as a substitute for standard compression tests but as a method for determining the uniformity of concrete in the structures, and comparing one concrete against another.

The rebound method has won considerable acceptance, and standards have been issued both by ASTM and ISO and by several countries for determining the rebound number of concrete.

Standardization of Rebound Test Method

ASTM Test Method for Rebound Number of Hardened Concrete (C 805) was revised in 2002; the significance and use statement of the test method as given in ASTM C 805-02 is as follows:

> 5.1 This test method is applicable to assess the in-place uniformity of concrete, to delineate regions in a structure of poor quality or deteriorated concrete, and to estimate in-place strength development.
>
> 5.2 To use this test method to estimate strength requires establishing a relationship between strength and rebound number. The relationship shall be established for a given concrete mixture and given apparatus. The relationship shall be established over the range of concrete strength that is of interest. To estimate strength during construction, establish the relationship by performing rebound number tests on molded specimens and measuring the strength of the same or companion molded specimens. To estimate strength in an existing structure, establish the relationship by correlating rebound numbers measured on the structure with the strengths of cores taken from corresponding locations. See ACI 228.1R [33] for additional information on developing the relationship and on using the relationship to estimate in-place strength.
>
> 5.3 For a given concrete mixture, the rebound number is affected by factors such as moisture content of the test surface, the method used to obtain the test surface (type of form material or type of finishing), and the depth of carbonation. These factors need to be considered in preparing the strength relationship and interpreting test results.
>
> 5.4 Different hammers of the same nominal design may give rebound numbers differing from 1 to 3 units. Therefore, tests should be made with the same hammer in order to compare results. If more than one hammer is to be used, perform tests on a range of typical concrete surfaces so as to determine the magnitude of the differences to be expected.
>
> 5.5 This test method is not intended as the basis for acceptance or rejection of concrete because of the inherent uncertainty in the estimated strength.

Probe Penetration Test

Penetration resistance methods are based on the determination of the depth of penetration of probes (steel rods or pins) into concrete. This provides a measure of the hardness or penetration resistance of the material that can be related to its strength [18].

The measurement of concrete hardness by probing techniques was reported by Voellmy [51] in 1954. Apart from the data reported by Voellmy, there is little other published work available on these tests, and they appear to have received little acceptance in Europe or elsewhere. Perhaps the introduction of the rebound method around 1950 was one of the reasons for the failure of these tests to achieve general acceptance [18].

In the 1960s, the Windsor probe test system was introduced in North America, and this was followed by a pin penetration test in the 1980s.

The Windsor probe test system was advanced for penetration testing of concrete in the laboratory as well as in-situ. The device was meant to estimate the quality and compressive strength of in-situ concrete by measuring the depth of penetration of probes driven into the concrete by means of a powder-actuated driver. The development of this technique was closely related to studies reported by Kopf [52]. Results of the investigations carried out by the New York Port Authority were presented by Cantor [53] in 1970. Meanwhile, a number of other organizations had initiated exploratory studies of this technique [54–56], and a few years later Arni [57,58] reported the results of a detailed investigation on the evaluation of the Windsor probe, while Malhotra [59–61] reported the results of his investigations on both 150 by 300-mm cylinders and 610 by 610 by 200-mm concrete slabs.

In 1972, Klotz [62] stated that extensive application of the Windsor probe test system had been made in investigations of in-place compressive strength of concrete and in determinations of concrete quality. The Windsor probe had been used to test reinforced concrete pipes, highway bridge piers, abutments, pavements, and concrete damaged by fire. In the 1970s, several U.S. federal agencies and state highway departments reported investigations on the assessment of the Windsor probe for in-situ testing of hardened concrete [63–67]. In 1984, Swamy and Al Hamed [68] in the United Kingdom published results of a study on the use of the Windsor probe system to estimate the in situ strength of both lightweight and normal-weight concretes.

Description of Test

The Windsor probe consists of a powder-actuated gun or driver, hardened alloy-steel probes, loaded cartridges, a depth gage for measuring the penetration of probes, and other related equipment. The probes have a tip diameter of 6.3 mm, a length of 79.5 mm, and a conical point. Probes 7.9 mm in diameter are also available for the testing of lightweight aggregate concrete. The rear of the probe is threaded and screws into a probe-driving head that is 12.7 mm in diameter and fits snugly into the bore of the driver. The probe is driven into the concrete by the firing of a precision powder charge [7,18]. For testing of relatively low-strength concrete, the power level can be reduced by pushing the driver head further into the barrel.

The method of testing is relatively simple and is given in ASTM Test Method for Penetration Resistance of Hardened Concrete (C 803). The area to be tested must have a brush finish or a smooth surface. To test structures with coarse finishes, the surface first must be ground smooth in the area of the test. Briefly, the powder-actuated driver is used to drive a probe into concrete. If flat surfaces are to be tested, a suitable locating template is used to provide a 178-mm equilateral triangular pattern, and three probes are driven into the concrete, one at each corner. The exposed lengths of the individual probes are measured by a depth gage. The manufacturer also supplies a mechanical averaging device for measuring the average exposed length of the three probes fired in the triangular pattern. The mechanical averaging device consists of two triangular plates. The reference plate with three legs slips over the three probes and rests on the surface of the concrete. The other triangular plate rests against the tops of the three probes. The distance between the two plates, giving the mechanical average of exposed lengths of the three probes, is measured by a depth gage inserted through a hole in the center of the top plate. For testing structures with curved surfaces, three probes are driven individually using the single probe locating template. In either case, the measured average value of exposed probe length may then be used to estimate the compressive strength of concrete by means of appropriate correlation data.

The manufacturer of the Windsor probe test system has published tables relating exposed length of the probe with compressive strength of concrete. For each exposed length value, different values for compressive strength are given, depending on the hardness of the aggregate as measured by the Mohs' scale of hardness. The tables are based on empirical relationships established in his laboratory. However, investigations carried out by Gaynor [55], Arni [57], Malhotra [59–61], and several others [56,64,69–71] indicate that the manufacturer's tables do not always give satisfactory results. Sometimes they considerably overestimate the actual strength and in other instances they underestimate the strength. It is therefore imperative for each user of the probe to correlate probe test results with the type of concrete being used. A practical procedure for developing such a relationship is described elsewhere [72].

The correlation published by several investigators for concretes made with limestone gravel, chert, and traprock aggregates are shown in Fig. 9. Note that different relationships have been obtained for concretes with aggregates having similar Mohs' hardness numbers.

There is no rigorous theoretical analysis of the probe penetration test available. Such analysis may, in fact, not be easy to achieve in view of the complex combinations of dynamic stresses developed during penetration of the probe and the heterogeneous nature of concrete. The test involves a given initial amount of kinetic energy of the probe that is absorbed during penetration, in large part through crushing and fracturing of the concrete and in lesser part through friction between the probe and the concrete. Penetration of the probe causes the concrete to fracture within a cone-shaped zone below the surface with cracks propagating up to the surface (Fig. 10).

The probe penetrations relate to some strength parameter of the concrete below the surface, which makes it possible to establish useful empirical relationships between the depth of penetration and compressive strength.

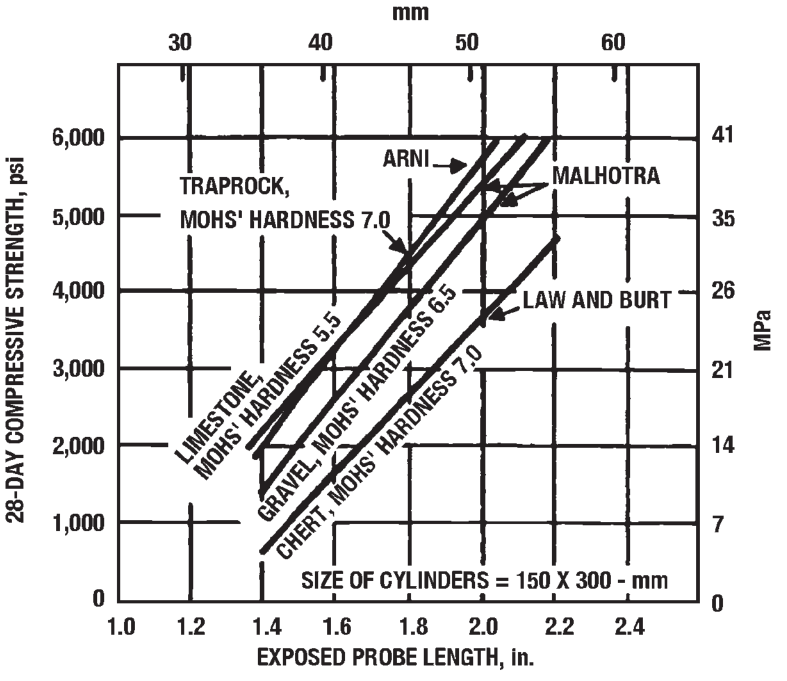

Fig. 9—Relationship between exposed probe length and 28-day compressive strength of concrete as obtained by different investigators (from Ref 72).

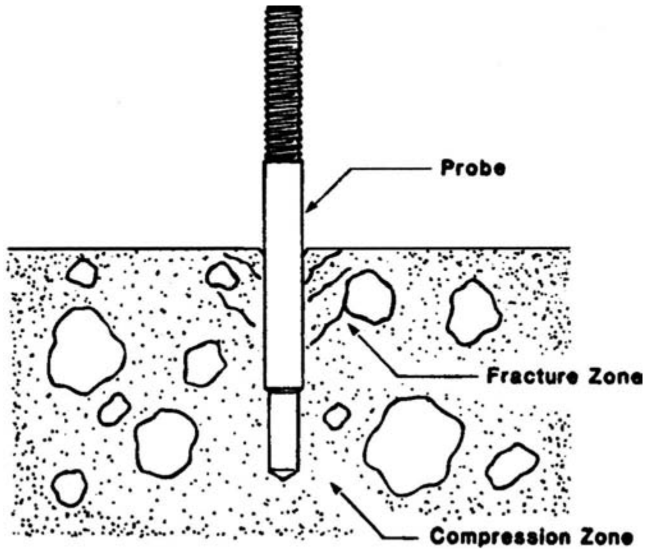

Fig. 10—Typical failure of mature concrete during probe penetration (from Ref 72).

There appear to have been no systematic attempts to determine the relative influences of these factors that could affect the probe penetration test results. However, it is generally agreed that the largest influence comes from the coarse aggregate. Apart from its hardness, the type and size of coarse aggregate used have been reported to have a significant effect on probe penetration [57,68,70]. The considerable differences shown in Fig. 9 tend to support the belief in the importance of the aggregate type. However, other parameters such as mixture proportions, moisture content, curing regime, and surface conditions are likely to have affected these correlations to some extent and could explain some of the observed differences.

The within-batch variability in the probe test results, as obtained by various investigators, is shown in Table 1. Variability is reported in terms of standard deviations and coefficients of variation with values in the latter case being calculated from the exposed length of the probe readings, although more correctly they should be based on the embedded lengths of the probe. These data show that for concrete with a maximum aggregate size of 19 mm, a typical value for the within-test coefficient of

TABLE 1—Within-Batch Standard Deviation and Coefficient of Variation of Probe Penetration Measurements

Investigation Reported by	Type of Aggregate Used	Maximum Aggregate Size, mm	Type of Specimens Tested	Total Number of Probes	Number of Probes per Test	Age of Test, days	Average Standard Deviation, mm	Average Coefficient of Variation %[a]
Arni [57]	gravel, limestone, trap rock	50	410 by 510 by 200-mm slabs	136	9	3, 7, 14, and 28	3.62	7.1
		25	410 by 510 by 200-mm slabs	198	9	3, 7, 14, and 28	2.66	5.4
Malhotra [72]	limestone	19	152 by 305-mm cylinders	20	2	7 and 28	3.14	7.7
	gravel	19	150 by 150 by 200-mm slabs	48	3	7 and 28	1.37	3.4
		19	150 by 150 by 1690-mm prisms	28	2	35	1.57	3.4
		19	610 by 610 by 200-mm slabs	48	3	7 and 28	2.21	5.5
Gaynor [55]	quartz	25	150 by 580 by 1210-mm walls	384	16	3 and 91	4.05	...
	semi-lightweight expanded shale as coarse aggregate	25	150 by 580 by 1210-mm walls	256	9	3 and 91	4.30	...
Carette, Malhotra [48]	limestone	19	300 by 1220 by 1220-mm slabs	72	6	1, 2, and 3	2.52	8.2 (5.4)
Keiller [69]	limestone, gravel	19	250 by 300 by 1500-mm prisms	45	3	7 and 28	1.91	3.5

[a] Based on exposed length of probe, except for value in parentheses that is based on depth of penetration.

variation (based on depth of penetration) is about 5 % [73]. Statistically, for such concrete, the minimum number of individual penetration tests required to ensure that the average penetration is known with the same degree of confidence as the average standard cylinder strength (assuming a coefficient of variation of 4 % based on two cylinders) would be three. This number, however, would not ensure that the in-situ strength is known with the same degree of confidence, since, obviously, the preceding within-test coefficient of variation for the probe penetration test does not take into account the uncertainty of the correlation relationship, which also affects the reliability of the estimated strength.

An increasingly important area of the application of nondestructive techniques is in the estimation of early-age strength of concrete for the determination of safe form-removal times. Relatively little information has been published in regard to the performance of the probe penetration test at early ages. However, by the late 1970s, it had been reported that the probe penetration test was probably the most widely used nondestructive method for the determination of safe stripping times [66]. One main advantage cited was the great simplicity of the test: “One simply fires the probes into the concrete and compares penetration to previously established criteria. If the probes penetrate too far, the contractor knows the concrete is not yet strong enough” [66].

Carette and Malhotra [48] have investigated in the laboratory the within-test variability at the ages of one to three days of the probe penetration test, and the ability of the test to indicate the early-age strength development of concrete for formwork removal purposes. The penetration tests were performed at one, two, and three days on plain concrete slabs, 300 by 1220 by 1220-mm in size, along with compression tests on standard cylinders and cores taken from the slabs. Excellent correlations between compressive strength and probe penetration were observed at these ages for each concrete. From the analysis of the test data, the authors concluded that unlike the rebound method, the probe penetration test can estimate the early-age strength development of concrete with a reasonable degree of accuracy, and thus can be applied to determine safe stripping times for the removal of formwork in concrete construction.

Probe Penetration Test Versus Core Testing

The determination of the strength of concrete in a structure may become necessary when standard cylinder strength test results fail to comply with specified values, or the quality of the concrete is being questioned because of inadequate placing or curing procedures. It may also be required in the case of older structures in which changes in the quality of the concrete must be investigated. In these instances, the most direct and common method of determining the strength of concrete is through drilled core testing; however, some nondestructive techniques such as the probe penetration test have been gaining acceptance as a means to estimate the in-situ strength of concrete [66,74,75].

It has been claimed that the probe penetration test is superior to core testing and should be considered as an alternative to the latter for estimating the compressive strength of concrete. It is true that the probe test can be carried out in a matter of minutes, whereas cores, if from exposed areas and if they have to be tested in accordance with ASTM Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete (C 42), must be soaked for 40 h; also, the cores may have to be transported to a testing laboratory, causing further delay in getting the results. However, the advantages of the probe penetration test should be judged against the precision of its test results, and the following statement by Gaynor [55] should be of interest in this regard:

> Based on these tests, the probe system does not supply the accuracy required if it is to replace conventional core tests. However, it will be useful in much the same manner that the rebound hammer is useful. In these tests, neither the probe system nor the rebound hammer provides precise quantitative estimates of compressive strength of marginal concretes. Both should be used to locate areas of relatively low- or relatively high-strength concretes in structures.

It must be stressed that in cases in which standard cylinder or cube strength is strictly the parameter of interest because of the specifications being expressed primarily in these terms, the core test that provides a direct measure of compressive strength clearly remains the most reliable means of estimating in-situ strength. In new construction, however, it has been found possible to establish, within certain limits of material composition and testing conditions, relationships between probe penetration and strength that are accurate enough that the probe test can be used as a satisfactory substitute for the core test [75].

In the early 1980s, a survey of concrete testing laboratories in Canada and the United States indicated that the Windsor probe penetration technique was the second most often used method for in-situ strength testing of concrete (see ASTM C 42). The survey included methods such as rebound hammer, probe penetration, pullout, pulse velocity, maturity, and cast-in-place cylinder. In terms of reliability, simplicity, accuracy, and economy, the probe test was given one of the best combined ratings.

Advantages and Disadvantages of the Probe Penetration Test

The probe penetration test system is simple to operate, rugged, and needs little maintenance except for occasional cleaning of the gun barrel. The system has a number of built-in safety features that prevent accidental discharge of the probe from the gun. However, wearing safety glasses is required. In the field, the probe penetration test offers the main advantages of speed and simplicity, and of requiring only one surface for the test. That its correlation with concrete strength is affected by a relatively small number of variables is an advantage over some other methods for in-situ strength testing.

However, the probe test has limitations that must be recognized. These include minimum size requirements for the concrete member to be tested. The minimum acceptable distance from a test location to any edges of the concrete member or between two given test locations is of the order of 150 to 200 mm, while the minimum thickness of the members is about three times the expected depth of penetration. Distance from reinforcement can also have an effect on depth of probe penetration especially when the distance is less than about 100 mm [76]. The importance of aggregate type and aggregate content on the correlation is emphasized.

As previously indicated, the uncertainty of the estimated strength value, in general, is relatively large and the test results may lack the degree of accuracy required for certain applications. The test is limited to a certain range of strength (<40 MPa), and the use of two different power levels to accommodate a larger range of concrete strength within a given investigation complicates the correlation procedures. Finally, as noted earlier, the test causes minor damage to the surface that generally needs to be repaired.

Pin Penetration Test

In the late 1980s, Nasser and Al-Manaseer [77,78] reported the development of a simple pin penetration test for the determination of early-age strength of concrete for removal of concrete formwork. Briefly, this apparatus consists of a device that grips a pin having a length of 30.5 mm, a diameter of 3.56 mm, and a tip machined at an angle of 22.5°. The pin is held within a shaft that is encased within the main body of the tester. The pin is driven into the concrete by a spring that is mechanically compressed when the device is prepared for a test. The spring is reported to have a stiffness of 49.7 N/mm and stores about 10.3 J of energy when compressed.

When ready for testing, the apparatus is held against the surface of the concrete to be tested, and a triggering device is used to release the spring forcing the pin into the concrete. Following this, the apparatus is removed, and the small hole created in the concrete is cleared by means of an air blower. A depth gage is used to measure the penetration depth.

The test, though simple in concept, has limitations. The pin penetrates only a small depth into the concrete, and therefore the results can be seriously affected by the conditions of the material at the surface. The test results are invalid when an aggregate particle is struck. The simplicity of the test makes it possible to obtain as many readings as necessary at little extra cost, but the equipment is rather heavy for field use and, because of the nature of the spring mechanism, cannot be used for concrete with strength greater than about 30 MPa. Calibration of the equipment is important to ensure a consistent level of energy and its frequent verification may be necessary.

Standardization of Penetration Resistance Techniques

ASTM Committee C9 initiated the development of a standard for the probe penetration technique in 1972 and a tentative test method covering its use was issued in 1975. A standard test method designated ASTM C 803 was issued in 1982, the latest edition was issued in 2003, and the pin penetration method was added as an alternative penetration test. The significance and use statement of the test method as given in the 2003 standard is as follows:

> 5.1 This test method is applicable to assess the uniformity of concrete and to delineate zones of poor quality or deteriorated concrete in structures.
>
> 5.2 This test method is applicable to estimate in-place strength, provided that a relationship has been experimentally established between penetration resistance and concrete strength. Such a relationship must be established for a given test apparatus (see also 9.1.5), using similar concrete materials and mixture proportions as in the structure. Use the procedures and statistical methods in ACI 228.1R for developing and using the strength relationship [33].
>
> NOTE 1: Since penetration results may be affected by the nature of the formed surfaces (for example, wooden forms versus steel forms), correlation testing should be performed on specimens with formed surfaces similar to those to be used during construction. Additional information on the factors affecting penetration test results and summaries of past research are available [79].
>
> 5.3 Steel probes are driven with a high-energy, powder-actuated driver, and probes may penetrate some aggregate particles. Probe penetration resistance is affected by concrete strength as well as the nature of the coarse aggregate. Steel pins are smaller in size than probes and are driven by a low energy, spring-actuated driver. Pins are intended to penetrate the mortar fraction only; therefore, a test in which a pin strikes a coarse aggregate particle is disregarded.
>
> 5.4 This test method results in surface damage to the concrete, which may require repair in exposed architectural finishes.

Pullout Test

A pullout test, by using a dynamometer and a reaction bearing ring, measures the force required to pull out from concrete a specially shaped insert whose enlarged end has been cast into the concrete. Because of its shape, the insert is pulled out with a cone of the concrete. The concrete is simultaneously in tension and in shear, and the generating lines of the cone are defined by the key dimensions of the insert and bearing ring (Fig. 11). The pullout force is then related to compressive strength by means of a previously established relationship.

The pullout techniques, though in use in the former Union of Soviet Socialist Republics [80] since 1935, are relatively new elsewhere [81,82]. In 1944 in the United States, Tremper [83] reported results of laboratory studies dealing with pullout tests covering strengths up to 35.2 MPa. In 1968, Tassios [84], in Greece, reported the development of a nail pullout test.

In the 1970s Kierkegaard-Hansen [85], in Denmark, and Richards [86], in the United States, advocated the use of pullout tests on structural concrete members. A number of researchers including Malhotra [81], Carino [87], and others [82] have published data dealing with laboratory studies and field testing. A pullout system known as LOK-Test based on Kierkegaard-Hansen's work is now available commercially. Carino [87] has summarized analytical and experimental stud-

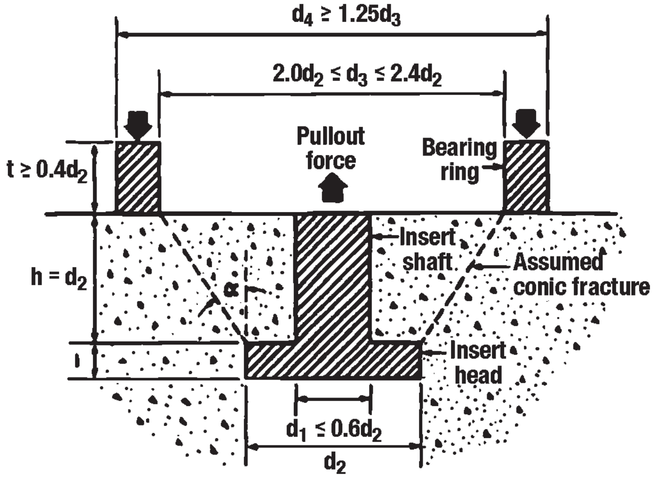

Fig. 11—Schematic cross section of pullout test (from ASTM C 900).

ies to gain an understanding of the fundamental failure mechanisms of the pullout test and he concluded as follows:

1. It is understood that the pullout test subjects the concrete to a nonuniform, three-dimensional state of stress. It also has been demonstrated that there are at least two circumferential crack systems involved: a stable primary system which initiates at the insert head at about ⅓ of the ultimate load and propagates into the concrete at a large apex angle; and a secondary system which defines the shape of the extracted cone. However, there is not a consensus on the failure mechanism at the ultimate load. Some believe that ultimate load occurs as a result of compressive failure of concrete along a line from the bottom of the bearing ring to the top face of the insert head. This could explain why good correlation exists between pullout strength and compressive strength. Others believe that the ultimate failure is governed by aggregate interlock across the secondary crack system, and the ultimate load is reached when sufficient aggregate particles have been pulled out of the matrix. In the latter case, it is argued that good correlation exists between pullout strength and compressive strength because both properties are controlled by the strength of the mortar.
2. Despite the lack of agreement on the exact failure mechanism, it has been shown that pullout strength has good correlation with compressive strength of concrete, and that the test has good repeatability.

The main advantage of the pullout test is that it provides a direct measure of the in-situ strength of concrete. The method is relatively simple and testing can be done in the field in a matter of minutes.

An example of the correlation between compressive strength and pullout strength or load is shown in Fig. 12. Such correlations depend on the geometry of the pullout test configuration and the coarse aggregate characteristics.

A major disadvantage of the pullout test is that minor damage to the concrete surface must be repaired. However, if a pullout force corresponding to a given minimum strength is applied without failure, it may be assumed that a minimum strength has been reached in the concrete and the pullout insert need not be stressed to failure.

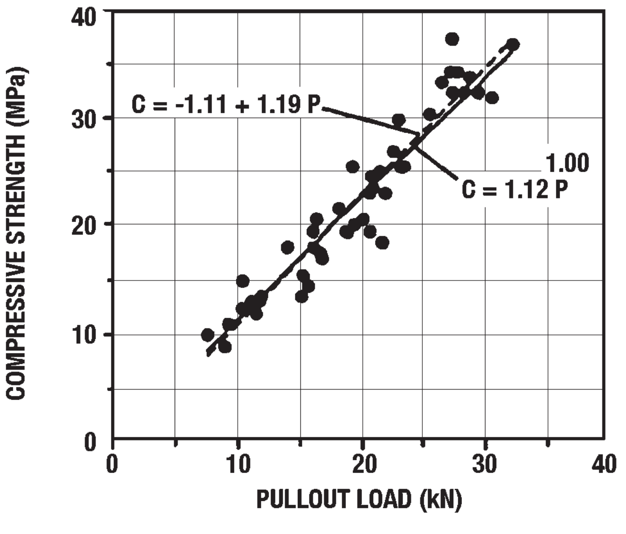

Fig. 12—Correlation data by Khoo and best-fit linear and power function relationship (from Ref 87).

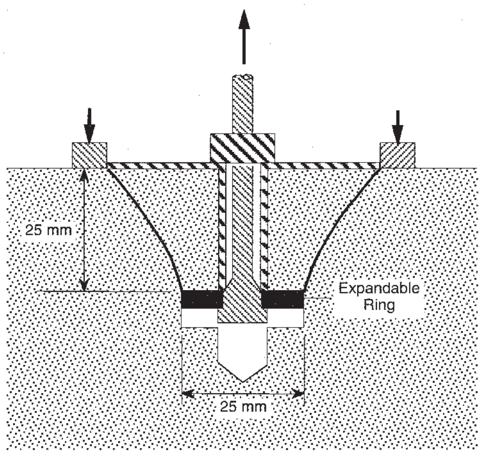

Fig. 13—The CAPO test by undercutting and using an expandable ring.

Other forms of the pullout tests include the core and pullout (CAPO) test and the drilled-in pullout tests [18]. In the CAPO test, a hole is drilled into concrete and a special milling tool is used to undercut a 25-mm-diameter slot at a depth of 25 mm. An expandable steel ring is placed into the hole, and the ring is expanded into the slot using a special device (Fig. 13).

The entire assembly used to expand the ring is then pulled out of the concrete using the LOK test loading system. However, the test has found very limited acceptance because of the high variability associated with the test data in the field tests [87]. Variability may be reduced by assuring a flat surface beneath the bearing ring.

The development of other drilled-in pullout tests have been reported by the Building Research Establishment (BRE) (United Kingdom) [88] and by Mailhot et al. [89]. In the BRE tests, an anchor bolt is placed in a hole drilled in hardened concrete, and a pullout force is applied to cause failure in concrete. There is a high degree of variability in the test results.

Another pullout method studied by Mailhot et al. [89] involved epoxy grouting a 16-mm-long threaded rod to a depth of 38 mm in a 19-mm-diameter hole. After the epoxy had cured, the rod was pulled out using a tension jack reacting against a bearing ring. Once again, the within-test variability was reported to be high.

Standardization of Pullout Tests

ASTM Committee C9 initiated the development of a standard for the pullout tests in the early 1970s, and a standard was issued in 1982 and the last revision was in 1999. The revised standard is designated as ASTM Test Method for Pullout Strength of Hardened Concrete (C 900). The pullout insert is either cast into the fresh concrete or installed in hardened concrete.

The significance and use statement as given in ASTM C 900 is reproduced here except Note 1, which refers to references given at the end of the chapter and to an appendix in the standard:

> 4.1 For a given concrete and a given test apparatus, pullout strengths can be related to compressive strength test results. Such strength relationships depend on the configuration of the embedded insert, bearing ring dimensions, depth of embedment, and level of strength development in that concrete. Pior to use, these relationships must be established for each system and

each new combination of concreting materials. Such relationships tend to be less variable where both pullout test specimens and compressive strength test specimens are of similar size, compacted to similar density, and cured under similar conditions.

4.2 Pullout tests are used to determine whether the in-place strength of concrete has reached a specified level so that, for example:

(1) post-tensioning may proceed;
(2) forms and shores may be removed; or
(3) winter protection and curing may be terminated.

In addition, post-installed pullout tests may be used to estimate the strength of concrete in existing constructions.

4.3 When planning pullout tests and analyzing test results, consideration should be given to the normally expected decrease of concrete strength with increasing height within a given concrete placement in a structural element. The measured pullout strength is indicative of the strength of concrete within the region represented by the conic frustum defined by the insert head and bearing ring. For typical surface installations, pullout strengths are indicative of the quality of the outer zone of concrete members and can be of benefit in evaluating the cover zone of reinforced concrete members.

Maturity Method

It is well known that the compressive strength of well-cured concrete increases with time. However, the increase in strength is governed by many factors other than curing time, the most important being the concrete temperature and the availability of moisture. The combined effect of time and temperature has been studied by several investigators since 1904, but no hypothesis was formulated in early years. Then, in the 1950s, the concept of maturity was advanced by McIntosh [90], Nurse [91], Saul [92], and others [93–95], and strength-maturity relationships were published. Maturity was defined as the product of time and temperature above a datum temperature of −10°C (14°F).

In 1956, Plowman [94] examined relationships between concrete strength and maturity and attempted to establish a rational basis for the datum temperature used for maturity to calculate the maturity index. He defined the datum temperature for maturity as the temperature at which the strength gain of hardened concrete ceases. From his investigations, Plowman concluded that the datum temperature was −12.2°C (10°F). These earlier studies have been extensively reviewed by Malhotra [95].

Maturity Functions

Maturity functions are mathematical expressions that convert the temperature history of concrete to an index indicative of its strength development.

The Nurse-Saul maturity function is as follows:

$$M(t) = = \sum (T_a - T_0)\Delta t \qquad (6)$$

where

$M(t)$ = the temperature-time factor at age t, degree-days or degree-hours;
Δt = a time interval, days or hours;
T_a = average concrete temperature during time interval, Δt, °C; and
T_0 = datum temperature, °C.

It has been shown that the Nurse-Saul function does not accurately represent time-temperature effects because it is based on the assumption that the rate of strength development is a linear function of temperature.

In order to overcome some of the limitations of the Nurse-Saul maturity function, the maturity function proposed by Freiesleben Hansen and Pederson [96] can be used to compute equivalent-age at a specified temperature as follows:

$$t_e = \sum e^{-[Q((1/T_a)-(1/T_s))]}\Delta t \qquad (7)$$

where

t = equivalent age at a specified temperature T_s, days or hours;
Q = activation energy divided by the gas constant, K;
T_a = average temperature of concrete during time interval Δt, K;
T_s = specified temperature, K; and
Δt = time interval, days or hours.

Equation 7 is based on the Arrhenius equation, which describes the influence of temperature on the rate of chemical reaction.

Byfors [97], Naik [98], Carino [99,100], Tank and Carino [101], and Carino and Tank [102] have shown that the equivalent age maturity function based on the Arrhenius equation accounts better for the effects of temperature on strength gain and is applicable over a wider temperature range than the Nurse-Saul maturity function.

One of the first engineering applications of the maturity method was by Swenson [103] in Canada who used it in forensic investigations to estimate the strength gain of concrete in structures.

The maturity method has been used to estimate the in-situ strength of concrete during construction. In the United States, Hudson and Steele [104] used the maturity approach to predict potential strength of concrete based upon early-age tests. Their results have been incorporated into ASTM Method for Developing Early Age Compression Test Values and Projecting Later Age Strengths (C 918). Malhotra [105] attempted to relate compressive strengths using accelerated-strength tests with the maturities for these tests.

The maturity of in-situ concrete can be monitored by thermocouples or by instruments called maturity meters. Basically, these maturity meters monitor and record the concrete temperature as a function of time using thermocouples or thermistors embedded in the fresh concrete and connected to strip-chart recorders or digital data recorders. The temperature-time factor or equivalent age is automatically computed and displayed. Also, disposable mini-maturity meters based on the Arrhenius equation have also become available. These primarily consist of a glass tube containing a liquid that has an activation energy for evaporation that is similar to the activation energy for strength gain in concrete; the amount of evaporation from the capillary tube at a given time is indicative of strength development in the concrete.

Standardization of the Maturity Method

ASTM Committee C9 initiated the development of a standard on the maturity method in 1984, and a standard was issued in 1987.

This standard is designated as ASTM Practice for Estimating Concrete Strength by the Maturity Method (C 1074). The current edition was approved in 1998 and published in 1999.

The significance and use statement as given in ASTM C 1074 is reproduced here:

1. This procedure can be used to estimate the in-place strength of concrete to allow the start of critical construction activities such as: (1) removal of formwork and reshoring; (2) post-tensioning of tendons; (3) termination of cold weather protection; and (4) opening of the roadways to traffic.
2. This procedure can be used to estimate strength of laboratory specimens cured under non-standard temperature conditions.
3. The major limitations of the maturity method are: (1) the concrete must be maintained in a condition that permits cement hydration; (2) the method does not take into account the effects of early-age concrete temperature on the long-term ultimate strength; and (3) this method needs to be supplemented by other indications of the potential strength of the concrete mixture.
4. The accuracy of the estimated strength depends on properly determining the maturity function for the particular concrete mixture.

Pulloff Tests

In the mid-1970s, researchers in the United Kingdom developed a surface pulloff test to estimate the in situ strength of concrete [106]. Although some test data have been published, the test has found little acceptance outside the United Kingdom.

Briefly, the pulloff test involves bonding a circular steel disk to the surface of the concrete under test by means of an epoxy resin. Prior to this bonding, sandpaper is used to abrade the surface of the concrete to remove laitance, followed by degreasing the surface using a suitable solvent. After the epoxy has cured, a tensile force is then applied to the steel disk. Because the tensile strength of the bond is greater than that of concrete, the latter fails in tension. From the area of the disk and the force applied at failure, it is possible to obtain a measure of the tensile strength of concrete.

The pulloff test is relatively simple to perform, and stress at failure is a direct measure of the tensile strength of concrete. The test gives reproducible results and does not require planning in advance of placing the concrete. However, the serious disadvantage of the test is that it is confined to the testing of the surface layers of concrete.

Combined Methods

In Europe in general and in Romania in particular, the use of more than one in situ/nondestructive testing technique to improve the accuracy of prediction of strength parameters of concrete has gained some credibility [107,108]. Some researchers have suggested the use of rebound hammer and pulse velocity techniques, while others have suggested the use of other combined methods. The proponents of this approach claim that the use of two methods, each measuring a different property, can overcome the limitation associated with the use of one method. Some case histories have been published supporting this claim; data disputing this claim are also available.

The use of more than one method to predict compressive strength of concrete may provide useful information in some instances but in general it is not advocated because of economy and time requirements, and because the possible increase in the accuracy of predicting compressive strength is only marginal.

Concluding Remarks

Considerable progress has been made over the past four decades in the development and use of nondestructive methods for estimating strength of concrete. A number of these methods have been standardized by ASTM, ISO, and other organizations, and several new methods are in the process of being standardized. However, research is needed to standardize those tests that determine properties other than strength. Radar scanning and impact/pulse echo techniques appear to be the most promising of these types of tests.

In-situ/nondestructive tests may not be considered as replacements for the standard cylinder test, but should be considered as additional techniques. When performed in conjuction with standard core tests, they can provide additional information and reduce the number of cores required for testing.

Unless comprehensive correlations have been established between the strength parameters to be predicted and the results of in-situ/nondestructive tests, the use of the latter to predict strength properties of concrete is not recommended.

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PART IV
Concrete Aggregates

29

Grading, Shape, and Surface Texture

Robin E. Graves[1]

Preface

IN PREPARATION OF THIS CHAPTER, THE CONTENTS of the prior edition, Chapter 35 of *ASTM STP 169C*, authored by J. E. Galloway, Jr. [1], have been retained since they continue to remain relevant to current concrete mixture technology. For information on historical development of this subject area within the *ASTM STP 169* editions, the reader is referred to the prior edition. This edition has been updated to include changes to test methods and new test methods developed since publication of the prior edition. Additionally, discussion is provided on recent research related to grading of manufactured fine aggregates, new imaging technologies for characterization of particle grading, shape, and surface texture, and emerging computer simulation techniques for concrete mixture design.

Introduction

The three attributes of concrete aggregates discussed in this chapter may be considered benign features because even a well-trained eye may not be able to discern changes unless they are in the extreme. However, even slight changes can have an effect on the characteristics of concrete mixtures.

Grading changes are perhaps more prevalent than shape and surface texture, in the case of a coarse aggregate, because of its natural tendency to segregate during stockpiling, transporting, and other forms of handling. Therefore, attention to every detail of these phases from the time of processing to the time of introduction into the concrete mixture is very important and can lessen problems later in the concrete-making process. Shape and surface texture properties are not as likely to change, but the changes that can occur will be discussed in this chapter. Unlike grading, these two attributes are not within the direct control of the user.

The items discussed here pertain to normal, lightweight, and heavyweight aggregates, because the aggregate as well as other ingredients fit together as absolute volumes in the concrete mixture and in no other way [2]. The effect these changes have will be discussed in this chapter.

Grading

Definition

Grading is simply the frequency distribution of the particle sizes of a given aggregate. This distribution is given in certain ranges for each sieve size, and each size is usually assigned an arbitrary number. ASTM C 33, Specification for Concrete Aggregates, gives a wide range for each sieve to accommodate materials from different localities and to allow for economical production considerations. These size numbers are widely used but other specifiers may have a different number assigned for virtually the same material. Until the early 1920s, aggregate “sizes” were concocted by almost everyone specifying or producing aggregate. In 1937 ASTM Committee D-4 adopted ASTM D 448, Classification for Sizes of Aggregate for Road and Bridge Construction, that listed standard sizes of aggregate in an attempt to bring uniformity within the industry. These grading sizes are reflected to a great degree in ASTM C 33, Table 2, which is largely used today for coarse aggregate by specifiers of concrete mixtures.

Test Method

The basic procedure for determining grading is ASTM C 136, Test Method for Sieve Analysis of Fine and Coarse Aggregates. This is done basically by separating the material on a nest of sieves meeting the requirements of ASTM E 11, Specification for Wire Cloth and Sieves for Testing Purposes, and determining the percent of each size present. Note that each successive sieve is approximately one-half the opening of its predecessor and all openings are expressed in SI units. Using such a system and employing a log scale, lines on a graph can be spaced at constant intervals to represent the successive sizes, and the fineness modulus can be computed [2].

The fineness modulus (FM) is an empirical figure derived by adding the cumulative percentages retained on a specified series of sieves and dividing by 100. These sieves are, in millimetres, 150 (6 in.), 76 (3 in.), 37.5 (1.5 in.), 19 (0.75 in.), 9.5 (0.375 in.), 4.75 (No. 4), 2.36 (No. 8), 1.18 (No. 16), 0.6 (No. 30), 0.3 (No. 50), and 0.15 (No. 100). A typical computation for the FM of a fine aggregate is shown in Table 1.

The FM of a coarse aggregate is computed in a similar way. It should be noted that all sieves in the series are always considered. Obviously, in the case of a coarse aggregate, 100 % would be retained on the smaller sieve sizes so a typical coarse aggregate, Size No. 57 from ASTM C 33, would have an FM of about 7.00. However, the FM on coarse aggregate is less commonly used than the FM for fine aggregate where the eye may not easily discern changes in grading. ASTM C 33 states that the FM shall not vary from the base by more than 0.20 for a given source.

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TABLE 1—Computation for the Fineness Modulus

Sieve Size, mm (No.)	Percent Retained	Cumulative Percent Retained
4.75 (4)	1	1
2.36 (8)	15	16
1.18 (16)	23	39
0.6 (30)	19	58
0.3 (50)	16	74
0.15 (100)	15	89
−0.15 (100)	11	
		277
	277 divided by 100 = 2.77 FM	

Significance

Since the FM of the fine aggregate is used in computing the mixture design in the solid volume process, changes beyond the 0.20 value would necessitate a redesign of the mixture. This fact is often overlooked and problems with workability are searched for in other areas or, in many cases, the actual FM was not determined originally, but assumed.

While ASTM C 33 contains fine aggregate grading limits, succeeding paragraphs modify these limits considerably even to the point of discarding the grading limits, provided similar aggregate from the same source has a demonstrated performance record or, in the absence of such record, if it can be shown that concrete of the class specified made with the aggregate in question has shown equal relevant properties. There are many qualifying adjectives in these statements and only someone qualified to judge these equals should make the decision.

As previously noted, the grading limits in ASTM C 33 are very broad to accommodate a wide variety of conditions. In any particular area, adjustments should be made to accommodate the local conditions. It is not meant to prohibit the concrete designer from modifying the grading range, that is, specifying more than one coarse aggregate size to be used, or any other modification, provided it can be done in an economical manner.

Once the ranges on each sieve are agreed upon by the supplier and the user, whether they are ASTM C 33 ranges or some other, the supplier should strive to produce to the midpoint of that range. Production at either extreme may statistically cause the product to fall out of the specification limits and result in problems with the concrete mixture.

What then is the ideal grading? The influence of aggregate on the properties of concrete has been extensively discussed in the technical literature during the past 125 years and many methods for arriving at "optimum" or "ideal" gradings have been presented. However, none of these has been accepted as being universally applicable because of economic considerations, differences in particle shape and texture of the aggregates, and the effects of entrained air and amount of cementitious material contained in the concrete [3–26].

Experience has demonstrated that either very fine or very coarse sand, or coarse aggregate having a large deficiency or excess of any size fraction, is usually undesirable, although aggregates with discontinuous or gap grading have sometimes been used to advantage [27–29]. These, however, are special circumstances and should only be considered by someone familiar with gap grading.

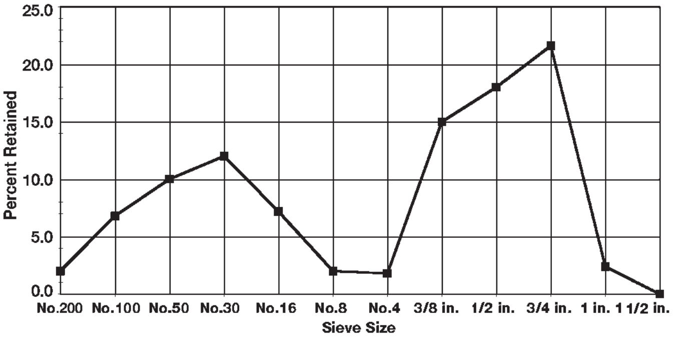

Fig. 1—Particle size distribution of combined coarse and fine aggregate showing a gap-graded aggregate mixture.

A well-graded material is the closest to ideal with a representative amount on each standard sieve size listed in that size specification. The scarcity of a particular sieve size could result in poor workability and even poor durability of the concrete. The following paragraphs address coarse and fine aggregates, but the trend is to consider the aggregate as a composite in the concrete mixture.

There have been efforts within ASTM to de-emphasize the designer's need to stay with one coarse aggregate size. That size (usually No. 57) can easily lead to a gap-graded aggregate. This would allow any grading the specifier chose and would not necessarily conform to one of the sizes in ASTM C 33.

Figure 1 is a particle size distribution chart of combined coarse and fine aggregate grading. Although the coarse aggregate meets the requirements of ASTM C 33, Size No. 57, and the concrete fine aggregate also meets C 33 requirements, the combination in this example results in a gap-graded aggregate (notice the significant peak-valley arrangement). There has been a move by some specifying agencies to require more well-graded combinations of aggregate. These gradings generally are believed to decrease water demand, improve workability, reduce segregation, decrease susceptibility to shrinkage, and improve long-term performance of concrete mixtures [30]. Figure 2 is an example of a combined grading plotted on an "8 to 18" percent retained chart that is in use by some agencies, often in conjunction with other guidelines such as coarseness and workability factors [31] and 0.45 power curves, to insure well-graded aggregate combinations. Achieving these types of gradings often requires blending of three or more aggregates to achieve the desired combined grading. A proposed standard

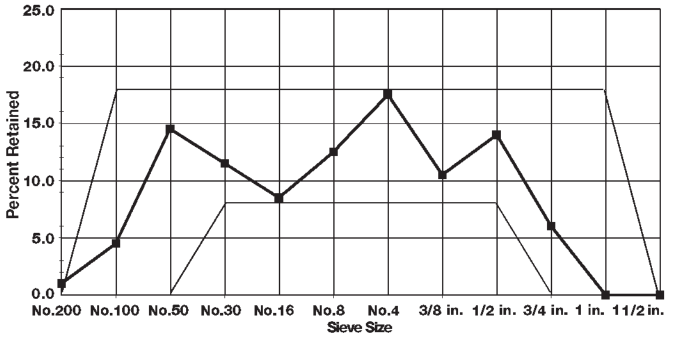

Fig. 2—Particle size distribution of combined coarse and fine aggregate, plotted on an "8 to 18" grading control chart, showing a well-graded aggregate mixture [30].

TABLE 2—Mortar Requirements for Workable Concrete With Various Maximum Aggregate Sizes

Maximum Size Aggregate	4.75 (sand)	9.5 mm	12.5 mm	19 mm	25 mm	37.5 mm	50 mm	76 mm	150 mm
NON-AIR-ENTRAINED CONCRETE									
Water, kg/m^3[a]	276	228	216	202	192	177	168	160	139
Cement, kg/m^3[b]	520	430	406	382	360	334	316	302	262
Fine aggregate, %[c]	100	62	54	49	44	40	37	34	28
Entrapped air, %	6	3	2.5	2	1.5	1	0.5	0.3	0.2
AIR-ENTRAINED CONCRETE									
Water, kg/m^3[a]	245	201	192	177	169	157	148	139	118
Cement, kg/m^3[b]	462	380	362	334	318	296	279	262	222
Fine aggregate, %[c]	100	58	50	45	40	37	34	31	25
Total air, %[d]	13	8	7	6	5	4.5	4	3.5	3

[a] Approximate amount of mixing in kilograms per cubic metre required for 75-mm slump with well-shaped angular coarse aggregate. Quantities listed can be reduced significantly through the addition of water-reducing admixture.

[b] Cement required in kilograms per cubic metre for 0.53 water/cement ratio by weight.

[c] Approximate percentage of fine aggregate of total aggregate by absolute volume.

[d] Recommended average total percentage of entrained air required for frost resistance from Table 5.3.3 of ACI Recommended Practice for Selecting Proportions for Normal and Heavy Weight Concrete (ACI 211.1).

practice from the Strategic Highway Research Program [32] will result in the most densely packed system and minimum void content for the aggregates under consideration.

Coarse Aggregate

The importance of grading has long been recognized. The original issue of ASTM C 33 in 1921 had very few requirements other than grading. The coarse aggregate was not designated by size number but rather by maximum size [33]. This is still the "starting point" of selecting the coarse aggregate "size" to be used and is dependent on thickness of section, spacing of reinforcement, availability, economics, and placement procedures (aggregates greater than 63 mm (2.5 in.) become more difficult to pump).

The terms "maximum size" and "nominal maximum size" should be understood. ASTM C 125, Terminology Relating to Concrete and Concrete Aggregates, defines maximum size as the smallest sieve opening through which the entire amount of aggregate is required to pass, and nominal maximum size as the smallest sieve opening through which the entire amount of aggregate is permitted to pass.

The importance of maximum size is illustrated in Table 2 that shows the relationship to aggregate size and the amount of water and cement needed [2]. Simply put, the smaller the aggregate size the more surface area is present for a given value and, therefore, the more mortar needed in the mix to surround the aggregate particle. There are limits on both ends where this does not hold true.

For compressive strengths below 21 MPa (3046 psi), the cementitious content of the concrete can be progressively reduced as the maximum aggregate size is increased while maintaining a constant water/cement ratio and strength. At the 28 MPa (4061 psi) level, no cementitious material can be saved by using aggregate above 76 mm (3 in.) in size. As the strength level is increased, smaller maximum aggregate sizes must be used for the most efficient use of cement with 37.5 mm (1.5 in.) for the 35 MPa (5076 psi) level, 19 mm (0.75 in.) for the 42 MPa (6092 psi) level, and 9.5–12.5 mm (0.375–0.5 in.) for strength above the 42 MPa (6092 psi) level [29,34].

In the production of high-strength concrete above 42 MPa (6092 psi), there seems to be no advantage in using maximum aggregate size below 12.5 mm (0.5 in.) [35,36]. A large particle of aggregate has less area for bonding with the mortar than an equal mass of smaller aggregate particles, and it is for this reason that it is not possible to obtain high strengths with large maximum aggregate size [37]. Also, it is not possible to obtain strengths above a practical level of 35 MPa (5076 psi) with the entrained air contents listed in Table 2 [38].

Because it is not practical to produce aggregate that is perfectly separated, ranges are given in grading specifications. There will be some finer material due to breakage, and sometimes some coarser material due to screen wear or the use of screens slightly larger than the specified size. These deviations are not normally significant when compared to the segregation problem mentioned earlier due to poor stockpiling or handling practices. Therefore, proper attention needs to be given to those procedures to avoid problems in the concrete mixture. Obviously, the less handling, the less likely segregation will occur.

Fine Aggregate

Fine aggregate grading has a much greater effect on workability of concrete than does coarse aggregate grading [39]. Along with the water and cement (and, in some cases, other mineral admixtures), the fine aggregate comprises the matrix in which the coarse aggregate resides. This matrix needs to coat the coarse aggregate particles and retain sufficient fluidity for placement purposes. Thus, the fine aggregate cannot be too coarse or harshness, bleeding, and segregation may occur. At the same time, if it is too fine, the additional surface area will require additional water and also may result in segregation. The grading in ASTM C 33 is usually satisfactory, but as previously mentioned, deviations are permitted because certain areas do not have native material containing these sizes.

In addition to the grading requirement, ASTM C 33 also prohibits more than 45 % passing any one sieve and retained on the next consecutive sieve shown in the standard grading. It also limits its FM to between 2.3 and 3.1. Working in conjunction with the fine aggregate grading is its particle shape that will be discussed later.

Regardless of the grading used, consistency is most important. If the grading varies considerably, problems with workability will probably result. Thus, good control calls for a constant check on the grading and computation of the FM. This is particularly true with fine aggregate where changes can occur without it being obvious. The material may well stay within the grading limits, but the FM may vary more than the 0.2 allowed in ASTM C 33. Changes have been noted over a range as much as 1.0 in FM in a day's production [40].

For the high cement contents used in the production of high-strength concrete, it has been found that a coarse sand with an FM of around 3.0 produced the best workability and highest compressive strength [35]. In general, manufactured sands require more fines than natural sands for equal workability [22].

Concrete for pumping must be very workable, with high fine aggregate contents. The fine aggregate must be well-graded and somewhat on the fine side, with 15 to 30 % passing the 0.3-mm (No. 50) sieve.

The amount passing the 0.3-mm (No. 50) and 0.15-mm (No. 100) sieves have a greater influence on workability, surface texture, and bleeding of concrete. The lower limit of 10 % in ASTM C 33 may be satisfactory where placing conditions are easy or where mechanical finishing is used such as in pavements. However, where hand finishing is used and a smooth texture is desired, at least 15 % should be passing the 0.3-mm (No. 50) sieve and a minimum of 3 % passing the 0.15-mm (No. 100) sieve [39].

Grading Effect on Air Entrainment

Table 2 shows the reduction in fine aggregate percentages that may be realized through the use of air entrainment in concrete. In addition to a reduction in the amount of fine aggregate that may be accomplished for a given set of materials and grading, it is sometimes possible to use a coarser or finer grading, or a jump grading, and obtain concrete of satisfactory workability with the proper amounts of entrained air.

Fine aggregate particles passing the 0.6-mm (No. 30) to 0.15-mm (No. 100) sieves entrain more air than either the finer or coarser particles. Therefore, fluctuations in these sieve sizes can affect the air entrainment of the concrete. Significant amounts passing the 0.15-mm (No. 100) sieve will cause a significant reduction in air content [41].

Manufactured Fine Aggregate Research

A substantial amount of research has been conducted in recent years to evaluate alternative gradings of manufactured fine aggregate for use in concrete mixtures [42]. This research has shown that increased levels of minus 75-μm (No. 200) particles (up to 18 %) can benefit concrete mixture rheology and workability without compromising concrete properties. Field trials utilizing high fines manufactured fine aggregate in concrete pavement sections have shown good potential for successful use of these materials [43]. However, factors such as appropriate admixture type and dosage to offset increased water demand, and potential for increased shrinkage continue to be researched. Additionally, proper characterization methods to insure that the fine particles primarily are composed of dust of fracture without deleterious clay mineral components are being evaluated. The ASTM subcommittee with jurisdiction over C 33 currently is reviewing these research results and considering adoption of alternative manufactured fine aggregate grading specifications.

Specifications

As noted throughout this discussion, ASTM C 33 is the document used to specify grading by most architects and engineers. However, other agencies such as the American Railway Association and the various State transportation departments may have their own grading specifications. Many of these specifications are very similar to ASTM C 33 but may call a particular grading by a different size number.

Shape

Definition

The concept of particle shape incorporates three geometrical ideas; namely, sphericity, roundness, and form, that are distinct and separately definable properties in the abstract or mathematical sense. They may be linked properties in the geological sense, in that a process that affects the expression of one property may, concurrently, promote or inhibit development of the others.

Sphericity is a measure of how nearly equal are the three axes or dimensions of a particle, based on the degree to which the volume of a particle fills the volume of a circumscribed sphere whose diameter is the maximum dimension of the particle. Roundness is a measure of the sharpness of the edges and corners of a fragment, or the degree to which the contour of a particle fits the curvature of the largest sphere that can be contained within the particle.

Sphericity and roundness can be visualized conveniently, but not defined rigorously, by the analogy of an irregular solid within which is a sphere of the largest possible size and around which is a sphere of the smallest possible size. The spheres may or may not be concentric. The congruence of the particle boundary to the inner sphere is an indication of roundness and its coincidence to the outer sphere, the sphericity. When the spheres coincide, the particle itself is a sphere with both a roundness and sphericity of 1—the maximum value.

The form of Folk [44] or the alternative shape factor of Ashenbrenner [45] is a measure of the relationship between the three dimensions of a particle based on ratios between the proportions of the long, medium, and short axes of the particle, or the smallest circumscribing ellipsoid. Form or shape factor distinguishes between particles of the same numerical sphericity but of different axial proportions. A more thorough discussion of how these dimensions interrelate is provided by Ozol [46].

The shape of natural coarse and fine aggregates is influenced by geologic factors such as transport mechanisms and depositional environments. The shape of manufactured coarse and fine aggregates is influenced by the natural breakage of the particle during mining (blasting) operations, by the type of crushing equipment used, or by the processing techniques such as the speed of feed to the crusher. A desirable shape is one that is round or a near perfect cube. Poorly shaped aggregate is more difficult to define and depends largely on the specification being followed.

ASTM D 4791, Test Method for Flat Particles, Elongated Particles, or Flat and Elongated Particles in Coarse Aggregate, provides definitions for particle shape. A flat particle is one in which the width (intermediate dimension) exceeds the thickness (minimum dimension) by a specified ratio. An elongated particle is one in which the length (maximum dimension) exceeds the width by a specified ratio. A flat and elongated particle is one in which the length exceeds the thickness by a specified ratio. Specifications typically use limiting ratios of either 3:1 or 5:1 to describe undesirable shapes.

Test Method

There are no ASTM test methods at the present time for determining particle shape directly. An indirect method that establishes a particle index is ASTM D 3398, Test Method for Index of Aggregate Particle Shape and Texture. This method and D 4791 are under the jurisdiction of ASTM Committee D 4 and are used primarily on aggregates for asphaltic concrete.

ASTM C 1252, Test Method for Uncompacted Void Content of Fine Aggregate (as Influenced by Particle Shape, Surface Texture, and Grading), provides an indirect method of measuring particle shape of fine aggregate. It consists of dropping the test material from a prescribed height into a vessel of known volume, and comparing the resultant mass with the theoretical solid mass of material filling the vessel's volume to calculate the uncompacted void content. The test may be conducted using a standard grading, individual size fractions, or the as-received grading.

With regard to coarse aggregate, ASTM D 4791 may be used. Some state transportation agencies follow a similar method used by the U.S. Army Corps of Engineers [47]. These methods require that the technician measure each individual particle in a set of proportional calipers set up according to the specification on a 2:1, 3:1, or 5:1 limiting ratio basis. While the method is effective in determining the mass or number of particles above the prescribed ratio, the method becomes tedious and time-consuming and can lead to carelessness or lethargy on the part of the operator. There are other methods that have been developed (see Ozol [46]); however, the ones described herein are those in more common use.

Considerable research has been conducted in recent years on digital imaging techniques for characterization of aggregate shape and surface texture [48–52]. Relationships between these characterization techniques and existing ASTM methods have been investigated, and current research is focusing on correlations of these improved techniques with performance of aggregates in concrete mixtures. Additionally, research into effects of aggregate shape and texture on concrete mixtures properties is being conducted using X-ray tomography and sophisticated computer simulation techniques [53].

Significance

The shape of the aggregate particle influences the fresh and hardened concrete. If all particles were spheres or cubes, we would have an ideal shape factor. As the particles become more elongated or flatter, the ease with which they move in the mixing and handling processes become increasingly more difficult. This is especially critical in pumped concrete where workability is of extreme importance.

If the number of poorly shaped particles is not too great, the workability problems may be overcome with the use of water-reducing admixtures. The question then becomes what quantity is considered too great? ASTM C 33 does not have any requirements with regard to this attribute. Some other specifying agencies such as State transportation departments do have limits generally ranging between 8 and 20 % maximum allowable on a 3:1 ratio.

One of the problems related to particle shape is the particle shape changing after trial mixtures are made or during the course of the project. The need for increased workability is solved many times on the job by the addition of unwanted and unauthorized water. Therefore, when workability problems occur, aggregate particle shape should be one of the areas to be investigated.

In the hardened concrete, the particle orientation may influence compressive and flexural strengths, elasticity, and the distribution of stresses. For instance, if such a thin, flat particle was oriented in the hardened concrete where outside stresses were introduced on that particle, the strength of the concrete might be significantly lowered because of the lack of this thin particle's ability to carry the stress. Thus, the importance of trying to find a source of relatively round to cubical-shaped aggregates is vital.

As previously mentioned, should the source be marginal with respect to this property, the use of designing techniques, including the use of admixtures, can still produce a quality end product. This may become necessary because in some areas locating good naturally occurring aggregates is becoming more difficult.

Rounded gravels that were rounded by eons of rolling to lower elevations along stream or river banks will not be replaced in the foreseeable future. The rapid development and, therefore, large use of good gravel aggregate in the past 50 years, coupled with stricter zoning laws, noise and dust pollution laws, stricter permit requirements, and use of land, particularly in urban areas, for more economic gain than quarrying, have joined together to limit the use of what is still left of the natural deposits. Therefore, the use of crushed stone and, more critically, the use of synthetic or reclaimed material as aggregates will become more prevalent, and the use of less than ideal-shaped material will increase [54].

Sphericity increases with size, although the relationship in naturally occurring materials is less pronounced than with crushed materials [46]. In a study of the shape of various, as-supplied coarse aggregates (two gravels and three crushed stones), Conway [55] found that the sphericity in all cases increased with increasing sieve size [46]. Similarly, coarser grains round more easily than fine ones and roundness increases with size [56].

The form and sphericity of aggregates are primarily the result of the degree of anisotropy of the material and the original shape of the particle, that is, the shape prior to the outside influence of blasting, handling, sizing, and transporting [46]. In the case of gravels, the original particle shape is controlled by larger-scale structural features of the bed rock, such as joints, fractures, faults, and bedding planes, that were then exploited by glacial action, stream action, and mechanical (frost) and chemical weathering. In the case of natural sands, the original particle shape, that is, its shape prior to transportation, was probably most strongly controlled by its mineral grain shape in the parent material.

With crushed stone, the analogous "original" particle shape is that produced by blasting of the bedrock. Those shapes are a complex function of the influence of the large-scale structural features of the bedrock, but additionally, are a function of the size and frequency of the cracks initiated by the action of shock waves on the smaller-scale flaws and discontinuities of the rock on the order of magnitude of the grain sizes. Most of the shot rock requires further size reduction by crushing, and the final shapes of the particles are influenced strongly by preferred orientations of any sort contributing to anisotropy. Rocks with bedding, schistosity, shale partings, mineral cleavage, etc., may yield particles more elongated or flatter than equidimensional. Equigranular rocks with no preferred orientations tend to yield more cubical particles on crushing. The characteristics of crushing equipment also influence the particle shape of the rock being crushed. Generally, the greater the reduction ratio, the more flat or elongated the product—slightly more so if the machine is of the compression (jaw, gyrator, or cone crusher) type. That effect is less pronounced with impact- or impeller-type machines, which would produce more nearly cubical particles at equivalent throughputs.

Rounding of sand and gravel particles is chiefly the result of those geologic factors that were of secondary importance in contributing to their form and sphericity; that is, the abrasion, attrition, chipping, rubbing, and (possibly) solution incident to their transportation and to the site of deposition. The roundability of particular mineral rock fragments depends directly on their hardness and toughness and inversely on the presence of cleavage or cracks, which tend to induce fracturing, negating what rounding had been accomplished [46].

The shape then becomes an important factor with regard to all aspects of the concrete and, while universally recognized methods of measurement are not at hand, the characteristic should be taken into account in the concrete design. Undoubtedly, technological advances in imaging methods and computer simulations will lead to improved aggregate characterization and better understanding of their effects on concrete mixtures in the future.

Specification

ASTM C 33 does not have any limitation on particle shape for either fine or coarse aggregates. As previously mentioned, ASTM C 1252 has been developed and an indirect test method for particle shape of fine aggregate, and ASTM D 4791 for coarse aggregate. However, these methods primarily are utilized in asphaltic concrete specifications, and no limitations are under consideration for fine or coarse aggregate particle shape within the ASTM C 33 specification.

Surface Texture

Definition

The surface texture of an aggregate particle is the degree to which the surface may be defined relative to arbitrary numbers as being rough or smooth (loosely referring to the height of the asperities) or coarse grained or fine grained (loosely referring to the spacing of the grains). Additional elements of surface texture, not easily incorporated into a concise comprehensive definition, include the lateral and vertical irregularity of the roughness; that is, the statistics of the height distribution of the population of asperities and their frequency of occurrence over an area, as well as their morphology.

Two independent geometric properties are the bias component of surface texture: (1) the degree of the surface relief, also called roughness or rugosity; and (2) the amount of surface area per unit of dimensional or projected area. The latter property, although it is the ratio of areas, has been defined by Wenzel [57,58] as the roughness factor, $R = A/a$, where A is the true (real) surface area, and a is the apparent or projected area. A rough surface (in the sense of degree of relief) does not necessarily have more surface area than a smooth surface of equivalent dimensions—it may or may not. The two properties are not related functionally but depend on the relative amplitude and frequency of the asperities on the surfaces being compared [46]. The criterion by which one surface is designated rougher than another, in terms of their reliefs, is variously defined depending on the intended use of the information, but almost invariably involves some measure of the deviation of a profile of the surface from a hypothetical reference surface [46].

Test Method

There are several methods for measuring surface texture [59]. These are research methods. There are no laboratory operational procedures, except that the tests measuring shape by one of the flow tests are indirectly influenced by the particle texture. For example, a rough-textured particle will rub against other similar particles and not flow as readily as smooth particles. Therefore, any flow test using time as a part of the measurement will be affected by the relative textures.

As mentioned previously under the discussion of particle shape, advances in digital imaging, X-ray tomography and computer simulation techniques also are being utilized for characterization of aggregate surface textures and their effects on concrete properties.

Significance

In work done by Kaplan [60], it was concluded that surface texture has no appreciable effect on the workability of concrete. It is obvious that it certainly does not affect the workability of the concrete to the degree that grading and particle shape do. However, other work by Kaplan [61] found that among the factors of angularity, texture, flakiness, and elongation indexes, the texture had the largest effect on compressive strength. This probably can be attributed to the extra mechanical interlock and increased surface area available for bond in the rough texture.

Patten [62] investigated the relative contributions of adhesion and “keying” (or mechanical interlock) by compressive and tensile strength measurements of concrete at five different ages in which the same coarse aggregate had been used in both a “bondless” (or surface-treated) and an untreated condition. The object was to eliminate adhesive bonds without affecting the mechanical interlock (or physical keying) between the aggregate and the mortar. The coarse aggregate was coated with a mold release agent. The elimination of adhesion between the mortar and the coarse aggregate reduced compressive strength by an average of about 23 %. Compared to the control, the strength of the bondless concrete decreased with age from 19 % difference (or reduction) at seven days to 27 % difference at six months. Tensile strength was less affected, ranging from 6 to 28 % difference, with an average reduction of about 17 %; but in this case, the strength of the bondless concrete increased with age as a percent of the control. At six months, in the high-strength series, the splitting tensile strength of the concrete with the bondless aggregate was 92 % of the strength of the concrete with the untreated aggregate; whereas at seven days, it was 79 %. Investigations along similar lines have been conducted by Darwin and Slate [63] and others [64,65].

Studies focusing further on the components of the adhesive force making up the percent contribution of adhesion to bond strength have endeavored to test the interface in as pure a state of tension as possible to avoid mechanical contributions. Various specimen configurations and techniques for measuring aggregate-cement bond strength have been reviewed by Alexander et al. [66]. Under conditions of tension, the total adhesive force is the product of: (1) the specific adhesion, that is, the strength of the adhesive force per unit area of surface from whatever physicochemical mechanism it derives; and (2) the amount of surface area available for bonding over which the adhesive force acts. Different rock types have been observed to have different tensile bond strengths to cement paste [67–69]. A possible explanation is that the bond is chemical and its specific adhesion differs significantly according to the particular chemistry of the mineral surface [70,71]. Alternative possibilities are that (1) the bond is chemical but its specific adhesion on the smallest unit area basis does not differ greatly between minerals, or (2) the bond is physical [72], deriving from the same sorts of forces that hold materials together in general. From either of these possibilities, an alternative hypothesis to explain differences in bond strength between different rock types is that surfaces of different lithologies have different roughness factors [46] and, therefore, present different true surface areas available for bonding [73].

Coatings

Coatings are defined as any material adhering to the particle whether it be foreign or fine material of the same mineralogy as the parent particle. Some coatings are a result of mining or processing procedures or may be due to a natural weathering of the rock. In any case, coatings are for the most part undesirable because they interfere with the bond between the aggregate and cement paste.

One of the more common forms of coatings occurs in stockpiles where the material may be stored in a wet condition and dust from passing traffic, or wind-borne dust coats the top layer. Another common problem can be the carelessness of an operator loading the material (usually with a front-end loader) into trucks for transportation to the concrete holding bins. If the operator is not careful, the loader scoop gets too low and part of the soil on which the stockpile rests is picked up. This, in turn, contaminates the aggregate and, in the case of clay or plastic soils, adheres to the material.

Fortunately, washing the aggregate usually removes coatings and, in the case of gravels that have been washed during the grading process, only careless contamination after processing presents a problem. In the case of crushed stone that is not generally washed, any coatings are usually the dust resulting from fracture and may be eliminated during the processing. If the coating is something that is tightly adhering, washing or scrubbing may be necessary. Most crushed-stone plants now use a dust suppressant for ecological reasons. Therefore, precautions should be taken so that this material does not increase the chances of contamination.

Soluble coatings also present a problem because they become dissolved in the matrix with undesirable results. One common coating is iron sulfide that stains the concrete. Chlorides and sulfates can also cause staining, efflorescence, and premature corrosion of imbedded reinforcing steel.

ASTM C 33 contains limits for the amount of material finer than the 75-μm (No. 200) sieve for fine and coarse aggregate. This amount is determined by sieving procedures, with more accurate values obtained by wet sieving in accordance with ASTM C 117, Test Method for Materials Finer than the 75-μm (No. 200) Sieve in Mineral Aggregates by Washing.

Specification

ASTM C 33 is silent with regard to requirements for surface texture, probably due to the fact that there is no easy way to measure texture and the fact that when texture reaches the point of interfering with the production of quality concrete other factors will have come into play.

Summary

Grading has a significant effect on concrete mixture proportioning and workability. The gradings listed in ASTM C 33 are generally satisfactory and may be used in several different combinations to achieve desired results. Alternative gradings are permitted by C 33 provided that acceptable concrete performance can be demonstrated. Recent and ongoing research on manufactured fine aggregate may lead to specifications for alternative gradings for these materials in the future. The main concern with regard to aggregate grading is inconsistency and the ability to detect changes, particularly with fine aggregate. The fineness modulus is one way to maintain a check on the fine aggregate grading that is not discernable by casual observation. Segregation of coarse aggregate during the handling process can be a major problem. Choosing sizes that do not range at the extremes of the grading scale will help. Many times, two coarse aggregate sizes are preferable, and can be recombined at the time they are introduced into the mixer or can be added separately, depending on the economics involved. Variations in the grading can change the surface area that is to be coated with mortar and, because this is done during the design and trial mix stages, variations in the grading (surface area) change the amount of optimum mortar and can lead to the addition of unwanted water. Continuous attention to grading is of paramount importance.

Particle shape also has a significant effect on both fresh and hardened concrete. Flat and elongated particles do not “roll” as well as rounded or cubical particles during the mixing process and, therefore, require more water for the same consistency. If the water-cement ratio is to be maintained (and this is usually mandatory), then more cement needs to be used for the same strength, thus becoming less economical. The fine aggregate particle shape is just as important as the coarse aggregate particle shape. A 1 % increase in void content as measured by ASTM C 1252 requires about one additional gallon of water per cubic yard for the same slump, all other things being maintained. In the hardened concrete, the orientation of the coarse particles could affect the stresses in the concrete mass leading to reduced strength. There are no standard ASTM test methods at this time that measure the particle shape of fine aggregate. Therefore, attention must be directed to void content tests. ASTM D 4791 may be used to evaluate flat and elongated particles within coarse aggregate. However, no specifications currently are provided for either fine or coarse aggregate shape within ASTM C 33.

The texture of aggregate particles has no appreciable effect on workability but does affect compressive strength. This is probably because the rough texture presents a better mechanical interlock with the matrix and, with an increased surface area, there is more interface with which the mortar may interact. Unfortunately, a rough texture increases the propensity to retain undesirable coatings. At the same time, unless the coating has a strong adherence, it may loosen during processing or handling. If such adherence is strong, washing or scrubbing may be necessary. Coatings can result as an inherent feature of the parent rock, but more often coatings are a result of carelessness during the stockpiling or handling phases. Coatings can prevent the matrix from adhering to the aggregate particle and may be chemically reactive to the point of staining the concrete. Also, coating fines may simply mix into the matrix, not affecting the bond, but adding fines to the matrix. Therefore, clean, uncoated aggregate is important to ensure a good quality concrete.

Recent advancements in digital imaging, X-ray tomography, and computer simulation techniques should provide improved methods for aggregate shape and texture characterization and greater understanding of their effects on concrete properties in the future.

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Bulk Density, Relative Density (Specific Gravity), Pore Structure, Absorption, and Surface Moisture

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Preface

THIS CHAPTER DISCUSSES THE APPLICATION AND provides some insight into the background of the test procedures for the basic aggregate properties of bulk density, relative density (specific gravity), absorption, and surface moisture. It is substantially a revision of the work of Landgren [1] in *ASTM STP 169C*, with an update of the terminology and the addition of a discussion on the importance of pore structure. The information from the original article by Timms [2] in *ASTM STP 169* and the revised articles by Brink and Timms [3] in *ASTM STP 169A* and by Mullen [4] in *ASTM STP 169B* remains pertinent and is included in the chapter.

Introduction

In a typical concrete mixture, with a cementitious content of 227 kg (500 lb), the aggregate will occupy approximately 80 % of the volume of the mixture. The aggregate is a collection of various sizes of irregularly shaped particles often with rough and/or irregular surfaces. The particles almost always have internal pores that will hold water. Because the particles do not fit together well, spaces are left between them called voids. Even in desert conditions the aggregate probably contains water in the pores of individual particles (absorbed water) and on the particle surfaces (free moisture).

A common procedure for proportioning concrete mixtures is the Absolute Volume Method. In this method the volume (yield) of freshly mixed concrete is equal to the sum of the absolute volumes of the cement, water (exclusive of that in the aggregate), aggregates, admixtures (when applicable), and air. The absolute volume is computed from a materials mass and relative density as follows [5]:

$$\text{Absolute Volume} = \frac{\text{Mass of Material}}{\text{Relative Density of Material} \times \text{Bulk Density of Water}}$$

To determine the proportions of ingredients in a concrete mixture, and prior to batching concrete, the following factors should be known:

1. The volume of voids that will be filled with paste such that the aggregate particles are coated with a paste film.
2. The volume of solids in the mixture.
3. Aggregate relative density, a useful factor in concrete mixture proportioning and control.
4. Absorption, because of its effect upon the volume of mix water added.
5. Aggregate surface moisture, which constitutes part of the mixing water.

Aggregates used in concrete are usually identified by their physical and mechanical properties. These properties are defined by standard specifications and test methods adopted by agencies such as ASTM International, the American Association of State Highway and Transportation Officials, and others.

For most aggregates, the physical properties that are usually identified are relative density, bulk density, absorption, and grading. Grading and bulk density are typically easily determined for almost all aggregates, while the determination of the relative density and absorption can be more difficult. For some porous or lightweight aggregates, the relative density and absorption may not be readily determined because of surface roughness and high porosity. Fortunately, it is possible to arrive at workable concrete mixture proportions for these aggregates through the use of alternative means, such as “relative density factors” [6].

Aggregate physical properties can vary by aggregate source, type, mineralogy, size, grading, shape, and surface texture. Typically these values are relatively stable for a given aggregate, but can vary by ledge, pit location, size, and surface texture for some materials. When these properties change significantly, it will be necessary to make adjustments to the concrete mixture.

The primary variable in most concrete production is the surface moisture content of the aggregate. This value usually is not required until after mixture proportions have been established, since most mixtures are designed with the aggregate in a saturated surface dry condition. Corrections for changes in

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[1] Director–Technical Services/Steel Mill Services Group, Edw. C. Levy Company, Portage, Indiana 46368.

the surface moisture content are made routinely without changing the basic concrete mixture.

Aggregate Shape and Texture

It is generally recognized that aggregate particles come in various sizes that are described for engineering identification purposes by separation on square-opening sieves. It is also recognized that aggregates having the same grading may vary widely in shape, angularity, and surface texture or roughness. The shapes within the grading may vary between rounded, subangular, or angular.

The surface of aggregate particles can vary from smooth to rough and irregular. Mineralogy and porosity of the source rock as well as natural polishing all contribute to the final surface texture. Generally, natural sands or gravels have smooth surfaces. They have been abraded and polished by wind, water, or glacial transportation. Crushed or manufactured aggregates generally have rougher surfaces and greater angularity than natural sands and gravels. Crushed, highly porous particles, such as slag or lightweight aggregates can have extremely rough surfaces. Mather [7], in *ASTM STP 169A*, used the terms sphericity, roundness, smoothness, and roughness as descriptors in discussing shape and surface texture of aggregate particles. Ozol [8] also expanded on the discussion of shape and texture in *ASTM STP 169B*.

The particle shape and texture of the aggregate affect its mobility, that is, the ease with which aggregate particles move past one another when manipulated. Aggregate mobility affects the energy required for compaction either of concrete or of concrete aggregate when making a standard test for aggregate bulk density. Visual evidence of differences in aggregate mobility between particles of different shapes and texture is shown in Fig. 1. It is possible to form a pile of unconfined crushed stone several particles deep while unconfined glass marbles can be piled only one particle deep. The mobility of an aggregate and the ratio of aggregate voids volume to solids volume affects the workability of fresh concrete as well as the proportion of coarse aggregate that may be used in a mixture. Goldbeck and Gray [9], in their landmark *"b/b*$_0$*"* procedure for concrete mixture proportioning, applied packing concepts to concrete mixture proportioning. Their measure of packing density was the dry rodded bulk density of coarse aggregate.

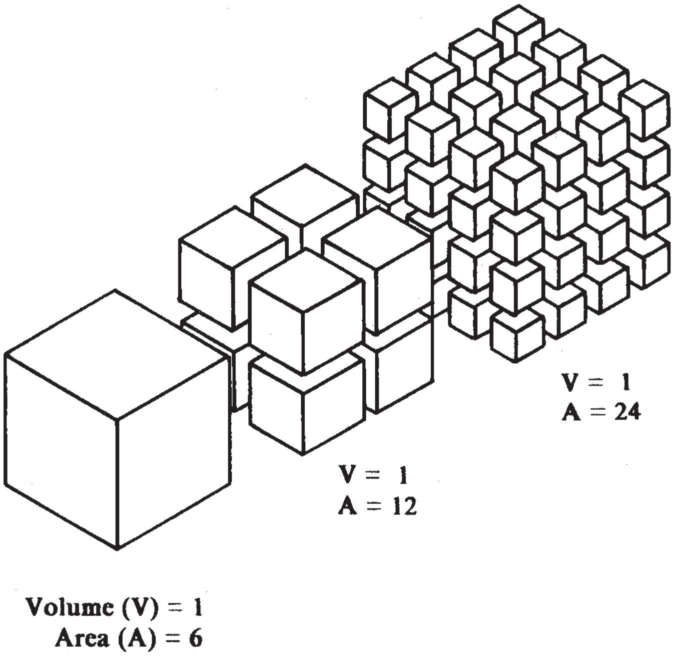

Fig. 2—Surface area versus particle size at equal volumes.

Aggregate packing can also be affected by both particle shape and grading. It is well known that a compacted sample containing a range of particle sizes will result in a void content smaller than one comprised of single-size particles. For example, compacted single-size spheres may have a void volume of less than 30 % of the total aggregate volume, while that percentage may exceed 50 % for compacted single-size angular particles. When graded particles with several sizes are compacted, small particles fit into the voids between larger particles. Classic studies in this area are those by Fuller and Thompson [10] and by Weymouth [11]. The maximum size of the aggregate used in a mixture and its range of individual sizes both affect bulk density significantly and can slightly affect absorption, free or surface moisture, and relative density. The last three properties can change because small particles have a larger surface area per unit volume (Fig. 2), a greater exposure of pore openings to the entrance of water, and short pore lengths. Small, highly porous particles of lightweight aggregate

Fig. 1—Aggregate mobility affected by shape and surface texture.

or slag have less pore volume and higher specific gravities than larger particles of the same aggregate.

Bulk Density and Voids

Bulk density and measures of bulk products, such as grain or aggregates, have been with us almost since recorded history. Bulk product particles do not fit together perfectly with the result being that voids exist between the particles. The shape of the particles affects the volume of voids, the distribution of the particles, compaction effort, and the orientation or packing arrangement of the particles as discussed previously. For certain aggregate packings, bulk density and voids are inversely related; when one increases, the other decreases.

Aggregate bulk density is defined by Brink and Timms [3] as "the mass of a unit volume of representative particles." Methods to determine bulk density and void content are given in ASTM Test Method for Bulk Density and Voids in Aggregate (C 29). That standard defines void content as a percentage relating the ratio of void volume to the volume of a bulk density-measuring container just filled with the granular material.

ASTM C 29 provides for the determination of bulk density of aggregates with a maximum size of 125 mm (5 in.) and smaller. The volume of the required measuring container decreases with aggregate size, with the minimum measure size being 100 L (3 ½ ft^3) for a 125 mm (5 in.) maximum size coarse aggregate, 14 L (½ ft^3) for a 37.5 mm (1½ in.) maximum size coarse aggregate, and 2.8 L (⅒ ft^3) for fine aggregate with a maximum size of less than 12.5 mm (½ in.).

The test method outlines procedures for compacted and loose bulk density determinations. The compact bulk density is determined by the rodding (as described in the next paragraph) for aggregates having a nominal maximum size of 37.5 mm (1 ½ in.) or less, or by jigging for aggregates having a nominal maximum size greater than 37.5 mm (1 ½ in.) and not exceeding 125 mm (5 in.). Whether the bulk density is compacted or loose, the aggregate is dried to essentially constant mass at 110 ± 5°C (230 ± 9°F) prior to testing.

Compaction usually is accomplished by rodding aggregates that are 37.5 mm (1 ½ in.) maximum size and smaller, while aggregates larger than that cannot be compacted effectively by rodding, hence "jigging" compaction of these aggregates is required. Compaction is accomplished by filling the measure one-third full and then leveling the surface with the fingers. The layer of aggregate is rodded with 25 strokes of the tamping rod evenly distributed over the surface. The measure is then filled to two-thirds full and again leveled and rodded as above. Finally the measure is filled to overflowing and rodded again in the manner previously mentioned. The surface of the aggregate is then leveled with the fingers or a straightedge in such a way that any slight projections of the larger pieces of the coarse aggregate approximately balance the larger voids in the surface below the surface of the measure. "Jigging compaction" follows the same procedure for filling the mold except that dropping or agitating the mold to allow the particles to arrange themselves in a densely compacted condition replaces the rodding.

Weak, friable lightweight aggregate that may be broken by rodding can be tested using the "Shoveling Procedure." The bulk density measure is filled to overflowing by gently shoveling the material, by means of a shovel or scoop, into a standard measure to obtain a "dry-loose bulk density." The top surface is leveled using a straightedge, or the fingers, in such a way as to balance projections and depressions in the upper surface.

The bulk density, either "dry rodded" or "loose," is directly useful in proportioning concrete mixtures where the b/b_0 method [9] or its variations [12,13] are used. For a given source of normal-weight aggregate, changes in bulk density indicate changes in angularity, and/or grading. Bulk density changes in lightweight aggregate may signal changes in aggregate relative density, hence they are particularly useful in showing the need for possible corrections to lightweight aggregate concrete mixtures. Bulk density measurements are also essential for calculations of grout quantities required for pre-placed aggregate concrete where work must be completely underground, underwater, or obscured from visual control of grout level [4].

Aggregates are sometimes classified in terms of their bulk density into categories such as nonstructural or insulating lightweight, structural lightweight, air-cooled slag, normal weight, and heavyweight. Table 1 contains such a classification.

Aggregate void contents are calculated with equations given in ASTM C 29. For aggregate of a given relative density, voids will vary inversely as the bulk density.

Some rules of thumb relate aggregate shape, grading, and compaction as follows:

1. Rounded particles pack more closely and have fewer voids than angular particles.
2. Graded aggregates pack more densely than one-sized aggregates because small particles fill the voids between larger particles.
3. Voids will decrease with compaction effort, due to particle breakdown or consolidation, until equilibrium is achieved.

TABLE 1—Bulk Density Classification for Aggregates

Aggregate Classification	Relative density Range, kg/m^3 (lb/ft^3)	Compaction Mode	Bulk density Source
Insulating	96 to196 (6 to 12)	dry loose	ASTM C 332[a]
Lightweight for masonry	880 to 1120 (55 to 70)	dry loose	ASTM C 331[a]
Lightweight for concrete	880 to 1120 (55 to 70)	dry loose	ASTM C 330[a]
Air cooled slag	>1120 (>70)	compacted	ASTM C 33[a]
Normal weight	1200 to 1760 (75 to 110)	compacted	PCA[b], ASTM C 29[c]
Heavyweight	1760 to 4640 (110 to 290)	compacted	PCA[b]

[a] ASTM Specification for Lightweight Aggregates for Insulating Concrete (C 332); ASTM Specification for Lightweight Aggregates for Concrete Masonry Units (C 331); ASTM Specification for Lightweight Aggregates for Structural Concrete (C 330); ASTM Specification for Concrete Aggregates (C 33), and ASTM C 29.

[b] *Design and Control of Concrete Mixtures,* Portland Cement Association, Skokie, IL, July 1968.

[c] Inferred from balance capacity required by ASTM C 29.

The bulk density procedures described here are laboratory methods for determining aggregate properties useful in concrete proportioning and control. Care should be exercised when applying laboratory dry bulk density to aggregate volumes elsewhere; for example, when calculating volumes of aggregate stockpiles. Aggregate stockpiles are usually wet and not uniformly compacted. Dampness causes masses of aggregate to "bulk" or expand to a volume larger than it would be if it were in a dry condition. The surface tension of water in the damp aggregate binds individual particles together, making it more difficult to compact or consolidate the aggregate than it would if the aggregate particles were dry. Conversely, when aggregate stockpiles are dry they can tend to be prone to segregation.

Aggregate Relative Density (Specific Gravity)

The terms relative density and density describe the relationship between the mass and volume of a substance. Because ASTM C 127 and C 128 use the term "relative density" to express the ratio of the mass of an aggregate to the mass of an equal volume of water, relative density will be used here.

The relative density for coarse aggregate, ASTM C 127, is determined as follows: (1) Dry the sample to constant mass. (2) Cool the sample in air at room temperature for 1 to 3 hours or until the aggregate has cooled to a temperature that is comfortable to handle (approximately 50°C). It is important for the aggregate to have cooled since this can affect the results. (3) Immerse the aggregate in water for 24 h ± 4 h. (4) Remove the sample and roll it in a large absorbent cloth until all visible films of water are removed. The importance of this determination will be further discussed in the water absorption section. (5) Determine the mass of the sample (this is the SSD condition). (6) Immediately place the SSD test sample in a container and determine the mass of the sample in water. The apparent loss of mass of the immersed sample is equal to the mass of the volume of water displaced by the aggregate sample.

Depending upon the procedure used the relative density, a dimensionless quantity, can be expressed as oven-dry (OD), saturated-surface-dry (SSD), or apparent relative density in both C 127 and C 128. The ASTM C 127 relative density definitions are as follows:

1. Relative density (OD) is the ratio of the density (OD) of the aggregate to the density of distilled water at a stated temperature. Inaccuracies in the measurement of aggregate absorption affect the value of the calculated relative density (OD).
2. Relative density (SSD) is the ratio of the density (SSD) (saturated-surface dry) of the aggregate to the density of distilled water at a stated temperature. Inaccuracies in the measurement of aggregate absorption affect the value of the calculated aggregate bulk relative density (SSD).
3. Apparent relative density is the ratio of the apparent density of the aggregate to the density of distilled water at a stated temperature. The apparent relative density can be determined absolutely and is not affected by inaccuracies in measurement of aggregate absorption.

An example of the formula for calculating the relative density (OD) from ASTM C 127 follows:

$$\text{Relative Density (OD)} = A / (B - C)$$

where

A = Mass of oven-dry sample in air, g,
B = Mass saturated-surface-dry sample in air, g,
C = Apparent mass of saturated sample in water, g.

The illustrations in Fig. 3 illustrate the "mass in air and mass in water" procedure used by ASTM C 127 to determine relative density of coarse aggregate. The difference between these two masses is the buoyancy afforded the aggregate by water equal in volume to that of the aggregate.

In ASTM C 128 the Saturated Surface Dry (SSD) condition can be determined by use of the Cone Test (see Fig. 4). This procedure requires that (1) the specimen be dried to a constant mass, (2) immersed in water for 24 h, (3) dried back to a surface dry condition, (4) placed into a conical mold, (5) the mold removed, and (6) when the aggregate slumps slightly it indicates that it has reached a surface dry condition. The final step may require you to make moisture adjustments until the

Fig. 3—ASTM C 127.

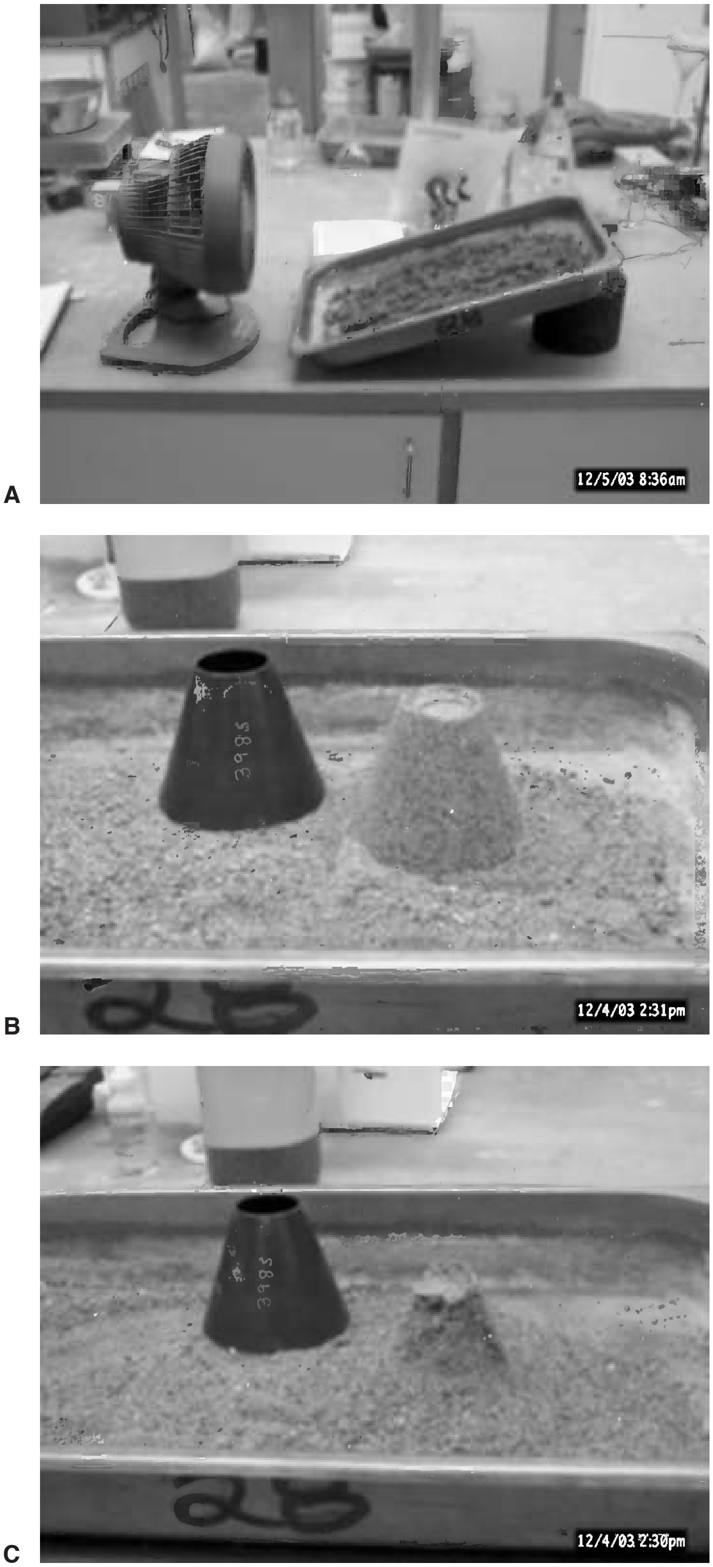

Fig. 4—ASTM C 128 determining SSD condition.

Fig. 5—Relative density; ASTM C 128 test alternatives.

appropriate slump is observed, and this adjustment is more difficult when testing manufactured sands since angularity affects mobility as discussed earlier. This material is then used in the fixed volume portion of the test. A pycnometer, or a Le Chatelier flask (Fig. 5), for an approximately 55 g test sample, may be used. Figure 6 depicts the pycnometer (fixed volume) procedure in ASTM C 128 for the determination of the relative density of the fine aggregate. The procedure determines the difference between (a) the mass of the aggregate plus the pycnometer filled to the mark only with water, and (b) the mass of the pycnometer containing aggregate and filled to the mark with water. That difference is the weight of water displaced by the aggregate. The formula for determining the relative density (OD) using the Gravimetric Procedure in C 128 is:

$$\text{Relative Density (OD)} = A / (B + S - C)$$

where

A = Mass of oven-dry sample, g,
B = Mass of pycnometer filled with water, to calibration mark, g,
C = Mass of pycnometer filled with sample and water to calibration mark, g,
S = Mass of the saturated surface-dry specimen, g.

Both C 127 and C 128 yield exactly the same information, that is, the mass of water displaced by the aggregate. As noted ASTM C 127 and C 128 define three relative density values.

Aggregate relative density values measured after 24 h of water absorption are significant properties of the aggregate. Aggregate with a moisture content different than found after 24 h of soaking will have a different relative density. The presence of water-filled pores inside aggregate particles (Fig. 7) complicates relative density determination. Equations relating specific gravities with absorption are given in Appendix to ASTM C 127 and C 128.

Pores and Pore Distribution

While ASTM C 29 Test Method for Bulk Density and Voids in Aggregate allows a concrete mix designer to assess the space between the aggregate particles, one of the most important features of an aggregate particle is the pore, or void, space within the individual particles. Virtually all aggregates contain pores; their size, number and distribution influence water absorption, relative density, and freeze-thaw durability.

The Aggregate Handbook [14] defines pore structure as "the size, volume, and shape of the void spaces within an aggregate particle. Pores can be either impermeable (isolated, enclosed cavities) or permeable (interconnected and extending to the surface of the particle). Continuous and interconnected,

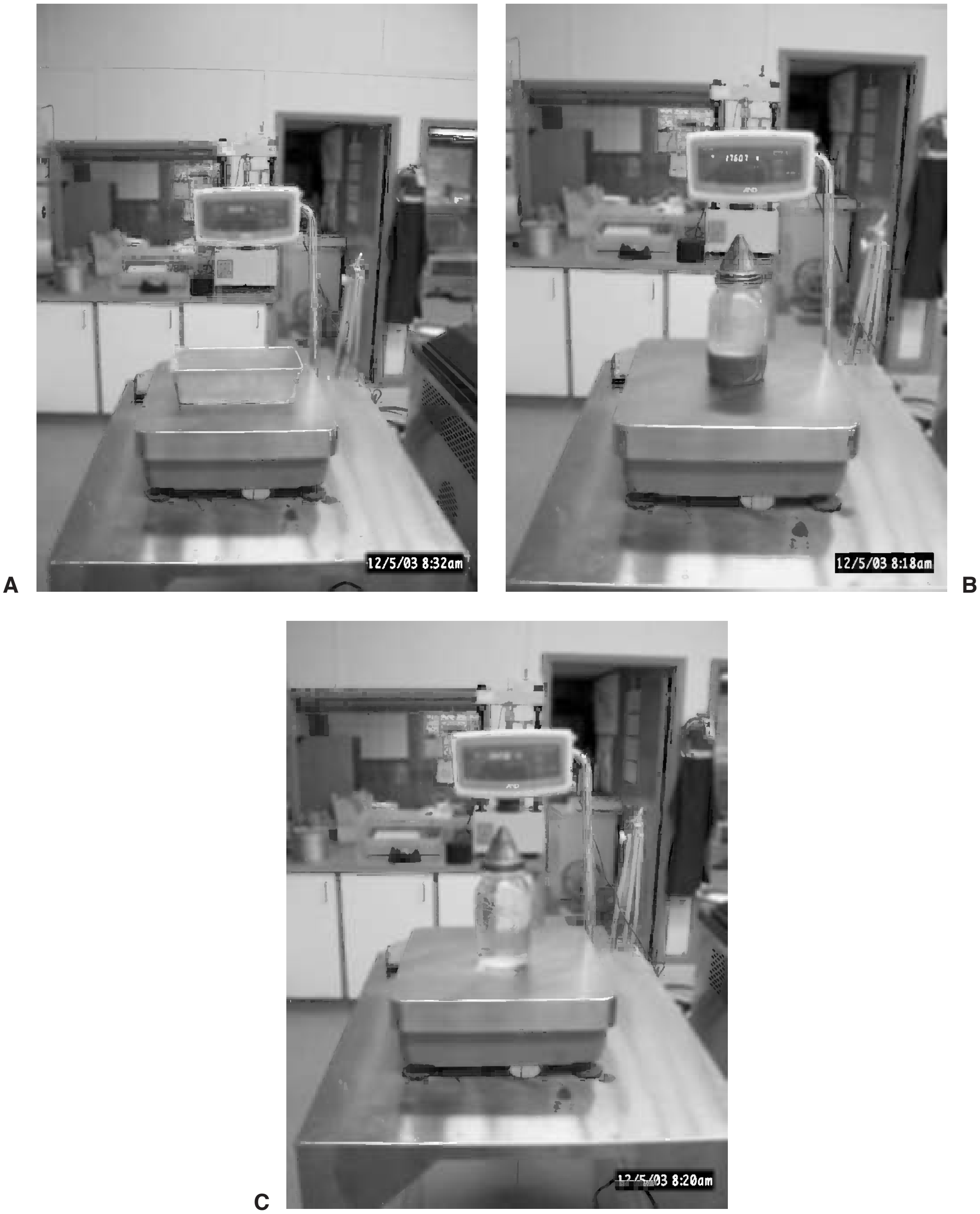

Fig. 6—ASTM C 128, pycnometer method.

large permeable pores can form large passageways through the aggregate. Impermeable pores typically influence the thermal properties, elastic modulii, etc., of the aggregate and are not of concern in this section.

Winslow in *ASTM STP 169C* [15] notes "there may be some ambiguity between pore space immediately adjacent to the surface and major depressions or irregularities in the surface." A functional differentiation might be to consider the pore space of an aggregate to be that non-solid volume that will not be filled with cement hydration products when the aggregate is used in portland cement concrete. In aggregates where these irregularities become an issue, the determination of the saturated surface dry condition also becomes a problem (as discussed later).

The influence of the pores on freezing and thawing is determined by their size and distribution. Pores may range in size from molecules (angstroms) to holes visible to the unaided eye (micrometres and millimetres) [16]. Some aggregates have pores that are too small and that do not allow water to freely move. The water in these pores will freeze and cause damage. When the pores are large, such as in slag or lightweight aggregate, they typically do not become highly saturated, allowing any hydraulic pressure to easily dissipate. Mercury intrusion techniques have been used to measure pore distribution ranging from 0.003 to 500 micrometres (0.5 mm).

Aggregate Water Absorption

ASTM C 127 defines absorption as the increase in the mass of aggregate due to water in the pores of the material, but not including water adhering to the outside surface of the particles,

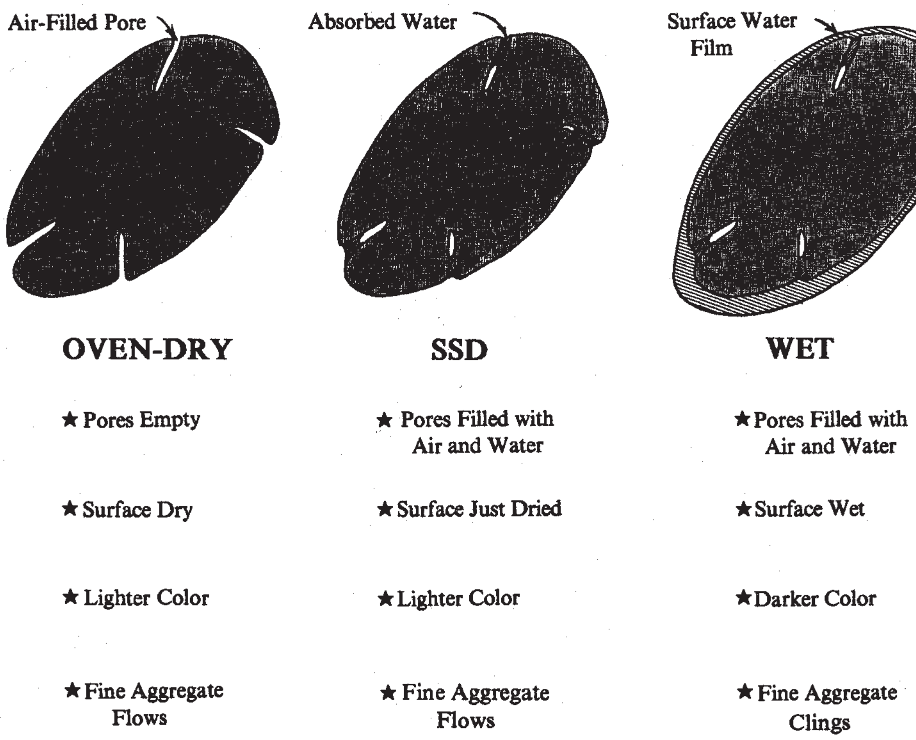

Fig. 7—Aggregate moisture conditions.

expressed as a percentage of the dry mass of the aggregate. Absorption is determined after soaking an initially dry aggregate for a 24-h period in water. Absorption test procedures are given in ASTM Test Method for Relative Density and Absorption of Coarse Aggregate (C 127) or ASTM Test Method for Relative Density and Absorption of Fine Aggregate (C 128). An ASTM absorption value may be regarded as an aggregate property that is a function of aggregate porosity and pore size. Absorption and other factors that are a function of aggregate porosity are critical in determining freeze-thaw durability of an aggregate and of concrete made with the aggregate.

The mass of water absorbed by the aggregate is the difference between the mass of the oven-dry aggregate and the "saturated surface dry" (SSD) mass of the aggregate after it has been soaked in water for 24 h. ASTM C 127 requires that saturated coarse aggregate surface dryness be accomplished by wiping the aggregate particles with an absorbent cloth. In the ASTM C 128 method, water-saturated fine aggregate is reduced to surface dryness by air-drying the aggregate until interparticle cohesion produced by surface moisture is lost.

Figure 7 shows factors differentiating three aggregate moisture conditions: oven-dry, SSD, and wet (containing surface moisture). The ASTM absorption testing involves oven-dry and SSD conditions only. The primary difference between these two moisture states is a pore-filling combination of water and entrapped air. The moisture condition of aggregates in field stockpiles usually brackets that of the SSD state. Typically stockpiled aggregates are wet; a few are "air-dry" and contain less pore moisture than the SSD condition.

The mass of absorbed water in the pores of an aggregate will generally increase with the time of immersion, of the initially dry aggregate, in water. During the initial phases of absorption, the introduction of water into aggregate pores compresses air originally occupying the pore space, forcing air bubbles out from the larger pore channels. These processes occur quickly, and initial absorption rates are high. Eventually, air trapped deep inside aggregate particles can only be displaced by dissolving that air in pore water and transporting it out of the aggregate by slow diffusion through the pore water. This accounts for the rapid initial absorption of pore water and the slow, but measurable absorption long after the dry aggregate is immersed in water.

In normal concrete operations, the amount of water absorbed in the pores of stockpiled aggregates might approximate the 24-h absorption determined by standard ASTM methods for measuring absorption. However, some active fine aggregate stockpiles can contain a significantly higher percentage of absorbed water than that determined by the ASTM procedure because the stockpiled aggregate usually has been kept wet for long periods of time before it is incorporated in the concrete mix. Alternately coarse aggregate stockpiles are typically less than SSD, unless there has been recent rainfall or the piles have been watered.

The primary difficulty in determining absorption in some aggregate, those with irregular surfaces, is the determination of the "surface dry" condition. This difficulty and alternative methods of measuring concrete aggregate absorption that have been proposed will be discussed later.

Aggregate Surface Moisture

Aggregate surface moisture is defined as all moisture in the aggregate except that absorbed inside the pores of individual aggregate particles. Aggregate surface moisture is a critical factor in concrete operations. Surface moisture on batched concrete aggregates is part of the water in the concrete mixture. Aggregate surface moisture compensation is necessary for proper mixture control. Concrete fine aggregate generally contains the greatest quantity of surface moisture, since the voids between particles are small and capable of retaining large percentages of moisture.

In concrete plants, sand surface moisture is usually monitored by moisture meters installed on batching equipment. Proper placement and maintenance of moisture meter equipment permits accurate, continuous measurement of aggregate surface moisture, provided other plant operations, such as bin filling and stockpiling, are done properly. One critical feature in plant management is to utilize sand stocks that have been inactive long enough that most excess water has drained from the pile. Concrete mix water control may suffer if sand stockpiles are too wet or if the moisture content is not uniform.

Moisture meter performance should be checked routinely. ASTM standard methods of doing this are the ASTM Test Method for Total Moisture Content of Aggregate by Drying (C 566) and the less widely used ASTM Test Method for Surface Moisture in Fine Aggregate (C 70).

ASTM C 566 covers the determination of total evaporable water in an aggregate by forced drying using either a hot plate, a hot plate together with alcohol additions to the aggregate, or a microwave oven. Evaporable water includes surface moisture and moisture within the pores, but does not include water that is chemically combined with the minerals in the aggregate. The last two methods accelerate drying but can be dangerous. Precautions given in the ASTM Standard should be observed if contemplating their use. The mass of surface moisture in the aggregate sample is determined as the total mass of evaporated water minus the calculated mass of water absorbed in the aggregate pores. Utilize the percentage of water actually absorbed in the pores of aggregate from typical plant stockpiles in that calculation. As discussed previously, aggregate from a stockpile that has been wet a long time may contain significantly more absorbed water than the same aggregate that has been soaked in water for 24 h and then tested for absorption by the methods of ASTM C 128 or C 127.

The water-immersion test method of ASTM C 70 determines aggregate surface moisture quickly, and with reasonable accuracy, provided the actual bulk relative density (SSD) of typical stockpiled material is used in the calculation. Stockpiled aggregate may have a relative density (OD) that is different than that determined for dry aggregate immersed in water only 24 h.

Expedient methods, not accepted by ASTM, have been utilized to determine sand surface moisture. One such method is the proprietary “Speedy Moisture Test” in which gas pressure generated by the reaction between acetylene crystals and the surface moisture of a weighed quantity of wet aggregate is determined and related to surface moisture percentage. This measurement is based upon the wet mass of the sample and should be corrected to the dry basis.

Another nonstandard meter calibration procedure is a variation of ASTM C 566. In the “frying pan moisture” test, a weighed sample of wet sand in a pan on a hot plate is lightly heated and stirred continuously until incipient color change and decreasing interparticle cohesion signal approaching surface dryness, and the pan is promptly removed from the burner. The slower surface moisture evaporation from the cooling aggregate sample permits determination of the precise time at which surface moisture appears to be lost and the sample of aggregate approximating surface dryness can be weighed. At this point, the percentage of surface moisture in terms of dry aggregate can be calculated on the basis of an assumed aggregate absorption. Alternatively, the sample can be further dried to constant mass, at which time the dry aggregate can be weighed and the approximate water absorption percentage of the aggregate calculated. A patient, proficient technician can utilize the methods just described to determine the mass of an aggregate sample in the wet, surface dry, and dry condition with accuracy sufficient for the control of concrete batching.

Tests of moist aggregate displacement in water have been used successfully to adjust directly the batching of aggregate and mix water when changes occur in the total moisture content of aggregates [17–19]. These procedures work well because displacement methods accurately measure total volume of wet aggregate. Consequently, if other factors remain the same, they can detect changes in aggregate volume caused by changes in aggregate surface moisture, and permit adjustments to these changes to be calculated.

The Accuracy of the Measurements

The possibility that the ASTM absorption measurement procedures are incorrect must be addressed. Intuitively, the ASTM C 127 and C 128 approaches to measure aggregate absorption appear correct; they monitor real manifestations of wet aggregate surface moisture and arrive at a point of aggregate surface-dryness when those manifestations disappear. In the case of the coarse aggregate (ASTM C 127), surface moisture is blotted with a towel. In the case of sand (ASTM C 128), surface moisture is removed by air-drying until the aggregate particles lose the cohesion afforded by the surface moisture. Furthermore, at or near the ASTM points of surface dryness the aggregates begin to change appearance and color, presumably because one is no longer looking at the particles through a film of surface water.

The pore structure as well as the surface texture of both the fine and coarse aggregate play a major role in the determination of when the “Surface Dry” condition is achieved. As the pore structure becomes larger and/or the surface texture of the coarse aggregate becomes more irregular, the surface dry determination becomes more subjective. For example, a normal weight aggregate with fine pores will not lose absorbed water to a towel during surface drying. Conversely, a few lightweight aggregates [20] have such large interconnected pores that toweling the aggregate surfaces removes some of the internal pore water.

The determination of the “Surface Dry” condition using ASTM C 128 for fine aggregate occurs when the mold is raised and the sample slumps. The difficulty is that concrete sands have different surface textures and shapes ranging from smooth, rounded river-sands to irregular, crushed “manufactured sands.” Particle mobility, illustrated for coarse materials in Fig. 1, must have similar variable effects upon the collapse of near-surface dried sands once the sand cone is raised from the compacted sand during the test for surface dryness. Manufactured sands, which hold their shape better than rounded sands when compacted, may well have lower measured absorptions simply because of their lack of particle mobility.

Intuition aside, there is little experimental evidence to confirm or discount the precision of ASTM relative density and absorption measurements. The interest in refining these measurements continues as both producers and specifiers approach performance specifications that will require better control of the concrete mixtures. An example of one attempt to reduce the variability determinations is where a Department of Transportation modified the AASHTO procedure to obtain the mass in water immediately after the soaking period instead of after attaining SSD. This appears to provide a more consistent water weight since it reduces the possibility of air being entrapped within the pores between towel drying and the submergence of the sample into the water. There are also two new procedures, which have recently been introduced, that intend to improve the consistency of the measurements by the use of proprietary equipment: one that utilizes an automated vacuum device to remove entrapped air and infrared reflectance detection for moisture determination, and another that uses a calibrated pycnometer and a vacuum sealing device to determine relative density and absorption. Only time and increased usage will tell if these procedures are effective across the broad range of aggregate particle shape and textures currently used. One of the concerns that occur when a new procedure is discussed is that while the procedure may appear to increase the repeatability of the measurements, it may also yield results that are different from those achieved using ASTM or AASHTO methods. This brings up the prospect of potentially changing specification limits and the ultimate effect on the enormous database of information that has been collected over the years.

Summary

Present ASTM relative density and absorption procedures have been utilized for a very long time; they provide a level of consistency and repeatability for most of the concrete aggregates currently in use. While it appears that they may become more subjective as particle shape and texture becomes more irregular, they have contributed to an enormous database of information derived from these tests upon which many calculations have been made, such as concrete water-cement ratio. Consequently, unless current or future research, or both, provides realistic alternatives that will improve both the accuracy and consistency across the broad range of aggregates utilized, it would be best to stay with the ASTM methods.

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Soundness, Deleterious Substances, and Coatings

Stephen W. Forster[1]

Preface

THE MATERIAL PRESENTED IN THIS CHAPTER IS A revision and update of the chapter by the same author in the previous edition (*169C*). While a number of changes have been made in this chapter (including the significantly expanded discussion of unconfined freeze-thaw testing), a great deal of the material continues to be based on the work of the chapter authors in the two editions of *ASTM STP 169* previous to *169C*. D. L. Bloem authored the section on soundness and deleterious substances in *ASTM STP 169A* and L. Dolar-Mantuani did the chapter of the same name in *ASTM STP 169B*. I am indebted to them for their work, most of which remains very valid today. Beginning with *ASTM STP 169C*, the subject of aggregate coatings was grouped with soundness and deleterious substances in this chapter. The discussion of coatings by M. A. Ozol in *ASTM STP 169B* was used as a basis for the inclusion of that subject. As with the previous edition, *ASTM STP 169C*, the chapter has been organized to concentrate on relevant ASTM standards and their use in dealing with these three aspects of aggregate quality.

Introduction

Standards Development

During the past 100 plus years, many tests have been developed to assess the quality and suitability of aggregates for use in concrete. Mather and Mather [1] provide some historical background on the early use and evaluation of aggregate materials. After a section on nomenclature, the remainder of this chapter is organized in three major headings according to the aggregate characteristic being discussed; that is, soundness, deleterious substances, and coatings. Under each heading there is a discussion of a series of aggregate quality tests, including a brief overview of each method, the characteristics of the aggregate that are being assessed, and the meaning and application of the test results. A final section presents a summary and conclusions based on the preceding discussions.

Nomenclature

Terminology that is used in the description and assessment of aggregate quality has evolved with time and usage, and continues to do so. As is often the case, the same term can have different meanings to different people. In order to make the rest of the chapter clearer, the relevant terms will be discussed and defined here.

Soundness

Bloem [2] used "soundness" as a general term to describe the durability characteristics of an aggregate as a whole. On the other hand, some literature limits the terms sound and unsound to chemical properties or mineralogy of the aggregate. In the scope section of the ASTM Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88), it is stated, "This test method covers the testing of aggregates to estimate their soundness when subjected to weathering action in concrete or other applications." This statement indicates that soundness is an aggregate's resistance to weathering, whether bound in concrete or not. Thus, soundness includes resistance to wetting and drying, heating and cooling, freezing and thawing, or any combination thereof. This is the context in which soundness is discussed in this chapter.

Deleterious Substances

Bloem [2] used "deleterious substances" to describe individual particles or contaminants that are detrimental to the aggregate's use in concrete. Rhoades and Mielenz [3] and Mielenz [4,5] used deleterious and its antonym, innocuous, to refer to chemical properties of aggregate particles. Swenson and Chaly [6] developed a detailed classification of harmful substances based on the published literature, and they used deleterious to mean any quality of an aggregate that is harmful to concrete. While their definition of deleterious is correct, this chapter is devoted to discussing "deleterious *substances*," herein defined as any material in the aggregate that is detrimental to concrete. For instance, Table 3 of ASTM Specification for Concrete Aggregates (C 33) specifically lists the following as deleterious substances:

clay lumps and friable particles,
chert (less than 2.40 saturated surface dry (SSD) specific gravity),
material finer than the 75 μm sieve, and coal and lignite.

Coatings

Ozol [7] described "coatings" as adhering materials that may be strongly or weakly cemented to the aggregate particle's surface. This description is certainly appropriate for this chapter,

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although the use of "bound" rather than "cemented" may better express the range of adherence included. Coatings may be found on sand, gravel, crushed stone, or artificial aggregate, and may be produced by either chemical or physical processes.

Soundness

Although individual particles may behave differently in concrete, it is desirable to have general criteria for the overall rating and acceptance of an aggregate source for use in concrete. It is convenient to test representative aggregate samples to obtain data that correlate well with the quality of concrete made from the aggregate. No individual test method accomplishes this. However, with respect to aggregate soundness, as defined earlier, ASTM C 88 is most frequently applied. This test, and others that have been used to evaluate soundness, are discussed below.

Sulfate Soundness Test

Description

The sulfate soundness test, ASTM C 88, was first published as a tentative test method in 1931, and finally approved as a standard method in 1963. As noted earlier, the scope of the method indicates that the test is a means to estimate the soundness of the aggregate in concrete or other applications. The test is conducted by repeated immersions of the aggregate sample in saturated solutions of sodium or magnesium sulfate alternating with oven drying to precipitate the salt in accessible pore spaces in the aggregate particles. Expansive (tensile) forces are exerted within the individual aggregate pieces by rehydration of the salt when the sample is re-immersed. This expansion is said to simulate the expansion of water upon freezing. Magnesium sulfate is generally more destructive than sodium sulfate. After the required number of cycles of soaking and drying, the amount of material that has been lost from the original particles is estimated. This is done by weighing the coarse aggregate size fractions retained as a result of hand sieving on sieves with openings five-sixths the size of those in the original sieves used to separate the sample. For fine aggregate, this material loss is determined by sieving using the same size sieves and method as was originally used to separate the aggregate. The loss is equal to the weighted sum of the difference between the initial and "after test" size fraction weights.

Examination of the test samples after testing reveals that the distress caused by the procedure may be manifested in a number of ways. Aggregate particles may show flaking, splitting, crumbling, granular disintegration, or some combination thereof. To better categorize the type of distress that occurred after completing the required number of cycles, the sample fraction retained on the 19.0 mm (3/4 in.) sieve is examined visually with or without the aid of magnification. Affected particles are categorized by type of distress, and the number of particles in each category recorded for comparison with a count taken on the number of plus 19.0 mm particles in the original test sample. Although the method calls for examining only the plus 19.0 mm size fraction, examination of the other sizes may help define the type of distress that is occurring. For further insight as to the type and progression of distress that is occurring, the test samples may be visually examined after each cycle. Shaley particles, with closely spaced planes of weakness, may split a number of times during the test without greatly reducing the sieve sizes of the particles. This phenomenon should be watched for, and noted in the report if detected.

In reporting the results of the test, a number of items should be included. The actual test results will provide the kind of solution used, the mass of each sample fraction before and after test, the percent loss for each size fraction, the weighted percent loss for each size fraction and for the fine and coarse aggregate portions, and, for the plus 19.0 mm size fractions, the number of particles before the test and the number of particles in each class of distress after the test. Information on the source of the sample is needed, and additional information on the aggregate, such as a general description and past performance, would prove useful in interpretation of the test results. Since the method currently allows flexibility in the number of cycles run, this must also be reported. Although there is this flexibility in the number of immersion and drying cycles, many laboratories use five cycles.

Precision and Bias

Before attempting to interpret the results of the soundness test (or any test), available information on the "precision" of the test (that is, maximum allowable difference between the results of two tests, properly conducted either by a single operator or by two different laboratories) and the "bias" of the test (a systematic error in the results that is inherent in the test method) must be carefully considered. In the case of the sulfate soundness test, since the soundness loss can be defined only in terms of the test method, bias cannot be determined. On the other hand, precision has been investigated and determined in a number of studies.

Precision is normally defined in terms of two variables: (1) the coefficient of variation, and (2) the difference between two tests. Precision is further broken down into within-laboratory, single-operator precision (repeatability), or between-laboratory precision (reproducibility). Early studies [8–11] indicated that numerous portions of the sulfate soundness test procedures and equipment were not well enough defined to yield results with good precision, particularly between laboratories. Improvements suggested by Wuerpel [12], Paul [13], and Woolf [14,15], among others, continued to be added to the method in the interest of better precision. Even with the continual search for and addition of improvements, the precision of the method remains poor. ASTM Subcommittee C9.20 recently conducted a carefully controlled round-robin test series on the soundness test, in yet another effort to better define the causes of poor precision. Although many laboratories feel that they produce repeatable results within their individual laboratories, the round-robin series indicated that poor precision persists within as well as between laboratories [16]. The failure to get results of even comparable magnitude appears to stem from two groups of factors, both related to the wide latitude allowed in conducting the test according to the method: (1) differences in materials or procedures used to conduct the test that are allowed under the current standard method; and (2) deviations from the prescribed method, either due to misunderstanding of the requirements or allowable changes made to the way the test is run over the years by the laboratory conducting the test. Table 1 lists some of the main factors affecting precision. Precision was reported to improve [16] with tighter control on a number of these factors noted in Table 1. As with any test method, obtaining a representative test sample is critical to getting good results. The difficulty in obtaining a representative sample is especially high for sand and gravel deposits of variable composition.

In order to improve the precision of the results, several avenues are open, the most obvious being to more closely

TABLE 1—Factors Affecting the Precision of the Sulfate Soundness Test (ASTM C 88)

- Design of containers used for immersing samples
- Temperature of the solution
- Temperature variation of the solution during test
- Chemical grade of the sulfate used
- Purity of the water used to make the solution
- Age of the solution
- Temperature of samples at time of immersion after oven drying (should match solution temperature)
- Temperature variation and rate of drying in oven

define the conditions under which the test may be conducted and the equipment and materials used in the test. Bloem [17] investigated the impact of using various options available in the method and discovered a number of trends. For instance, fine aggregates have a loss from the test about twice as large when magnesium sulfate is used as when sodium sulfate is used. For coarse aggregate, magnesium sulfate is also usually more severe, but the trend is not so clear, and can be reversed.

As would be expected, as the number of cycles of immersion and drying is increased, the loss also increases. Ten cycle losses average about 50 % higher than five cycle losses for both coarse and fine aggregate tested with either type of salt. Precision indices given in the latest version of ASTM C 88 indicate that much better precision can be obtained with magnesium sulfate than with sodium sulfate (see Table 2). These results may be due to magnesium sulfate having a solubility with a lower temperature sensitivity than that of sodium sulfate (thus making close temperature control of the immersion bath less critical to the precision of results with magnesium sulfate). In spite of the apparent advantage of magnesium sulfate, laboratories that have traditionally used sodium sulfate are reluctant to switch because direct comparison to past test results would then be lost.

Interpretation of Sulfate Test Results

In order to correctly interpret the results of this test, its significance must be clearly understood. As stated in the definition, soundness is an aggregate's resistance to weathering. How accurately the sulfate test measures soundness is the subject of debate that has continued unabated since the inception of the test. The test is most often related to that portion of weathering due to frost action.

TABLE 2—Precision Indices for the Sulfate Soundness Test (ASTM C 88)

	Coefficient of Variation (1S%), %	Difference Between Two Tests (D2S%), % of Average
Multilaboratory		
Sodium sulfate	41	116
Magnesium sulfate	25	71
Single-operator		
Sodium sulfate	24	68
Magnesium sulfate	11	31

NOTE—For further explanation of 1S% and D2S%, see ASTM Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials (C 670).

There have been a number of explanations of the process that occurs during the sulfate test that causes the disintegration of the aggregate particles [8–10,12,18]. There is general agreement that the cycles of soaking in the sulfate solution and oven drying cause the salt to accumulate in the cracks and pores in the aggregate particles. After some variable number of cycles, depending on the susceptibility of the aggregate particle, the quantity of salt in these spaces becomes more than can be accommodated during hydration in the next soaking phase. At this point, disruptive forces are exerted within the particle. Magnesium sulfate results in a greater loss than sodium sulfate because a saturated solution of it contains more material, both in mass and volume [2], thus resulting in a more rapid accumulation of salt during each drying cycle, and a greater expansion potential (tensile stress in the particles) during each subsequent immersion cycle.

Evidence suggests that there are other forces acting on the particles in addition to those exerted by rehydration of the salt during immersion. Experiments run with distilled water in place of the solution resulted in significant disintegration and loss, indicating that wetting and drying or heating and cooling are factors in the test results [10]. For carbonate aggregates, Hudec and Rodgers [19] found that disintegration was influenced by the quantity of water adsorbed on the particles. This result points to wetting and drying as being a factor in the test results. It has been found that continuing the oven drying beyond the time required to dehydrate the sulfate crystals results in greater disintegration of the aggregate, indicating the potential influence of heating and cooling.

During the early development and experiments with the test, it was suggested that the resulting disintegration was more the result of the number of pores in the aggregate than their size and distribution [11,20–25]. It should be pointed out, however, that this early work was mostly done prior to the era of air-entrained concrete and the development of some understanding of the role that the characteristics of a void system can have on freeze-thaw resistance. Various authors have pointed out that there is little theoretical basis for equating/relating the results of the sulfate soundness test with freeze-thaw disintegration/resistance in the field. However, in a statistical analysis of test results on 70 samples, Vollick and Skillman [26] did find limited correlation between the sulfate soundness test results and the freeze-thaw resistance of concrete containing the same coarse aggregates. The amount of scatter in the data indicates that there certainly were other major influencing factors. Porosity of concrete, porosity of aggregates, and freeze-thaw resistance of concrete are discussed in other chapters of this volume.

In view of the level of precision of the test method as just discussed, and the uncertainty as to what performance characteristics of the aggregate the test actually relates, it can easily be seen that interpretation of the results must proceed with caution. One suggestion was to accept aggregates that passed the test, but not to reject those that fail it [15]. The reasoning is that the test is severe and, therefore, aggregates that survive it are certainly "good," but those that deteriorate under test may or may not be good. This theory may hold for some aggregate uses, but not for others. For example, some aggregates that fail the sulfate soundness test have shown good freeze-thaw resistance in portland cement concrete, while conversely, other aggregates that have performed very well in the sulfate soundness test cause concrete to fail under freeze-thaw conditions [26].

In summary, interpreters of the soundness test results must proceed with caution. A low soundness loss will "usually" indicate a durable aggregate; however, collaborative evidence from other tests, or, ideally, prior service records in the same application lend more credence to any conclusions reached.

Specification Limits

In view of the preceding discussions on precision and interpretation of results, the difficulty in setting specification limits for the soundness test can easily be seen. Normally, specifications provide some flexibility to the user to consider other evidence when making final determination of the suitability of an aggregate. ASTM C 33 allows use of fine aggregate that has exceeded the specified limits of the soundness test results if "concrete of comparable properties, made from similar aggregate from the same source, has given satisfactory service when exposed to weathering similar to that to be encountered" or, lacking a service record, "provided it gives satisfactory results in concrete subject to freezing and thawing tests." For coarse aggregate, ASTM C 33 similarly allows the use of aggregate that has exceeded specified limits of the soundness test results "provided that concrete made with similar aggregate from the same source has given satisfactory service when exposed in a similar manner to that to be encountered; or in the absence of a demonstrable service record, provided that the aggregate produces concrete having satisfactory relevant properties."

These statements thoroughly demonstrate that the authors of this specification well realized the shortcomings of the sulfate soundness test to unfailingly discriminate between sound and unsound aggregate for use in concrete. The way the specification is written allows the specifiers to apply the soundness test as an acceptance test to the degree of strictness they are comfortable with.

Other Tests as Indicators of Soundness

Because of the imperfect prediction of soundness by the sulfate soundness test, a number of other tests have been used in lieu of, or to supplement, the results of the sulfate test. The more commonly used tests are briefly discussed subsequently. These tests often address only one aspect of soundness as defined; that is, resistance to either freezing and thawing, or wetting and drying, or heating and cooling.

Absorption Tests

It has been suggested that results of ASTM Test Method for Specific Gravity and Absorption of Coarse Aggregate (C 127) and ASTM Test Method for Specific Gravity and Absorption of Fine Aggregate (C 128) might be useful as indicators of the soundness of aggregate. Originally, it was felt that since absorption was a direct measure of accessible pore space in the aggregate, it would be closely related to the freeze-thaw behavior of the aggregate in concrete [24,25]. This relationship has not proven to be reliable; however, absorption can serve as an initial general indicator of the soundness tendency of an aggregate, prior to the completion of more reliable, definitive tests. Also, there have been some efforts to combine absorption with other tests to reach a more definitive conclusion [27].

Freeze-Thaw Testing of Unconfined Aggregate

Since the sulfate soundness test is often considered a simulation of the action on aggregate particles due to freezing and thawing, a logical extension of the soundness test is the testing of loose (unconfined) aggregate by actual freeze-thaw action. Such tests have the virtue of exposing the aggregate to an environment that will often be encountered in practice. Various cycling rates, sample conditionings, numbers of cycles, and types of additives to the freezing water have been tried in an attempt to more closely duplicate actual field performance in concrete (see, for example, Ref 28). Currently a number of states, the American Association of State Highway and Transportation Officials (AASHTO), and the Ontario Ministry of Transportation (MTO) have some version of an unconfined freeze-thaw test on their books. The Iowa DOT tests aggregate using either 16 cycles of freezing and thawing in a 0.5 % water-alcohol solution, or, alternatively, 25 cycles in plain water. For either test the aggregates are vacuum saturated. The New York DOT and the MTO both use tests with a sodium chloride solution. The use of either of these solutions increases the damage caused by each cycle, thus accelerating the test by reducing the number of cycles needed to achieve definitive losses.

A brief description of the MTO test, Unconfined Freeze-Thaw Test for Coarse Aggregate (MTO LS-614) [27] is provided herein as an example. The test was under development for ten years, with the objective of simulating the conditions of freeze-thaw cycling in the presence of moisture and de-icing salts. The test is conducted by placing three size fractions of the aggregate (see Table 3) in three separate 0.5 or 1 L jars [27]. The samples are then soaked for 24 h in a 3 % sodium chloride solution. After the 24 h, the samples are drained and sealed, then put through five cycles of freezing and thawing. For each cycle they are frozen for 16 h at −18 °C, then thawed at room temperature for 8 h. After the five cycles, the samples are drained, washed, dried, and re-sieved on the same sieves as initially used. Loss in the test is expressed as percent mass loss of material through the sieve on which the aggregate was first sieved. Each sieve fraction is tested and reported as the weighted average loss [27]. In order to continually calibrate the equipment and procedure used, a standard control material (with an average loss of 24.5 %) is routinely run along with the aggregate being tested. It has been found that intensity and length of sieving will affect test result (loss) in this test, as well as other tests that depend on sieving for results, due to continued breakdown during sieving of the weakened particles. Sieving should be closely monitored to produce consistent results.

MTO experience with the test [27] indicates that based on field performance, the test can identify marginal (fair performing) aggregates, and separate them from good and poor performers, especially when test results are considered in concert with aggregate absorption values (see Fig. 1) or micro-Deval losses (see Fig. 2). The New York State DOT also feels comfortable using a laboratory freeze-thaw test as a part of the process of aggregate acceptance. For more details on the New York DOT procedure, see Ref 29.

TABLE 3—Gradation and Mass of Freeze-Thaw Test Sample

Passing	Retained	Mass (g)
19.0 mm	13.2 mm	1250
13.2 mm	9.5 mm	1000
9.5 mm	4.75 mm	500

NOTE—Information after Ref 26.

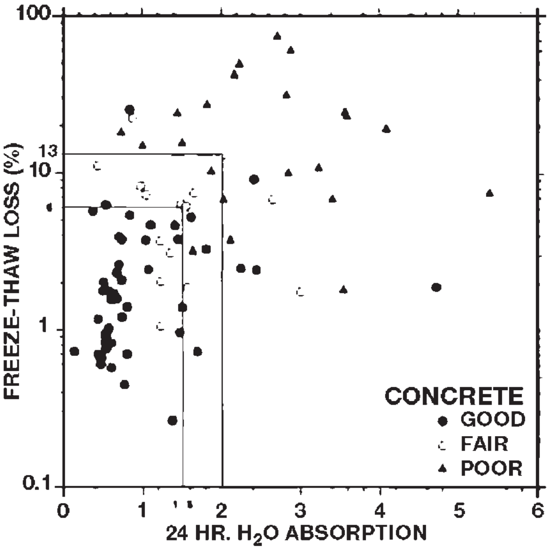

Fig. 1—Freeze-thaw test results and water absorption versus field performance (after Ref 27).

Freezing and Freeze-Thaw Tests in Concrete

Application of freeze-thaw tests to aggregate contained in concrete of the proposed mix design is the most satisfactory laboratory method of predicting the field performance of an aggregate in concrete exposed to freeze-thaw conditions. There have been a number of ASTM freeze-thaw tests on the books over the years, including ASTM Test Method for Resistance of Concrete to Rapid Freezing and Thawing (C 666), ASTM Test Method for Critical Dilation of Concrete Specimens Subjected to Freezing (C 671), and ASTM Practice for Evaluation of Frost Resistance of Coarse Aggregates in Air-Entrained Concrete by Critical Dilation Procedures (C 682). The last two, C 671 and C682, were balloted for withdrawal (due to lack of use) and withdrawn in 2003. ASTM C 666 is a current test, and has optional Procedures A and B, which, along with the conditioning of the test specimens prior to the start of the test, can have major effects on the results obtained. These effects all relate to the moisture content of the concrete (and aggregates) during testing. Even concrete that is not resistant to distress due to freeze-thaw cycling will not be adversely affected by the C 666 test if it is not critically saturated. This low moisture level may occur because of air storage of the specimens prior to test, or other steps in sample preparation and curing that lead to some drying of the test specimens. For example, it is generally agreed that ASTM C 666 Procedure B (freezing in air) is less severe than Procedure A (freezing in water), perhaps because of the potential water loss from the specimens during the freezing step.

Although now withdrawn, ASTM C 682 points out several areas of concern when interpreting test results that bear repeating because of their applicability to other tests, including C 666. First, as noted earlier, minor changes in the conditioning of the specimens can have major effects on the test results. Therefore, repeatability of test results should be one of the factors used in choosing a conditioning procedure. Sorting of the aggregate by a petrographer into homogeneous fractions prior to testing will improve the precision of the test results. Even if it is decided to test the heterogeneous aggregate sample as a whole, identifying the lithologies present, condition, and percentage of each fraction will provide insight for the interpretation of the test results. Any one test cannot serve as the definitive answer for all aggregates. A combination of tests and evaluation by a trained petrographer is the best approach. This thought will be revisited at the end of this chapter.

The freezing and freeze-thaw methods are discussed in detail in another chapter of this volume.

Other Tests

A number of other tests are or have been used as indicators of aggregate soundness. These tests include the copper nitrate test as described by Dolar-Mantuani [30]. This test, conducted by immersing the test sample in copper nitrate solution overnight, has a similar effect on shaley or argillaceous particles as the sulfate soundness test. Other tests that may be indicative of soundness and that are described elsewhere in this volume include the two impact and abrasion tests using the Los Angeles machine (ASTM Test Method for Resistance to Degradation of Small-Size Coarse Aggregate by Abrasion and Impact in the Los Angeles Machine (C 131) and ASTM Test Method for Resistance to Degradation of Large-Size Coarse Aggregate by Abrasion and Impact in the Los Angeles Machine (C 535)), ASTM Test Method for Lightweight Pieces in Aggregate (C 123), and ASTM Test Method for Clay Lumps and Friable Particles in Aggregates (C 142).

Petrographic Examination for Soundness

The evaluation of an aggregate sample by a trained, experienced petrographer both independent of, and in concert with, other tests is an invaluable tool in correctly predicting the soundness of an aggregate source. ASTM Guide for Petrographic Examination of Aggregates for Concrete (C 295) provides a good outline for conducting a systematic evaluation. The lithology (rock type) and state of weathering are two important factors that must be considered when evaluating the soundness of an aggregate. The procedure is discussed in detail in Chapter 22 of this publication. Beyond the specifics of the practice, the basic lesson to be learned from the general approach of the petrographic examination, and something equally applicable to any

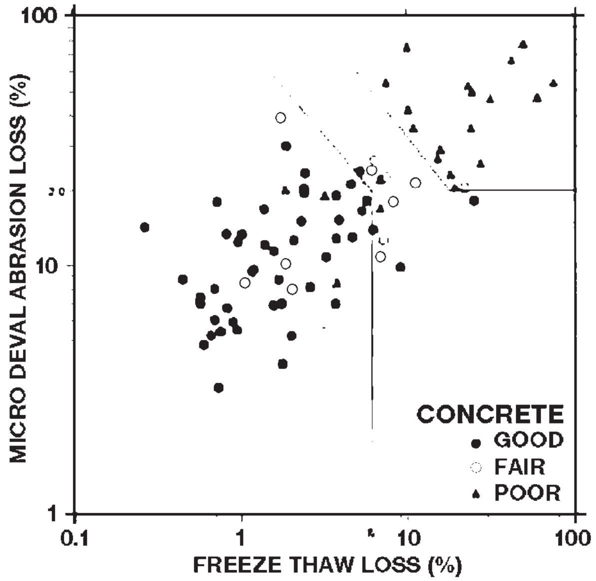

Fig. 2—Freeze-thaw test results and micro-Deval loss versus field performance (after Ref 27).

type of evaluation, is to "take advantage of all available pertinent information prior to reaching a conclusion."

As was noted earlier, ASTM C 88 requires the qualitative examination of the sample particles larger than 19.0 mm for categorizing the types of distress present. (Examination of the smaller sizes will often add additional insight on the aggregate's performance.) If conducted by a qualified petrographer, this examination, in concert with a petrographic examination of the sample prior to testing, will provide great insight as to the causes for the aggregate being susceptible to the conditions of the soundness test. Knowing the causes of distress in the affected particles, and their overall percentage in the aggregate source, a better prediction of the actual performance of the aggregate in concrete in service can be made, as well as recommendations on the need and practicality of benefication of the aggregate.

Deleterious Substances

As noted in the preceding section on nomenclature, a deleterious substance (as used in this chapter) refers to any material in the aggregate that is detrimental to concrete. According to ASTM C 33, deleterious substances include the following categories: clay lumps and friable particles, chert (less than 2.40 SSD specific gravity), material finer than the 75 μm sieve, and coal and lignite. Each of these categories will be briefly described.

Description

Clay Lumps

The term, clay lumps (or clay balls), refers to lumps of clay to fine sand-sized particles that are present during and after the aggregate processing. The lumps would have to be mechanically broken up to be effectively dispersed. If they survive the aggregate processing procedures, they also will usually survive the mechanical action associated with the mixing and placing of concrete. They will, however, be subject to some surface abrasion during the mixing and, as a consequence, often contribute additional, unaccounted-for fines to the mix.

Friable Particles

Friable particles are those aggregate pieces that have little bond between the mineral grains that compose the aggregate particles. The individual minerals may or may not be soft; however, the fact that the bond between grains is weak results in the aggregate particles breaking down into smaller pieces or rapidly losing grains, or both, from their surface during the mechanical action of processing or concrete mixing and placing. Examples of friable aggregates are a sandstone composed of quartz grains weakly cemented together by a clay or calcite matrix, and a poorly indurated shale.

Chert (SSD Specific Gravity Less Than 2.40)

Based on empirical evidence, chert particles with an SSD specific gravity of less than 2.40 are considered to be usually objectionable as aggregate in concrete due to frost susceptibility and the resulting cracking of the concrete or the formation of popouts at the concrete surface. Where individual aggregate particles are composed of some lightweight chert and the parent rock (limestone or dolomite), the particles may be above the 2.40 specific gravity but still cause these problems due to the chert. These aggregates will therefore have to be examined carefully.

Material Finer Than the 75 mm Sieve

Particles that pass the 75 μm sieve are generally referred to as silt and clay. This includes the fine material that can occur in sand and gravel deposits as well as the dust of fracture that may result from the crushing and mechanical processing of aggregate. If this material adheres to the coarser aggregate particles during processing, it can create problems if it provides too much unanticipated fines to the concrete during mixing or results in a poor coarse aggregate-to-paste bond in the hardened concrete. If this material is essentially free of clay "minerals" or shale (as is usually the case with dust of fracture), it may be present in greater amounts without adverse effect on the concrete. Fine material from crushed stone production (dust of fracture) is reported to not be detrimental, and may even be of benefit to strength for lean concrete mixes [31].

Coal and Lignite

Coal and lignite may occur as discrete coarse particles in the aggregate or as fine material disseminated throughout. If not derived through contamination from another source, coal and lignite are often associated with shale, something not normally used in concrete; however, coal and lignite may also be found associated with other rock types.

Other Deleterious Substances

Other deleterious substances not specifically mentioned earlier include lightweight pieces other than chert, soft particles, and organic impurities other than coal and lignite. Lightweight pieces include highly porous aggregates that float on a liquid of density 2.0. These particles often lack durability. Soft particles, as distinguished from friable particles, are composed of minerals that are soft and therefore are very susceptible to abrasion and wear. They are often the result of long-term (in a geologic sense) weathering and chemical alteration of originally durable rock. Organic particles other than coal and lignite include plant roots, twigs, and other vegetable and animal material.

Effect of Deleterious Substances on Concrete

When clay lumps survive the processing of the aggregate, and also the mixing and placing of the concrete, they are subject to breakdown during wetting and drying, and freezing and thawing of the hardened concrete. This can result in popouts near the concrete surface and also the appearance of a pock-marked surface where the clay lumps have weathered away.

Friable particles and soft pieces are easily broken down into smaller particles or create additional fines. If the deterioration of the friable particles continues once the concrete is in place, it may result in freeze-thaw damage to the concrete. If soft particles are present in sufficient percentages, a lowering of the concrete strength and durability will result. Soft or friable particles exposed at the concrete surface will certainly lower its abrasion resistance and will often quickly weather away leaving a pitted or pock-marked surface.

Low specific gravity chert particles are susceptible to frost action due to their internal pore structure. Their deterioration in concrete can result in cracking of the concrete or popouts if the aggregates are located just below the concrete surface.

An excessive amount of minus 75 μm material in the aggregate results in high water demand by the concrete for mixing, placing, and finishing. Once in place, the excess water increases drying shrinkage and the likelihood of cracking. The excess water also leads to lower strengths. If the fines include one or more of the swelling clay minerals (montmorillonite, smectite, or nontronite), volume change during wetting and drying in the hardened state can be increased, with the formation of microcracks (see ASTM C 295, Ref 32 and Ref 33). If the fines remain adhered to the coarse aggregate during mixing, a poor coarse aggregate-to-paste bond may develop, resulting in lower strengths. As noted earlier, if the minus 75 μm material is essentially free of clay minerals and shale, higher percentages may be allowable (see ASTM C 33); however, the total amount of this material in the coarse and fine aggregate must be considered. A small percentage of this material may have strength, workability, and density benefits for lean concrete mixes.

Finely divided organic material may retard the hardening of the concrete and reduce the concrete strength. This material may also interact with or affect the effectiveness of any air-entraining or other admixtures used in the concrete. Coal and lignite are usually present in larger-sized pieces and often result in pits or popouts at the surface of the concrete due to subsequent weathering. Staining of the surface may also result from coal/lignite. Brown lignite coal, wood, tall oil residues, and pine oil contain materials that readily disperse in the alkaline solution present in concrete and may cause dark brown stains and zones of weakness [34].

Tests for Deleterious Substances

Clay Lumps and Friable Particles

Testing for these two classes of deleterious substances is combined in one ASTM test, ASTM C 142. This method is applied to an aggregate sample after it has undergone a washed gradation (ASTM Test Method for Material Finer Than 75-μm (No. 200) Sieve in Mineral Aggregates by Washing (C 117)) to determine the amount of minus 75 μm material in the aggregate. As a result, any clay lumps or friable particles (or portions thereof) that break down in the wet sieving process will not be measured in the ASTM C 142 test. The test procedure itself consists of a 24-h soak of the sample in distilled water (after the sample has undergone the ASTM C 117 test) followed by manual manipulation by the fingers to break any susceptible particles into smaller sizes. After manipulation, the sample is wet-sieved over a designated sieve according to the size range of the original sample. The percentage of clay lumps and friable particles is the change in mass of the sample due to the soaking manipulation and sieving processes.

Chert (SSD Specific Gravity Less Than 2.40) and Other Lightweight Pieces

Due to its pore structure, chert that has the potential for physical disruption of concrete under freeze-thaw conditions usually has an SSD specific gravity of less than 2.40, and this characteristic is used as a distinguishing criteria in ASTM C 33. It should be noted that this criteria applies only when the chert is present in the aggregate in small amounts; if the aggregate is predominantly chert, its potential effect on the durability of the aggregate must be determined by other means, preferably prior service record in similar concrete, structure, and exposure. ASTM C 123 is used to separate aggregate pieces of different specific gravity. The procedure employs a heavy liquid that can be mixed to possess the specific gravity required as the separation value (2.40 for chert, 2.0 for coal and lignite). Then it is simply a matter of placing the test aggregate in the heavy liquid, skimming off the lightweight (reject) pieces that float, and calculating their percentage of the total sample.

It has been reported [35] that sand-sized particles of chert (which are detrimental to the durability of concrete in coarse aggregate sizes) do not damage concrete containing them when it is subjected to ASTM C 666. The vulnerability of the chert to freeze-thaw damage depends on a number of interrelated factors, including the characteristics of the pore system and the particle size, as well as the characteristics of the surrounding concrete paste. As is often the case, directly comparable service records are the surest means to predict performance.

If there is any doubt about the composition of the lightweight pieces identified by ASTM C 123, the particles should be examined by a petrographer. In addition to the detrimental materials noted earlier and deeply weathered soft (nondurable) aggregate particles, certain man-made aggregates and natural volcanic rocks that perform well in concrete may float in the test.

Material Finer Than 75 μm Sieve

ASTM C 117 is the method used to determine the amount of this material in an aggregate sample. The test involves washing the material over a 75 μm sieve, and any material carried through the sieve by the wash water (or less frequently dissolved and removed in the water) is the amount of minus 75 μm material in the sample. The precision statement for ASTM C 117 indicates that for coarse aggregate, the results of two properly conducted tests should differ by no more than 0.28 % for a single operator or 0.62 % for two different laboratories. Similarly, for fine aggregate, the difference should be no greater than 0.43 % for a single operator or 0.82 % for two different laboratories.

Other tests have been developed that are reported to indicate the amount or proportion of clay-sized fines in concrete aggregate [32]. Of these, the ASTM Test Method for Sand Equivalent Value of Soils and Fine Aggregate (D 2419) is the most notable. By means of water suspension and settlement procedures, this test provides a rapid indication of the relative proportion of "clay-like or plastic fines and dusts" in a fine aggregate or soil. Tests conducted by Gaynor [36] on 130 samples showed, however, that there was little correlation between the amount of minus 75 μm material in the sample and the results of the sand-equivalent test. Others have reported some correlation between the sand-equivalent test and the water demand of a concrete mix [34]. In order to determine the mineralogy of the minus 75 μm material, and whether it is potentially detrimental clay minerals or dust of fracture or other material, it may be necessary to run X-ray diffraction or other analysis techniques. A petrographer should be consulted in this regard.

Soft Particles

Although often confused, soft particles and friable particles are distinct. As noted earlier, the component mineral grains that make up a soft particle are soft (often weathered), whereas the component grains of a friable particle may or may not be soft; rather it is the weak bond holding the grain together that defines a friable particle. Discontinued ASTM Test for Scratch Hardness of Coarse Aggregate Particles (C 235) (later included as ASTM C 851) was used for some time as a means of identifying soft particles. Deletion of ASTM C 851 from the *ASTM Book of Standards* followed a number of years of debate in committee meetings as to the merits of the test. Percentages of soft particles in an aggregate seemed to have little correlation with concrete strength. This low correlation may have been due to the difficulty in differentiating between soft particles and friable particles using the test method.

Two test methods, ASTM C 131 and ASTM C 535, have also been used as a measure of the presence and amount of soft particles. As with the scratch test, the results should be interpreted carefully since many factors other than the softness of the particles can influence the test results. Petrographic examination of the material will help in identifying the characteristics that led to the test results obtained.

Organic Impurities

ASTM Test Method for Organic Impurities in Fine Aggregates for Concrete (C 40) is a simple screening test for determining the presence of organic material. The procedure consists of - immersing a given amount of fine aggregate in a standard solution of sodium hydroxide and noting the color change in the solution after 24 h due to reaction of the solution with any organic material present in the aggregate. The color of the solution is compared to a standard solution or standard color glass plates to qualitatively determine the amount of organic matter. Since the test solution reacts with all types of organic matter, including bits of wood and twigs that do not affect concrete strength when present in small amounts, it is only a screening test. Aggregates resulting in colors darker than the standard should be further evaluated by other tests such as mortar strength and setting time. ASTM Test Method for Effect of Organic Impurities in Fine Aggregate on Strength of Mortar (C 87) compares strength results of mortar samples containing the subject aggregate as received with strength results of mortar samples made using the subject aggregate after it has been washed to remove the organic matter.

In addition to strength and setting time, some organic matter may reinforce or detract from the efficiency of admixtures. Air entrainment is a particular area of concern [37] in that the organic material may act as an air-entraining agent of variable efficiency.

Coatings

Description

Naturally Occurring Coatings

Naturally occurring coatings are those that are deposited on the surfaces of the aggregate particles by means of natural processes. Within this context, this class of coatings is limited to occurrence in sand and gravel deposits, since only this type of aggregate source has the aggregate particle surfaces exposed for the requisite period of time for these natural processes to take place. The chemical and mineralogical composition of the coating can vary over a wide range. The two most important chemically precipitated coatings are calcium carbonate and silica. Deposition of these coatings often results from solution of the minerals from one portion of the deposit (by percolating groundwater) and redeposition in another (usually lower) portion of the deposit. Since the source of the coatings is the particles in the deposit itself, the coatings and the aggregate particles on which they precipitate are often of the same composition. Other possible chemically precipitated coatings include gypsum, other sulfates, iron oxide, opal, and phosphates. There also are physically deposited coatings in sand and gravel deposits; namely, a layer or layers of silt and clay-sized particles. These coatings are also transported by the moving groundwater in the sand and gravel deposit, but migrate as solid particles rather than being dissolved, transported in solution, and then precipitated as in the chemical coatings process.

Artificially Generated Coatings

Coatings on aggregate particles may also result from one or more of the steps necessary to produce an aggregate from a source material. These coatings usually consist of fine materials of the same composition as the particles they coat because they are derived from these particles through impact and abrasion during handling. Loading, crushing, unloading sizing, and stockpiling all produce a certain amount of fines (often called "dust of fracture"), the amount dependent on the characteristics of the source material being processed. A hard, polish, and wear-resistant source material will produce only small amounts of fines during processing, usually not nearly enough to produce any undesirable coating of the larger aggregate pieces. A soft, wear, and polish-susceptible aggregate source material, on the other hand, may produce enough fine material to form a coating on the coarse aggregate. This coating may have to be dealt with either by additional aggregate washing to remove it or, if the coating of fines is shown to be removed from the aggregate during mixing, by compensation for the additional fines in the mix during the design of the concrete.

Identification

The usual approach used for identifying the composition and extent of coatings is a petrographic examination, ASTM C 295. As noted earlier, sand and gravel deposits may have either chemically or physically deposited coatings, whereas a crushed stone can have only a physically deposited coating. This practice allows a wide latitude in the equipment and procedures used depending on the nature of the material being identified and other characteristics to be determined. If the coatings are considered to contain materials (such as opal, gypsum, organic material, or easily soluble salt) likely to be deleterious when the aggregate is used in concrete, the coatings must be positively identified by whatever necessary means. The strength of the bond of the coating to the particles should also be determined, first qualitatively during the petrographic examination, and then, if necessary, by conducting strength tests on concrete containing the aggregate, including post-test examination of the break surfaces.

Effect of Coatings on Concrete

From a physical standpoint, aggregate coatings may be detrimental to the strength properties of concrete if the bond of the cement paste to the coating is greater than the bond of the coating to the aggregate particles. When this occurs, the concrete will fail at the coating/aggregate interface at a smaller load than it would otherwise fail. In this regard, Goldbeck [38] reported that flexural strength reductions of up to 1.5 %, and compressive strength reductions of up to 2 %, can occur for every percent of dust of fracture included in the aggregate. As noted elsewhere, if the dust of fracture occurs not as a coarse aggregate coating, but dispersed in the mix in small quantities, it may not be detrimental to the concrete and may in fact be of benefit to some properties of the plastic and hardened concrete.

Soluble or chemically reactive coatings will cause the same deleterious results in the concrete as they would if included in the concrete in any other form, such as coarse or fine aggregate particles. Certain siliceous coatings (opal, for example) may react with the alkalis in the cement, depending on the amount of alkalis present, other components in the mix, and additional factors [39]. The products of this reaction are susceptible to swelling in the presence of moisture, producing tensile stresses in the concrete that can lead to cracking. Coatings composed of iron compounds may result in staining of the concrete surface, as well as a weakening of the aggregate/paste bond mentioned earlier. Chlorides and sulfates can cause staining and efflorescence at the concrete surface and may also lead to corrosion of reinforcing steel [7].

In summary, aggregate coatings may be innocuous or deleterious to concrete depending on their solubility in the concrete and their effect on any chemical reaction that might occur, their bond to the aggregate relative to the cement paste's bond to them, and the effect that any fine particles derived from them have on the properties and characteristics of the plastic and hardened concrete. Examination of specimens (both before and after strength testing) made during the mix design process will often help to verify conclusions reached about coatings during the petrographic examination of the aggregate.

Conclusions

An aggregate's "soundness" may be best equated to its resistance to all aspects of weathering when used in concrete or other applications. Weathering may include any combination of heating and cooling, wetting and drying, or freezing and thawing. Because it is so complex, no one test adequately measures the soundness of an aggregate, although several tests certainly provide good insight as to how an aggregate will behave. Results of ASTM C 88 are often considered to be an aggregate's soundness without considering all that the term implies. The unconfined freeze-thaw test of aggregate is also beginning to be used more widely as an indicator of soundness. ASTM C 88 certainly can be a useful indicator of soundness and, when used along with other information (preferably including a prior service record), a reliable assessment of soundness can be made.

A deleterious substance, as defined herein, refers to any material in the aggregate (other than coatings) that may be detrimental to the concrete in which the aggregate is used. By convention, the term deleterious substance usually refers to accessory or minor constituents in the aggregate rather than major components. Deleterious substances include clay lumps and friable particles, low SSD specific gravity chert, fine material, coal and lignite, and others. The effect of deleterious substances on concrete, and the tests used to determine their presence and amount, will depend on the type of substance under consideration, the amount of the constituent present, and the structure and environment in which the concrete will be placed. Certain types of fine material, such as dust of fracture from crushed stone production, are reported to be not detrimental and even beneficial in some instances [31].

Coatings may be subdivided into naturally occurring coatings and those generated by artificial means through processing of the aggregate. Because of the wide variety of materials included under coatings, evaluation by a qualified petrographer using ASTM C 295 is the most practical approach. Following identification of the coatings involved, specific tests may be prescribed depending on the identity, nature, and quantity of the coating.

In all three of these areas of aggregate quality assessment, the surest means of prediction of performance of an aggregate in concrete is the availability of a prior service record. To be most reliable, the service record should involve the same aggregate (often just the same source is not specific enough), the same concrete mix design and components, incorporation into the same type of structure, and should be subject to the same environment. Usually, one or more of these components of the service-record is different from the intended use and, therefore, some interpretation will be necessary. It is at this point that examination of test results, a petrographic evaluation, and good engineering judgment must be employed.

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Degradation Resistance, Strength, and Related Properties of Aggregates

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Preface

THIS CHAPTER CONCENTRATES ON TESTS RELATING to degradation of aggregates—both wet and dry tests—and includes a discussion of the effect of aggregate strength on concrete. Also, the effect of aggregates on wear and frictional properties of concrete in service is briefly covered.

The two previous editions of this chapter by the same author [1,2] were longer. The 1978 version included material concerning research on strength and abrasion properties of aggregates and a review of North American and European methods and research. It also included additional references and discussion of the mineralogic nature of the breakdown of aggregates in tests and information on hardness, impact, and toughness. The 1994 version included information on ASTM Method C 1137 on degradation of fine aggregate in water in a vane-type attrition chamber. That method is rarely used now, so it is not discussed in any detail here. It is recommended that engineers or researchers interested in these topics refer to the *ASTM STP 169 B* and *C* editions referenced above. In earlier editions of this book, *ASTM STP 169* and *ASTM STP 169A*, D. O. Woolf covered this topic in his chapter on "Toughness, Hardness, Abrasion, Strength, and Elastic Properties."

Introduction

Aggregates for use in concrete, and to a large extent the individual constituent particles, must possess a reasonably high degree of inherent strength, tenacity, and stability to resist, without detrimental degradation, the static and dynamic stresses, impacts, and wearing actions to which they may be exposed both in concrete production operations and, ultimately, in concrete in service. In many uses of concrete, the roughest treatment to which an aggregate may be subjected in terms of mechanical forces and attrition is in the concrete production process. Actions involved in production are fairly predictable; those in service may be less predictable. In end uses such as beams, columns, covered slabs, walls, footings, and other mostly structural or architectural elements, the only real strength property needed of the aggregate after the concrete is in place is that necessary to give the concrete enough strength to resist the distributed service loads. Other uses may expose aggregate near or at the surface to a variety of localized impact and abrasive stresses that will be of overriding importance in aggregate evaluation and selection. Examples include pavements and slabs exposed to heavy traffic and hydraulic structures subject to eroding forces of moving water and sediment material.

Some recognition must be given to the consideration of an aggregate as a whole versus the properties of the individual particles present. In many crushed stone aggregates and some sands and gravels, the lithology of the particles and, consequently, their mechanical properties show little variation. In other cases, great variations may exist among the mineral and rock particles involved. A small percentage of weak particles in most instances is not objectionable and may be thought of as an impurity or deleterious substance, and therefore they are not the proper subject of this discussion. These weaker or less abrasion resistant particles allowed in small percentages may cause a slight increase of fines during mixing or may contribute to occasional surface imperfections not particularly harmful to overall performance. In other instances, eliminating even very small percentages of friable or weak particles is desired. An example is high-velocity hydraulic structures where high surface strength and very accurate surface alignment is needed to resist cavitation damage that can spread rapidly once started.

Concrete carrying a distributed static or dynamic load, and not subjected to local stress concentrations, requires aggregate that will bond with the surrounding cement paste permitting the transfer of stress through the aggregate particles. Here, the particles are expected to possess enough strength and rigidity to carry the stresses without mechanical breakdown or excessive deformation—for usual aggregates and typical stress levels in concrete, this is not a problem. The rock, mineral, or synthetic materials making up aggregates are usually much stronger in compression or tension than the concrete; and the weakest link in the system is the bond between the paste and aggregate. High-strength aggregate materials will probably be required in the production of very high-strength concrete, in the range 70 MPa (10 000 psi) to 140 MPa (20 000 psi). In some cases, however, aggregate materials are used that possess compressive strength of the same order of magnitude as the concrete in which they are used. Examples are lightweight aggregates, some porous marine limestones, and volcanic cinders sometimes used in concrete. These weaker materials are used because of economic reasons or to produce concrete of lighter unit weight.

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Impact, abrasion, scuffing, attrition, and other wearing actions, either in a dry environment or in the presence of water, can be important factors. These loadings or actions usually are applied to the surface of the concrete so that only the properties of the aggregate at or near the surface are of any substantial importance in determining whether the long-term performance of the concrete will be acceptable. In some cases, special aggregates, concrete mixtures, or coatings may be used at the surface to improve the surface properties locally.

Surface actions are of two basic modes–impact and rubbing. Impact is where hard particles or objects impinge against the concrete surface with enough momentum to cause shattering, yielding, or debonding of aggregate particles. Materials with good impact resistance are said to possess good toughness. In the rubbing or scratching mode, damage can be inflicted by the movement of particles or objects on the surface under enough load to cause indentation, for the relative hardness involved, and therefore cause scratching or gouging as movement occurs. Scratches and gouges from relatively large particles or objects are on a macroscopic scale and are termed wear. In other words, the amount of material removed by such action, if repeated over and over again, will wear away a significant quantity of surface mortar, exposing more and more coarse aggregate with time. Rubbing or scratching action from much smaller particles–on a microscopic scale–does not have the capacity to remove much material, but it can cause a polishing action at the surface. In highway uses, and occasionally other types of service, aggregates that allow rapid polishing can contribute to poor frictional properties of wet surfaces.

Applicable ASTM Standards

The ASTM standards given here fall under the general purview of this chapter. Some relate directly to aggregate properties, and others relate to properties of concrete in which the performance of different aggregates can be compared.

1. ASTM Specification for Concrete Aggregates (C 33)
2. ASTM Test Method for Resistance to Degradation of Small-Size Coarse Aggregate by Abrasion and Impact in the Los Angeles Machine (C 131)
3. ASTM Test Method for Resistance to Degradation of Large-Size Coarse Aggregate by Abrasion and Impact in the Los Angeles Machine (C 535)
4. ASTM Test Method for Aggregate Durability Index (D 3744)
5. ASTM Test Method for Abrasion Resistance of Concrete by Sandblasting (C 418)
6. ASTM Test Method for Abrasion Resistance of Concrete (Underwater Method) (C 1138)
7. ASTM Test Method for Abrasion Resistance of Concrete or Mortar Surfaces by the Rotating-Cutter Method (C 944)
8. ASTM Test Method for Abrasion Resistance of Horizontal Concrete Surfaces (Procedure A–Revolving Disks; Procedure B–Dressing Wheels; Procedure C–Ball Bearings) (C 779)
9. ASTM Test Method for Accelerated Polishing of Aggregates Using the British Wheel (D 3319)
10. ASTM Test Method for Insoluble Residue in Carbonate Aggregates (D 3042)
11. ASTM Test Method for Skid Resistance of Paved Surfaces Using a Full-Scale Tire (Locked Wheel Skid Trailer) (E 274)
12. ASTM Test Method for Skid Resistance Measurements Using the North Carolina State University Variable-Speed Friction Tester (E 707)
13. ASTM Practice for Accelerated Polishing of Aggregates or Pavement Surfaces Using a Small-Wheel, Circular Track, Polishing Machine (E 660)
14. ASTM Test Method for Degradation of Fine Aggregate Due to Attrition (C 1137)–This test is rarely used and has not been recently reapproved or revised. The Micro-Deval test is now considered best for testing the wet degradation of coarse or fine aggregate for construction.
15. ASTM Test Method for Resistance of Coarse Aggregate to Degradation by Abrasion in the Micro-Deval Apparatus (D 6928)–ASTM and AASHTO have standardized the coarse aggregate test, and ASTM is working on standardizing the fine aggregate Micro-Deval procedure. CSA has standardized both the fine and coarse aggregate Micro-Deval tests.

ASTM C 33 for concrete aggregates includes a modular format where many of the required properties of coarse aggregates are varied depending on severity of exposure to weather and the end use of the concrete. However, due to the lack of enough significantly explicit data relating concrete performance with degradation in the Los Angeles Machine (ASTM C 131 or C 535), the “abrasion” limit in ASTM C 33 has been set at 50 % for all categories. (Note: ASTM C 131 and C 535 now properly refer to “degradation” in the tests by “abrasion and impact in the Los Angeles machine;” however, the terms: “abrasion,” “abrasion loss,” “L.A. abrasion,” and “L.A. wear” are often used to refer to the percentage loss in these tests and as an abbreviated name for the tests.) In addition, judgment provisions such as the following have been included in many specifications to allow use of known satisfactory materials: “Coarse aggregates having test results exceeding the limits specified. . .may be accepted provided that concrete made with similar aggregate from the same source has given satisfactory service. . .or in the absence of a demonstrable service record provided that the aggregate produces concrete having satisfactory relevant properties. . .” when tested in the laboratory. ASTM C 33 specifies requirements for a wide range of aggregate sizes. The determination of which grading to use in ASTM C 131 or C 535, to test an aggregate product, is based on the requirement that degradation in the L.A. machine is to be determined using the grading most nearly corresponding to the grading to be used in the concrete. Crushed air-cooled blast-furnace slag is excluded from the L.A. abrasion requirement in ASTM C 33. In its place, a minimum compact unit weight is required.

The test methods listed here are discussed in subsequent sections relating to the properties measured. Other than the percent loss limits for degradation of coarse aggregate by abrasion in the L.A. machine, as measured by ASTM C 131 or C 535, there are generally no other specified limits with respect to the effect of coarse or fine aggregate as measured in the other tests unless there are special requirements limiting degradation of aggregates in handling and mixing, limiting abrasion resistance of concrete surfaces, or requirements pertaining to the frictional properties of pavement surfaces after being subjected to traffic [3].

Degradation of Coarse Aggregate (Los Angeles Abrasion)

ASTM C 131 and C 535 are accepted and used almost universally in the United States as specification qualification tests for coarse aggregate for concrete. They are also used widely around the world to evaluate coarse aggregates for various applications.

ASTM C 131 was first adopted as a tentative standard test in 1937. At first, it provided for testing only 37.5 or 19.0-mm (1 1/2 or 3/4-in.) maximum size aggregates (Gradings A and B). Later, 9.5-mm (3/8-in.) and 4.75-mm (No. 4) sizes were added (Gradings C and D). In 1947, a revision was prepared to add three more gradings for large aggregate sizes–75-mm (3-in.), 50-mm (2-in.), and a second, narrower 37.5-mm (1 1/2-in.) maximum size grading (Gradings E, F, and G)–with an increased sample size from 5000 to 10 000 g and a doubling of the number of revolutions from 500 to 1000 for these larger sizes. In 1964 and 1965, the larger sizes were deleted from ASTM C 131, and a new test method, ASTM C 535, was adopted for them.

Briefly, the method is a dry abrasion and impact test that involves placing a sample of aggregate and a charge of steel balls into a hollow steel cylinder that is 508 mm (20 in.) long and 711 mm (28 in.) in diameter, is closed on the ends, and has a single rigid steel shelf extending 89 mm (3 1/2 in.) into the chamber. The cylinder is rotated at 30–33 rpm for 500 revolutions in C 131 (1000 revolutions for C 535.) Percent loss is obtained as the difference between the original sample mass and the amount of the final sample coarser than the 1.70-mm (No. 12) sieve, expressed as a percentage of the original mass. Often the loss is determined at 100 and 500 revolutions (200 and 1000 revolutions for C 535) to give an indication of the rate of loss. For a material degrading at a uniform rate, the ratio of the loss after 100 revolutions to the loss after 500 revolutions should not greatly exceed 0.2; a small percentage of very soft, friable, or brittle material or a large number of flat or elongated pieces might cause this ratio to increase.

The precision statement in ASTM C 131 is based on 19.0-mm (3/4-in.) maximum size aggregate samples distributed to a large number of laboratories throughout the United States by the AASHTO Materials Reference Laboratory. The samples were tested by both ASTM and AASHTO procedures, which differ only in minor respects for this particular test. Multilaboratory coefficient of variation was found to be 4.5 %, and single operator coefficient of variation was 2.0 %. These data are based on samples ranging in loss from 10–45 %. In another precision investigation within one state, the data show a higher level of variability on samples having a loss of 13–18 % [4].

The same equipment is used for ASTM C 535. The required charge of steel spheres is 12 for all three gradings, whereas the charge varies with grading in C 131. In C 535, 1000 revolutions of the drum are used. Loss is again determined using the 1.70-mm (No. 12) sieve. No data are available to use in the development of a precision statement for ASTM C 535.

Caution should be exercised in comparing values obtained from ASTM C 131 and C 535. They may give different results for similar materials. For this reason, specification limits for larger sizes may not necessarily be the same as those judged to be needed for the smaller sizes of coarse aggregate.

Impact probably causes more loss, at least during early revolutions of the drum before any fines are created that might tend to cushion the impact forces. To some extent, harder rocks and minerals, even though they are strong, tend to shatter more than softer materials that can better absorb the force. On the other hand, softer minerals will be more susceptible to surface wearing. The product of the degradation action in that case may be more of a dust rather than the larger angular pieces resulting from the shattering. Also, dust produced early in the test may provide a cushioning effect, giving possible misleading results.

Smith [5] used coarse aggregates in concrete with L.A. abrasion losses ranging from 13–39 %. With one exception he found no significant correlation between L.A. loss and the abrasion resistance of concrete containing the aggregates using three different test procedures. The most significant correlations with concrete abrasion resistance were with concrete strength and water-cement ratio. One soft, fossiliferrous limestone did decrease the concrete abrasion resistance at strengths below 50 MPa (7200 psi). Above that strength, no coarse aggregate effect could be detected.

The loss in the L.A. abrasion test has been correlated by Woolf [6], with the strength of concrete prepared with a wide variety of aggregates. However, laboratory studies reported by Bloem and Gaynor [7] failed to show such a relationship with strength. Jumper [8] and Walker and Bloem [9] conducted research where aggregates of high and low abrasion loss were blended to produce a range of values. These simulated coarse aggregate combinations did show some correlation of reductions in abrasion values with increased strength. Jumper blended aggregate to obtain loss values from 42–58 %. Over that range, the decrease in compressive strength for constant cement factor concrete was from about 27–23 MPa (3900–3300 psi). Walker and Bloem did not detect any dependence of concrete compressive strength on abrasion loss, but they did find that high abrasion losses had lower flexural strength with the aggregates studied. Bartel and Walker [10] and Walker [11] showed a slight drop-off of compressive and flexural strength with increased abrasion losses. Correlation of L.A. abrasion with concrete strength is inconclusive.

With respect to the freezing and thawing durability of aggregates tested in concrete specimens, Gaynor and Meininger [12] did not find a relationship between concrete durability and abrasion loss at 500 revolutions or 100 revolutions. This study included 56 coarse aggregates, ranging in abrasion loss from about 15–65 %. Gaynor [13] also reports this and other freezing and thawing data.

Kohler [14], in a comprehensive study reported to the Highway Research Board, compared the results of aggregates tested by the German Impact Test, the L.A. abrasion test, the older Deval Abrasion Test, the British Impact Test (British Standard 812), and a modified Marshall Impact Test. He found that even though the methods use different loads and procedures, the tests characterize “the same or at least similar properties of the aggregates.” Good correlation was found between all the tests run in one laboratory. High variation of results was found for the L.A. abrasion and German Impact Tests when tests were run in different laboratories, suggesting that further strict standardization of procedures may be necessary.

Over the past several years, European Standards have been developed for aggregates under CEN/TC 154. Of those for mechanical and physical properties of aggregates are the following:

1. EN 1097-1:1996 includes the Micro-Deval test of coarse aggregate using a 500-g specimen.
2. EN 1097-2:1998 includes the L.A. abrasion test and a drop-hammer impact test of coarse aggregate to determine resistance to fragmentation.
3. EN 1097-8:1999 includes the polished stone value of coarse aggregate that is similar to the British wheel test.
4. EN 1097-9:1998 includes the resistance to wear of studded tires on coarse aggregates as measured in the Nordic ball mill test.

In spite of the drawbacks of the L.A. abrasion test, it is a fairly fast test with known variability; and as a general test, it does provide a means of identifying coarse aggregate that may degrade readily during handling.

Wet Degradation and Attrition Tests

There has been increased interest in abrasion and attrition procedures run in a wet environment. The California durability index test for fine and coarse aggregate, which measures the tendency of an aggregate to break down into plastic clay-like fines, has been standardized as ASTM D 3744. Some aggregates degrade in a different manner in the presence of water than those in dry test methods [15]. For concrete aggregates, the principal value of such tests are identification of aggregates that may create undesirable fines during handling, batching, and mixing that could affect concrete properties, such as strength and water requirement. These tests also have significance for base courses and aggregate surfaced roads where the possibility of interparticle movement in the presence of water may occur over the period of service of the material.

Liu [16] reported on research that led to the development of an underwater abrasion test method (ASTM C 1138) designed to assess the abrasive effect of waterborne gravel, rocks, and other debris traveling over concrete surfaces in hydraulic structures. He concluded that the results of this test were correlated with concrete strength and the hardness of the aggregate. However, the underwater abrasion of concrete was not related to the L.A. abrasion of the coarse aggregate.

Highway Research Board Circular 144 [17] indicates use by state transportation agencies of various test methods relating to aggregate durability (other than freezing and thawing). The L.A. abrasion (a dry test) was used by the great majority; however, a number also indicated use of some type of wet abrasion or attrition procedure including: Durability Index, Washington Degradation Procedure, Idaho Degradation, Nevada Air Test, the Oregon Air Test, and modified L.A. abrasion tests using water. A few relied on petrographic analysis to identify mineral constituents that are associated with rapid weathering. Almost all agencies reported some degradation problems; in most cases, the problems were termed moderate.

Breese [18] gives a history of the development of wet abrasion tests in the Western United States. He experimented with different procedures designed to detect comminution degradation and alteration degradation. He concluded that Nevada had a number of degradation susceptible aggregates and that the California durability index test is not as harsh as some of the other procedures. Ekse and Morris [19] compared properties of fines produced from a standard L.A. abrasion test and an extended 4-h test without the steel balls. The aggregates were crushed basalt, a pit-run river gravel, and a crushed river gravel all from sources in the State of Washington. Plasticity index of the fines produced from the extended test were respectively 6.8, 2.0, and 1.0. Fines from the standard L.A. abrasion test were found to be nonplastic.

Minor [20] describes the development of the Washington degradation test that was prompted by the poor performance of several basalt crushed stones and volcanic gravels that gave losses in the standard L.A. abrasion test ranging from 12–50. These aggregates contained altered minerals that contributed to rapid weathering. In studies at the Pennsylvania DOT and at Purdue University in Indiana [21,22] to help identify the properties of shales that might help determine suitability for its use in embankment or as a granular material, the Washington degradation test was one of the tests that interested these researchers. In addition to the Washington test, the ultrasonic disaggregation test and the ethylene glycol soak test used by the Corps of Engineers (CRD-C 148, Method of Testing Stone for Expansive Breakdown on Soaking in Ethylene Glycol) were good indicators of performance. At Purdue, the Swiss Hammer was found to be a potentially useful tool in identifying hard shales. High rebound readings were obtained for two shales that showed less than 40 % loss in the L.A. abrasion test and that gave good results in the Washington Test.

The durability index for fine and coarse aggregate (ASTM D 3744) was developed to measure breakdown during construction and normal use under traffic conditions. It is intended to complement the sand equivalent, cleanness value, L.A. abrasion, and sulfate soundness procedures. There is little correlation between the durability index and the L.A. abrasion test. The durability index tests start with a washed sample and then measure the quality of the fines generated from interparticle abrasion during a wet agitation period. Figure 1 shows durability index values versus L.A. abrasion.

In work with the Washington degradation test, Goodewardane [23] looked into sources of variation. He concluded that both surface attrition and leaching of clays from the aggregate pores were involved in the test. The value obtained is very dependent on the surface area of the charge; finer samples will indicate more degradation. Aggregate quality improved with repeated agitation cycles as found by Hveem and Smith [24].

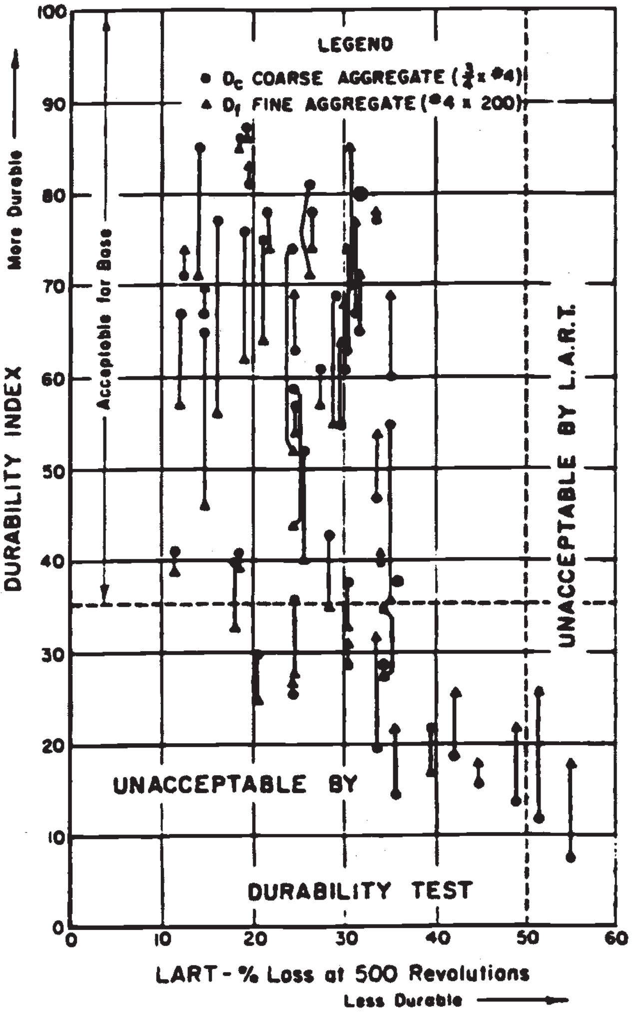

Fig. 1—L.A. abrasion loss versus California durability index [24].

Weathered basalt was found to produce lower durability results than fresh basalt.

Degradation or attrition of aggregates during mixing and agitating of concrete has been the subject of several investigations. In studies by Slater [25] and more recently by Gaynor [26] and McKisson [27], it was identified that during prolonged mixing the fine aggregate with its much greater surface area tended to break down by attrition much more readily than the coarse aggregate. In 2 h of mixing in a laboratory mixer in one case, and 2 h and 40 min in another case, little change was noted in the coarse aggregate grading. The amount of minus 150-μm (No. 100) material generated from the fine aggregate depended on aggregate hardness. A fine aggregate known to break down, caused an increase in minus 150-μm (No. 100) material of almost 12 % in 2 h in one study. In the other study, a soft limestone sand caused an increase in minus 150-μm (No. 100) size of about 6 % in 160 min. Hard quartz sand only caused an increase of 1 % in the same 160-min mixing period.

A number of tests have been used to measure the propensity of fine aggregates to degrade due to attrition during concrete mixing [28–31]. In 1990, ASTM C 1137 was approved. The method is based on work reported in the preceding references using a vane-type attrition chamber, mounted in a drill press and turned at 800 rpm. However, the test is not used much, and has not been recently reapproved or revised.

The Micro-Deval test is now being used in Canada, Europe, and the U.S. for the evaluation of coarse and fine aggregates [31–34]. It is a wet attrition test developed in France during the 1960s using a 500-g specimen for coarse aggregates. Modifications were made in Canada to a larger 1500-g sample of coarse aggregate to improve the repeatability of the test [33], and these procedures have been used in the U.S. [35,36]. The test provides a combination of abrasion, attrition, and grinding with steel balls in the presence of water. It has gained popularity in Canada in evaluating the suitability of aggregates subject to weathering and abrasive action and has been included as an alternative test for aggregates for concrete. Subsequent research in the U.S. has shown its potential relationship to the performance of different types of coarse aggregate in highway applications, and it is has been standardized as ASTM D 6928 for the Resistance of Coarse Aggregate to Degradation by Abrasion in the Micro-Deval Apparatus. The equipment is readily available, and a number of state transportation departments are evaluating its use. For fine aggregate, the Ministry of Transportation in Ontario (MTO) modified the test.

The Micro-Deval tests have been standardized by the Canadian Standards Association as CSA A23.2-23A for fine aggregate and 29A for coarse aggregate. CSA also has included alternative specification limits for Micro-Deval loss of a maximum of 20 % loss for fine aggregate. For coarse aggregate the Micro-Deval loss limit is 14 % for use in concrete exposed to freezing and thawing and/or to chlorides in service. For other exposures, the coarse aggregate Micro-Deval loss limit is 17 %. ASTM is now considering the fine aggregate test for standardization.

The fine aggregate Micro-Deval test uses a 700-g sample of sand washed to remove material passing the 75-μm (No. 200) sieve. The sample is placed in a steel jar mill with steel balls and water and rotated at 100 rpm for 15 min. Loss is expressed as the percentage of minus 75-μm (No. 200) material generated in the test. In the Ontario study, the Micro-Deval test was found to correlate with sulfate soundness and water absorption of fine aggregate. They also included the ASTM C 1137 attrition test and a similar Ontario (MTO) attrition test with a more robust impeller and a reduction in the rpm from 850 to 390 rpm to reduce splashing problems. Table 1 shows a comparison of the three tests, both amount of fines produced and the coefficient of variation. It was found that the Micro-Deval test had the lowest within-laboratory variability, and it was relatively insensitive to fine aggregate grading. It is being used in Ontario to evaluate fine aggregate.

In a similar Ontario study of coarse aggregate tests, the Micro-Deval coarse aggregate test was evaluated and compared to three British Standard Tests (BS 812) for aggregate impact value, polished stone value, and aggregate abrasion value. In the Micro-Deval test for coarse aggregate, 1500 g of a graded sample are soaked and then placed with 9.5 mm (3/8-in.) steel balls and water in a 5-L steel jar mill that is rotated at 100 rpm for 2 h. The loss is measured by the amount passing the 1.18-mm (No. 16) sieve. Also included in the research were the Ontario Test Method for unconfined freeze-thaw of coarse aggregate, the magnesium sulfate soundness test, and the L.A. abrasion tests. The Micro-Deval test correlated with sulfate soundness, but it had lower variation (see Fig. 2). It was concluded that the Micro-Deval and unconfined freeze-thaw tests approximate the deterioration due to weathering and that, when used in conjunction with water absorption and petrographic examination, they are better predictors of field performance for marginal aggregates. Rogers [33] discusses the maximum loss specifications used in Ontario for the Micro-Deval tests for concrete, asphalt, and base course applications of both coarse and fine aggregate, and in the U.S. NCHRP, research has been conducted relating the Micro-Deval test to the performance of aggregates in asphalt concrete and in unbound

TABLE 1—Within-Laboratory Variability of Attrition and Micro-Deval Tests of Fine Aggregate[a]

	Natural Sand		Shaley Limestone Screenings	
	Mean Loss	Coefficient of Variation	Mean Loss	Coefficient of Variation
Micro-Deval	13.8 %	1.9 %	23.4 %	1.1 %
ASTM attrition	13.3 %	11.0 %	38.8 %	6.8 %
MTO attrition	4.1 %	14.1 %	20.1 %	5.4 %

[a] Number of replicates = 10.

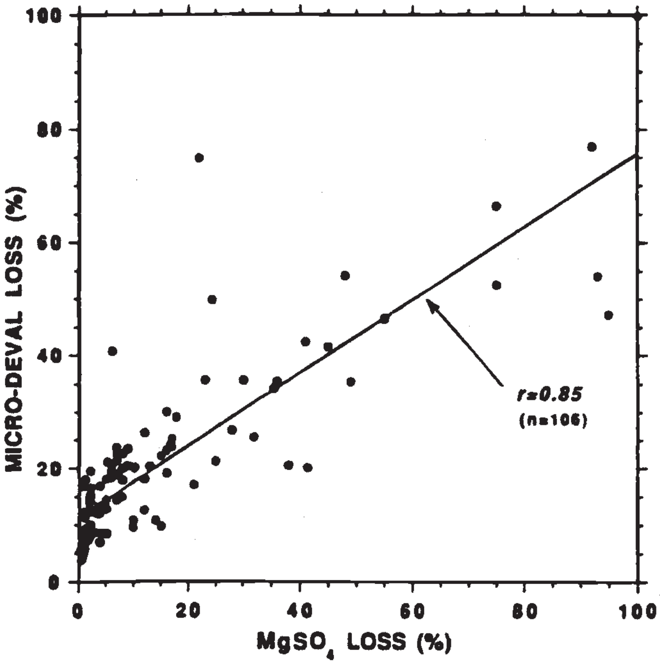

Fig. 2—Correlation of Micro-Deval abrasion against magnesium sulfate soundness of coarse aggregates [32].

bases [35,36]. In the Ontario work [32], the British aggregate impact value test was seen as a practical substitute for the L.A. abrasion test (see Fig. 3).

North American railroads have been using mill abrasion tests of coarse aggregate for ballast, with procedures differing from the Micro-Deval test and with the mill abrasion loss reported as the amount passing the No. 200 sieve after the test. In an effort to standardize the procedure and look at sources of testing variability, a railway engineering group published the Mill Abrasion Test Method [37] and the results of round-robin testing using six sources of crushed stone ballast coarse aggregate tested in seven different laboratories. Higgs [29] reported another fine aggregate wet attrition test using a paint shaker to attrition a 1000-g regraded sample of fine aggregate. He investigated a number of basalt fine aggregates and found that those causing rapid slump loss in concrete yielded more than 8 or 9 % minus 75-μm (No. 200) material in the shaker test. These sands also had high magnesium sulfate soundness losses.

Strength of Aggregate Material

Measurements of unconfined and confined compressive (or crushing) strength of rock and mineral specimens drilled or sawn from rock ledges, or in some instances cobbles or boulders from gravel deposits, are quite often made in connection with rock mechanics investigations for foundations, tunnels, and various mining applications. Certain inferences can be drawn from these data as to the nature and distribution of the strengths of the materials constituting aggregate particles. However, there are several problems involved in translating this information to the behavior of aggregate in concrete: (1) conditions of confinement in concrete are certainly different than those of unconfined tests, and triaxial tests may not truly represent the situation in concrete; (2) strength tests of rock specimens usually involve measures to distribute the stress evenly over the entire cross section, whereas in concrete, localized stress concentrations may develop due to the nonhomogeneous nature of the concrete; and (3) rock properties are most often reported for specimens that have a minimum of flaws, cracks, and fissures, but the strength of an individual aggregate particle can be influenced greatly by such defects.

Udd [38] gives a review of the methods used in testing rock and some examples of the range and variability of results. Compressive strength increases with confinement, and both elastic and plastic deformations occur. There are many sources for data on compressive strength of rock and in some cases tensile strength or modulus of elasticity. Bond [39] shows compressive strengths of small cubes and cylinders cut from rock material. For limestone, the values ranged from 90–270 MPa (13 000–39 000 psi); for traprock, 105–235 MPa (15 000–34 000 psi); and for granite, 85–275 MPa (12 000–40 000 psi). These values represent ranges from a number of sources. He also reports compressive strength results from one gravel source ranging from 165–235 MPa (24 000–34 000 psi). The great bulk of the strength data available is for stone materials where specimens of the required size can be obtained, and usually the material is less variable than gravel deposits.

Tests of two types of rocks used as aggregate by Iyer et al. [40] yielded, for a limestone source, compressive strengths of 105–200 MPa (15 000–29 000 psi) and tensile strengths of 2.1–6.2 MPa (300–900 psi), and for a quartzite source, compressive strengths of 340–470 MPa (49 000–68 000 psi) and tensile strengths of 7.6–15.8 MPa (1100–2300 psi). Kaplan [41] tested gravel, limestone, granite, and basalt aggregates and obtained compressive strengths ranging from about 160 MPa (23 000 psi) for the gravel and the granite on the low end to 305 MPa (44 000 psi) for the basalt on the high end. The tensile strength measured for the gravel, limestone, and granite ranged from about 6.2–11.7 MPa (900–1700 psi). The basalt had a tensile strength of 15.2 MPa (2200 psi). Of the almost 1000 compressive strengths reported by Woolf, for rock from which crushed stone coarse aggregates are made, in his state-by-state summary [42], only

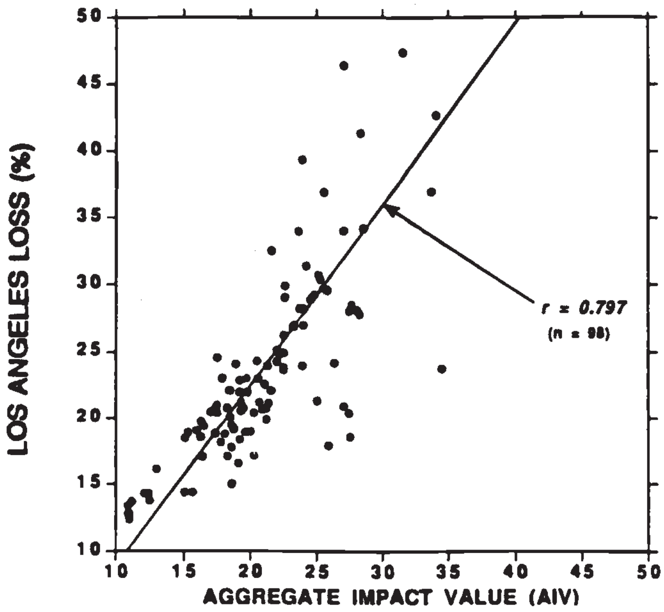

Fig. 3—Relationship between loss in the Los Angeles test and aggregate impact value of coarse aggregates [32].

about 60 results indicated compressive strength levels less than 70 MPa (10 000 psi).

From the foregoing, it is apparent that the great majorities of the aggregate-making rock materials are strong in compression, over 70 MPa (10 000 psi), and like all brittle materials relatively weak in tension. There is also a proportion of aggregates used for concrete that are weak enough, less than 70 MPa (10 000 psi), that their strength characteristic may be an important limitation on the properties of concrete made with these materials. Kaplan [41], in using a number of strong aggregate materials in concrete, concluded that "no relationship between the strength of the coarse aggregate and the strength of concrete was established. This finding should not be taken to mean that aggregates of low strength will not affect concrete strength." Johnston [43], using a 48 MPa (6900 psi) sandstone coarse aggregate in concrete, and Collins and Hsu [44], using a 28 MPa (4000 psi) oolite coarse aggregate in concrete, were both able to produce concrete over 40 MPa (6000 psi) at 28 days age. These data suggest, as does broad experience with lightweight structural aggregates and experience using Florida limestone [45], volcanic cinders [46], and coral in concrete [47], that aggregate strength need only be of the same order of magnitude as the concrete strength needed. Tanigawa [48] in his model analysis with various aggregate and mortar strengths concluded that the best results in terms of delay in formation of bond cracks at the coarse aggregate-mortar interface were obtained when the aggregate and mortar were about the same strength.

It is recognized that coarse aggregate-to-mortar bond is extremely important to concrete strength and that a reasonably high-strength aggregate with excellent bonding characteristics is necessary in the production of very high-strength concrete over 55 MPa (8000 psi). The strength of individual fine aggregate particles is apparently not too important as long as some minimum level is achieved, principally so that it does not break down and create excess fines and surface area during concrete production. Bond is not as important with fine aggregate because of its large surface area-to-volume ratio. The influence of fine aggregate on concrete strength is almost exclusively through its shape, texture, and grading effects on mixing water demand needed for workability.

The significance of strength tests of aggregates is not well established. There are no ASTM test methods or specification requirements in this area. The usual practice is to evaluate the aggregate's performance in concrete to see if it will produce concrete of the needed strength. Aggregate strength may be an indicator of other aggregate properties, but relationships have not been established.

Elastic Properties

Modulus of elasticity and related values such as the Poisson's ratio of concrete aggregates are not included in aggregate specifications, and there are no ASTM aggregate test methods designed to yield this information. The influence of aggregate source on concrete modulus of elasticity is determined normally by testing concrete mixtures containing each of the aggregates. As is the case for compressive strength, a good deal of information from rock mechanics investigations is available concerning rock deposits [49–51], but little is known about the elastic properties of gravel or sand particles other than what can be inferred from tests of parent rock material. A number of investigators has also determined rock modulus of elasticity in connection with researches of aggregate and concrete properties [40,41,43,44]. There has been a good deal of activity in the more theoretical area of developing equations to predict the resulting modulus of elasticity of a composite material from a knowledge of the elastic properties of the aggregate and the matrix [52–56].

Figure 4 gives an indication of the range of Young's modulus of elasticity for various rock types. Both static and dynamic E data are included. Generally, the dynamic values are higher, and there is a good linear relationship between dynamic and static values determined in the laboratory. A direct linear relationship has also been found between rock compressive strength and modulus of elasticity [50,51].

Both in compression and tension, stress-strain plots for stone [49] and for cement paste [43,57] are normally fairly straight lines, indicating that the aggregate and matrix are reasonably elastic. Concrete and mortar, on the other hand, have a curved stress-strain plot when the stress exceeds about 30 % of ultimate strength. This is due to the formation of bond cracks and slipping at the aggregate paste interface. With a curved stress-strain plot, the modulus of elasticity computed for the concrete will vary depending on which of the several recognized definitions is used. Because of this and because unknown factors can affect the influence of the paste-aggregate bond on the stress-strain curve, there is no simple relationship between aggregate modulus of elasticity and concrete modulus of elasticity.

It does not always hold that an increase in aggregate modulus of elasticity will increase the *E* of the concrete even if the properties of the paste are held relatively constant. An example of this is contained in Houghton's paper [58], where natural rounded aggregate with an *E* of about 41 400 MPa (6×10^6 psi) produced concrete with *E* ranging from 28 000–34 000 MPa ($4–5 \times 10^6$ psi) at 90 days compared to a quarried aggregate with an *E* of about 62 000 MPa (9×10^6 psi) that produced concrete with an *E* of 21 000–28 000 MPa ($3–4 \times 10^6$ psi). Hirsch [56], however, shows data where aggregates with *E* values of about 14, 34, 62, 76, and 207×10^3 MPa (2, 5, 9, 11, and 30×10^6 psi) were used in different proportions in concrete indicating "that the modulus of elasticity of concrete is a function of the elastic moduli of the constituents. An increase or decrease in the modulus of either the aggregate or matrix constituent will produce a corresponding effect on the concrete." The equations advanced by Hirsch and others are complex and include, of necessity, various assumptions.

Less work has been done on the tensile modulus of elasticity. Johnston [43,59] gives values of tensile modulus of elasticity of rock. The values are somewhat lower than the corresponding compression values. Houghton [58] discusses the concept of predicting the tensile strain capacity of mass concrete so that tensile strains caused by thermal volume changes can be accommodated by the concrete without cracking. Relationships with age for tensile strength and modulus of elasticity enter into the determination of strain capacity for rapidly loaded concrete. Creep is a third factor that enters into the estimate when stress is applied over long periods.

Hardness, Wear, Frictional Properties

"Hardness is the single most important characteristic that controls aggregate wear," according to Stiffler [60]. Mineral and rock materials when subjected to rubbing, scratching, and gouging actions, particularly with the aid of an abrasive, are worn

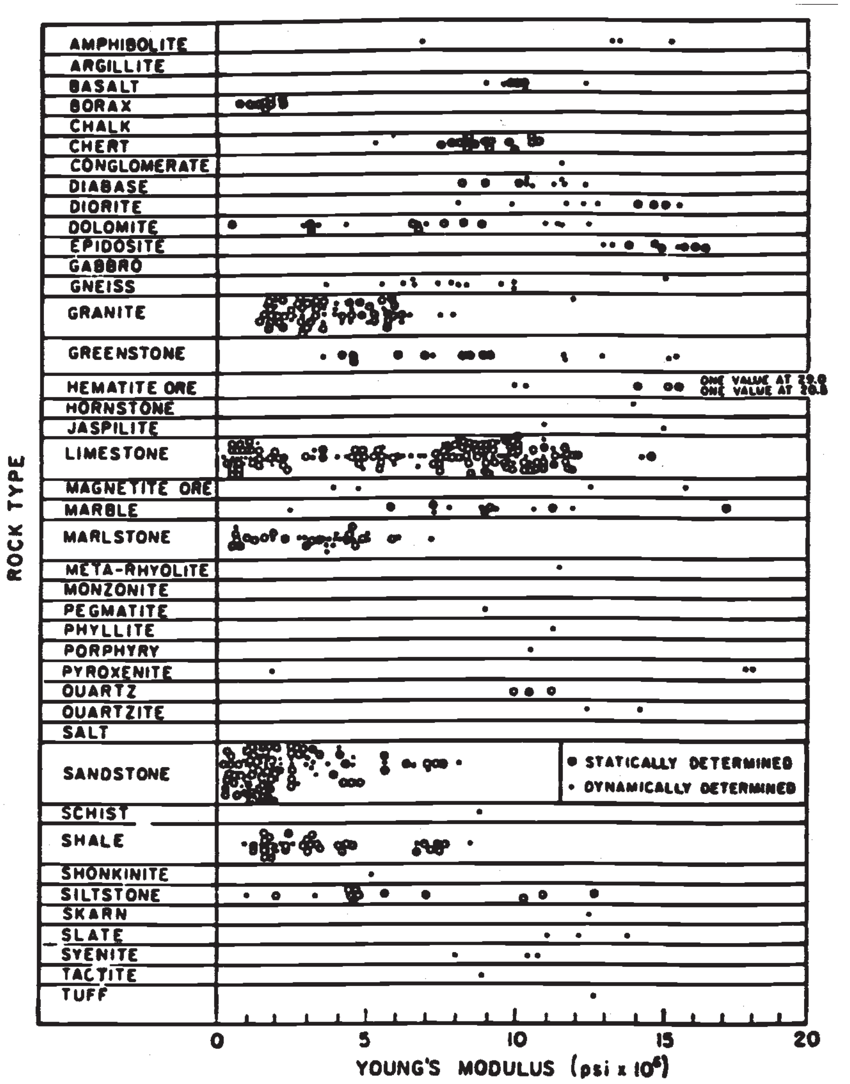

Fig. 4—Relationship between rock type and Young's modulus: 1 psi × 10^6 = 6895 MPa [50].

down due to the scratching and pitting that occurs. Stiffler has observed that, unlike metals, which are just scratched when subjected to the movement of an abrasive on the surface under a load, minerals are pitted as well. This is apparently due to mineral grains or particles being pulled from the matrix. There are many potential ways of measuring the hardness [61].

1. Indention Hardness–Vickers hardness and Rockwell hardness [62].
2. Rebound Hardness–Shore scleroscope and Swiss rebound hammer.
3. Scratch Hardness–Moh's hardness scale.
4. Wear or Abrasion Hardness–Dorry hardness where a core of rock is subjected to wear with an abrasive on a revolving horizontal wheel [63] and British aggregate abrasion value (British Standard 812).
5. Rate of cutting or drilling.

Hardness is a term used often in describing desirable aggregate properties, but there has been little agreement as to how measurements should be made and whether hardness should be a property that is specified for concrete aggregates. At one time, ASTM had a scratch hardness test, but it was withdrawn because of the high variability of the results, no way to correct the test, and the inability to correlate results with performance. ASTM C 33 has no limits on soft particles and no other provisions referring to hardness.

As a general rule, concrete strength has been found to be the most significant factor in rate of wear of concrete [64], but fine and coarse aggregate may be an important factor in some instances for concrete surfaces subjected to heavy traffic or abrasive forces, particularly lower-strength concrete. Initially, the hardness of the fine aggregate is important. The coarse aggregate will become involved only if there is enough loss of surface to expose a significant amount of the coarse particles.

Polishing is a special form of wear where abrasive size is quite small, such as typical road grit at 10–40 μm; and the action is such that any texture such as existing pits, gouges, or scratches are smoothed and polished gradually. This can happen particularly to exposed cement paste and the top surfaces of fine or coarse aggregate particles. Wear is "waste or diminish by continual attrition, scraping, percussion or the like," [65]. It is any removal of particles that produces shape changes on a microscopic or macroscopic scale [60].

The mechanism of concrete wear can vary [6]. For example, in concrete abrasion tests using the steel balls or dressing wheel, harder aggregates retard abrasion loss and stand out from the surface. A softer limestone aggregate, on the other hand, wears along with the paste in these tests. The reverse is true for a shot blast test where the limestone cushioned the abrasive shot and decreased the wear compared to more brittle aggregate.

Wear of highway pavements is due in large measure to the fine mineral abrasive present on the pavements. Studded tires have also been an important factor in road wear. With the phasing out of the studs in many areas, this factor has diminished.

In Canada and the United States, studded tire wear was studied intensively. Smith and Schonfeld [66] found that in an area where almost one-third of the passenger vehicles was equipped with studs, over the winter of 1969–70, wear on both portland cement concrete and bituminous concrete approached 5–8 mm in one season. Pavements with aggregates of similar hardness to the matrix showed uniform wear. With harder aggregates, the matrix was preferentially worn down around them until the particles were dislodged by the studs because of lack of embedment. It was determined that studded tires did not change the frictional properties much. In concrete containing a sand with a substantial amount of soft minerals, pavements with traprock and limestone coarse aggregates wore at about the same overall rate even though the mechanism was somewhat different. When a 100 % silica sand was used with the harder traprock, wear of both the matrix and the overall pavement was reduced.

Keyser [67] found that the age of the mortar and whether or not limestone coarse aggregate was used were significant factors in studded tire wear studies. Other aggregate types, size of coarse aggregate, and concrete compressive strength were not significant for the materials used. In reporting test track studies, Preus [68] showed that studs produced more than 100 times as much wear as regular tires even when sand and salt were applied to the surface. Rosenthal et al. [69] show photos of stud wear marks. They confirmed that studs tend to skid over hard aggregate and leave grooves in softer material.

In a review of pavement wear testing, Stiffler [60] reports that sliding movement between rubber and pavement of up to 6.4-mm (1/4-in.) occur in normal rolling tires. Therefore, many pavement-wear researchers, as well as ASTM D 3319 (British wheel) and ASTM E 660 (circular track polishing machine), utilize a wearing mode of rubber on pavement specimens, or rubber on mineral aggregate samples, with an abrasive supplied to simulate road grit. Polishing occurs when fine abrasive is used and the surface is made up of materials of similar hardness. Regeneration of the surface can occur when two components of differing hardness are present and particles of the weaker, softer material wear faster, causing the harder material to protrude and eventually be undercut and torn out, leaving an unpolished surface.

Frictional properties of pavement surfaces in wet weather depend on microtexture (amplitude less than 0.5 mm) and also on macrotexture (amplitude more than 0.5 mm) if significant speeds are involved. The definitions for texture are taken from ASTM Definitions of Terms Relating to Traveled Surface Characteristics (E 867). Macrotexture is controlled initially by concrete finishing operations, and it is important in removing excess water from between the tire and pavement. Microtexture is controlled by the polishing tendency of the exposed cement paste or aggregate surfaces. Meyer [70], in using a number of concrete finishing textures, silica gravel and limestone coarse aggregates, and silica or lightweight fine aggregate, found good frictional properties in all cases. The lightweight fine aggregate did wear faster.

In other studies where calcareous fine aggregates were used in concrete, low frictional properties have been found [71]. For concrete pavements exposed to normal traffic wear that does not expose much coarse aggregate during the life of the pavement, it is the fine aggregate that tends to control the polishing rate and microtexture [72]. Colley et al. [71] in tests at the Portland Cement Association showed, after polishing, that the energy needed to turn a rubber tire against a fixed concrete specimen decreased markedly as the siliceous particle count in the fine aggregate was decreased below 25 %. Mullen and Dahir [73] studied the wearing and polishing characteristics of a number of aggregate sources. They found no general correlation of properties for all aggregates. For a granite aggregate, there was an inverse relationship between wear resistance and frictional properties; and, for sandstone, synthetic aggregate, and one mountain gravel, friction increased as absorption and surface capacity of the particles increased. For carbonate aggregates, a direct relationship between sand-size, acid-insolubility, residue, and friction properties was implied.

Some highway agencies [74] have advocated the use of ASTM D 3042 as a tool in selecting fine or coarse carbonate aggregates for use in surface courses. Insoluble material may be clay or siliceous material. A higher insoluble residue retained on the 75-μm sieve indicates a higher percentage of harder and perhaps more polish resistant minerals. The ASTM D 3319 (British wheel) procedure involves polishing oriented coarse aggregate particles held by an epoxy backing using a rotating rubber tire running against the specimens that are mounted around the perimeter of a second wheel. Abrasive and water are fed onto the tire-specimen interface at a constant rate. Degree of polish is measured using ASTM Method of Measuring Surface Frictional Properties Using the British Pendulum Tester (E 303). Several other laboratory procedures use a circular track with a rubber-tired wheel for evaluating pavement materials and mixtures for polishing.

Franklin and Calder [72] report results of frictional properties research using several types of fine aggregate that are in decreasing order of performance: (1) calcined bauxite fines with both good polishing and abrasion resistance; (2) a gritty sandstone material with good polish resistance, but poor abrasion resistance; (3) a flint sand with poor polish resistance, but good abrasion resistance; and (4) a carbonate fine aggregate with both poor polishing and abrasion performance.

Weller and Maynard [75] at the British Road Research Laboratory developed an accelerated wear machine for pavement samples. They use a dry wearing cycle of 50 h with a small flint gravel as an abrasive followed by 5 h of wet polishing with a fine emery abrasive. The most important characteristic of the sands tested is hardness. Harder sands stand out from the surface and show higher frictional properties after dry wearing than after wet polishing. Conversely, for soft dolomite and limestone fine aggregates, the particles were worn flush with the cement paste during the dry wearing cycle; and since portland cement paste polishes more during the dry cycle and gains frictional properties during wet polishing, the friction numbers of the pavement surfaces containing the soft fine aggregate were lowest after dry wearing and improved during wet polishing.

Impact on Specifications

Tests that directly measure degradation resistance, strength, or toughness characteristics of an aggregate are not generally referenced in specifications for concrete aggregates in the United States. The one exception is the L.A. abrasion test for coarse aggregate that is used in ASTM C 33 and ASTM Specification for Aggregates for Radiation-Shielding Concrete (C 637) as well as in a large number of agency specifications.

The practice is first to rely more on past service record and judgment for those aggregates that have performed satisfactorily, and, second, to test the properties of concrete made with the aggregate in question if special abrasion, impact, or strength properties are required. These methods can be used to evaluate the performance of alternative aggregate sources as well as various mix proportions. With respect to aggregate strength, ASTM Specification for Lightweight Aggregates for Structural Concrete (C 330) requires the aggregate to be of sufficient strength to yield certain minimum compressive and tensile splitting values when tested in concrete.

With respect to an aggregate's effect on frictional properties of pavements, many state highway agencies rely on friction trailer performance history of highway surfaces and assign aggregate classifications on that basis. Others use petrographic or acid solubility data or both to classify aggregates, and several states use the British wheel (ASTM D 3319) as a specification tool for aggregates for highway surfaces [74,76].

Concluding Remarks

There is a good deal of history and data available concerning the measurement of physical properties of aggregates and attempts to relate these properties with concrete properties. However, the evaluation and testing of concrete containing alternative materials remains the best approach to assuring performance when special abrasion resistance, strength, toughness, or frictional properties are needed.

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Petrographic Evaluation of Concrete Aggregates

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Preface

CHAPTERS AUTHORED BY R. MIELENZ ON PETROgraphic examination of concrete aggregate were included in the four previous editions of this ASTM Special Technical Publication, *ASTM STP 169, ASTM STP 169A, ASTM STP 169B,* and *ASTM STP 169C.* Experience and literature pertinent to examination of all types of concrete aggregate are cited as appropriate. Among the revisions to this addition, a major section on qualifications of personnel performing petrographic examination for the purposes of qualifying aggregate for use in portland cement concrete has been added. This chapter is dedicated to Dr. Richard C. Mielenz for his long and dedicated service to ASTM Committee C9 on Concrete and Concrete Aggregates.

Introduction

Petrographic examination of concrete aggregate is visual examination and analysis in terms of both lithology and properties of the individual particles. The procedure requires the use of a hand lens and petrographic and stereoscopic microscopes. Less commonly, X-ray diffraction, differential thermal analysis, or electron microscopy are used to supplement the examination using optical microscopes. By petrographic examination, the relative abundance of specific types of rocks and minerals is established; the physical and chemical attributes of each, such as particle shape, surface texture, pore characteristics, hardness, and potential chemical reactivity are described; coatings are identified and described; and the presence of contaminating substances is determined—each in relation to proposed or prospective conditions of service in concrete constructions.

As will be discussed subsequently, petrographic examination contributes in several ways to the investigation, selection, testing, and monitoring of aggregate uniformity. Consequently, the method is progressively being applied more widely. Since 1936, all aggregates used in concrete construction by the Bureau of Reclamation, U.S. Department of the Interior, have been examined petrographically as part of the basis for their selection [1,2]. The method has been applied similarly by the Corps of Engineers since before 1940 [3]. Coarse aggregates proposed for use in either portland-cement concrete or bituminous concrete are examined by petrographic methods in laboratories of the Ministry of Transportation–Ontario, and specifications governing acceptance are based upon the results [4]. Petrographic examination is performed also by several other agencies of the U.S. government and state departments of transportation, and may be obtained through some testing and engineering laboratories.

In 1952, ASTM Tentative Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295) was accepted and was adopted as a standard in 1954. Minor modifications were made subsequently, most recently in 2004. The document has been published as the Guide for Petrographic Examination of Aggregates for Concrete in the 1991 *Annual Book of ASTM Standards.* Petrographic examination is also cited in ASTM Specification for Concrete Aggregates (C 33).

The abundant data obtained and the rapidity with which petrographic examination can be completed justify more general use of the method in investigation, selection, manufacture, and use of concrete aggregates. However, in all cases, the petrographer should be supplied with available information on the conditions of service to which the concrete is to be exposed.

This paper summarizes the objectives and applications of petrographic examination of aggregates with reference to gravel, sand, crushed stone, blast-furnace slag, the most common types of lightweight aggregates, and aggregates produced by the recycling of hardened concrete from constructions. Special attention has been given to developments in this field since the publication of *ASTM STP 169C* in 1994. Techniques of the examination are treated briefly because instructions have been published elsewhere [3–11] and are included in ASTM C 295.

Qualification for Concrete Petrographers

Education

The petrographic examination of aggregate is a forensic treatment of material for potential use as concrete aggregate and brings to use many forms of analysis to provide an overall estimation of the quality of the aggregate. Chemistry, physics, engineering properties of materials, and geochemistry are elements that are addressed in a typical petrographic report. Differing educational backgrounds can provide the basic tools required to perform a petrographic examination of aggregate

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for use in the production of concrete. Historically a BS degree in geology provided the basic tools required to execute the petrographic examination, but with the advent of more specialization in undergraduate education, traditionally required courses such as optical mineralogy, igneous petrography, metamorphic petrography, sedimentary petrography, etc. have fallen from the basic BS geology requirements in many institutions. An individual should have basic geological background and specific knowledge on techniques to perform an examination as outlined in ASTM C 295.

Experience

Petrographic examination provides a consolidated interpretation of all data produced in the testing and examination phase of any program designed to investigate potential aggregate sources. The requirements are often different for the various clients depending on their specific needs. For example, state highway departments focus on pavements and slabs, while some customers may focus on marine concrete or architectural concrete. The petrographer must remain vigilant as to any unusual details in test requirements of a project. Agencies sometimes use standard specifications and test methods, but may require different sampling or sample sizes for the testing.

An example is the military specifications for airfield pavements. They basically rely on ASTM methods, but sample sizes are much larger in order to insure the detection of relatively small amounts of deleterious materials.

Often the petrographer is the central figure in the aggregate investigation. The petrographer interprets the results and draws conclusions as to the acceptability of an aggregate source for use in the intended project. The petrographer must have working knowledge of all tests associated with the performance of aggregate. The conclusions of a petrographic examination are often to recommend more definitive testing such as ASTM C 1260 for alkali-silica reactivity or ASTM C 586 for alkali-carbonate reactivity or recommendations as to the performance and the potential use of the aggregate source. These additional tests are part of the knowledge base and experience that is expected of the petrographer in making judgments and recommendations.

The petrographer may be the only individual in the organization that has this unique ability and knowledge, thus it is very important that this individual have the experience necessary to make these critical decisions. This working knowledge is best obtained while working in the field of petrography under the guidance of an experienced concrete petrographer. A qualified petrographer should meet the same experience requirements in his or her field as those required of laboratory directors. ASTM Standard Practice for Laboratories Testing Concrete and Concrete Aggregates for Use in Construction and Criteria for Laboratory Evaluation (C 1077) requires laboratory directors to have a minimum of five years of experience.

Purpose of the Petrographic Examination of Concrete Aggregates

Preliminary Determination of Quality

Preliminary petrographic examination of concrete aggregate is performed either in the field or in the laboratory as an adjunct to geologic examination, exploration, mapping, and sampling. The examination assists the geologist or materials engineer in determining the extent to which consideration of an undeveloped deposit is justified. Also, the preliminary petrographic examination indicates the relative quality of aggregates from alternate sites. By revealing variations in the material, examination of exposures or cores from pilot drill holes establishes the minimum program of exploration and sampling necessary for acceptance or rejection of the deposit.

Establishing Properties and Probable Performance

Petrographic examination is primarily a supplement to the acceptance tests. Probable performance of concrete aggregate is estimated in two general ways by petrographic examination. First, the examination reveals the composition and physical and chemical characteristics of the constituents. From this information, one can estimate how these properties of the aggregates will affect mixing, placing, consolidation, and development of early-age properties. This examination also identifies features of the aggregate that affect long term durability.

Some aggregate features that may affect concrete placement include organics in the aggregate affecting air-entrainment, soluble salt affecting setting time, angularity in fine aggregate affecting bleeding characteristics, absorption by porous particles causing slump loss, angular particles increasing flexural strength in concrete, soft particles breaking down in mixing adding fines and reducing workability, and the presence of expansive clays causing stiffening of the concrete mixture. Long-term effects including the probable response of the aggregate to such phenomena as attack by cement alkalis, freezing-thawing, wetting-drying, heating-cooling, or high temperatures usually can be estimated. The rapidity with which the petrographic examination predicts potential alkali reactivity of aggregate is especially valuable because of the widespread occurrence of this condition and the long time commonly required by realistic tests of concrete or mortar.

The second way the petrographic examination helps to estimate performance is to provide a basis of comparison with aggregates from other sources. The petrographic examination establishes the fundamental nature of aggregates so that aggregates from unfamiliar sources can be compared with aggregates upon which information is available. This application is discussed subsequently.

Correlation of Samples with Aggregates Previously Tested or Used

Detailed petrographic examination is the only procedure that permits comparison and correlation of samples with aggregates previously used or tested. Thus, data and experience previously obtained by use and long-time tests of similar aggregates can be applied in the selection of materials proposed for current work, even though the materials come from new sources.

By relating the sample to aggregates previously used in construction, aggregate determined to be unsound by standard tests may be found adequate, or, conversely, aggregate indicated to be sound in standard tests might be found unsatisfactory for the intended use.

For example, gravel in certain deposits near Jackson, Michigan meets usual specification requirements for soundness, abrasion resistance, and content of soft particles, yet produces objectionable popouts in pavements after exposure for two winters (Fig. 1). Petrographic examination of the gravel and of individual particles producing popouts during service has identified the unsound rock types and the critical minimum size required for failure. Examination of proposed

Fig. 1—Popouts produced by claystone, shale, and chert in concrete pavement near Jackson, Michigan (courtesy of Michigan Department of Transportation).

materials will demonstrate the presence or absence of such particles and will thus indicate, in the light of other data, whether the aggregate should be accepted, rejected, or subjected to special tests.

Petrographic examination of aggregate may be a part of an investigation of concrete constructions as described in ASTM Practice for Examination and Sampling of Hardened Concrete in Constructions (C 823) and ASTM Practice for Petrographic Examination of Hardened Concrete (C 856). The examination may be applied to determine the source of aggregate used in the constructions and whether it was obtained from an approved or an unapproved source.

Selecting and Interpreting Other Tests

All of the properties of aggregates influencing performance of concrete in service ordinarily are not evaluated by test before selection of the aggregate to be used in the work, primarily because of the cost and time required. Such factors as thermal properties and volume change with wetting-drying rarely are determined. Other properties, such as chemical reactivity or effect of the aggregate on the freeze-thaw resistance of concrete are usually not determined. Consequently, it is worthwhile to apply a procedure by which the relative significance of such properties can be determined and the need for supplementary quantitative tests indicated.

Petrographic examination aids interpretation of other tests. For example, are the particles identified as clay lumps in accordance with ASTM Test Method for Clay Lumps and Friable Particles in Aggregates (C 142) indeed clay lumps or are they merely friable or pulverulent particles of some other composition? What is the cause of unexpected failure of concrete specimens in freezing and thawing? Is it the presence of unsound particles that do not disintegrate in the sulfate soundness test yet expand in freezing and thawing? Is it the result of an alkali-aggregate reaction? Is failure in the soundness or abrasion test the result of a complete breakdown of a small proportion of unsound or soft particles or partial disintegration of the greater proportion of the aggregate?

Detection of Contamination

Petrographic examination is the best method by which potentially deleterious and extraneous substances can be detected and determined quantitatively. Inadvertent contamination with natural substances, industrial products, or wastes, such as overburden or contaminants from trucks or railroad cars not properly cleaned of previous cargo, may markedly decrease the quality of aggregate. Contamination by containers may invalidate samples. Such substances as clay, soil, coal, vegetable matter, chemical fertilizers, petroleum products, or refractories containing free calcium or magnesium oxides are especially important. Incomplete processing of synthetic aggregates may contaminate the finished product with raw or partly fired materials or coal.

Undesirable substances inherent in the material, such as coatings, clay, plant remains, coal, and soluble salts, are detected easily and can be determined quantitatively by petrographic methods or by other procedures that can be selected most definitively pursuant to petrographic identification.

Determining Effects of Processing

Petrographic examination aids in the production and processing of aggregate. The relative merit of alternative processing methods and equipment can be determined quickly by comparison of the original material with the processed aggregate. Comparison can be based on particle shape, content of unsound or chemically reactive constituents, removal of coatings, amount of microfracturing, or production of rock dust.

The feasibility of beneficiation by removal of unsound or deleterious constituents depends on the properties of the particles and their abundance. Through petrographic examination, the undesirable particles can be identified and separated. Their properties can be evaluated and compared with properties of the remainder of the aggregate. If the undesirable particles are unusually soft, friable, dense, lightweight, or high in magnetic susceptibility, separation may be feasible on a commercial scale.

Petrographic examination can be used to control the manufacture of synthetic aggregate, such as expanded shale or clay, perlite, slag, and other types. Microscopical examination will reveal the presence of raw or underburned materials, alkali-reactive phases, and contaminants such as coal (Fig. 2). X-ray diffraction analysis is the most dependable means of

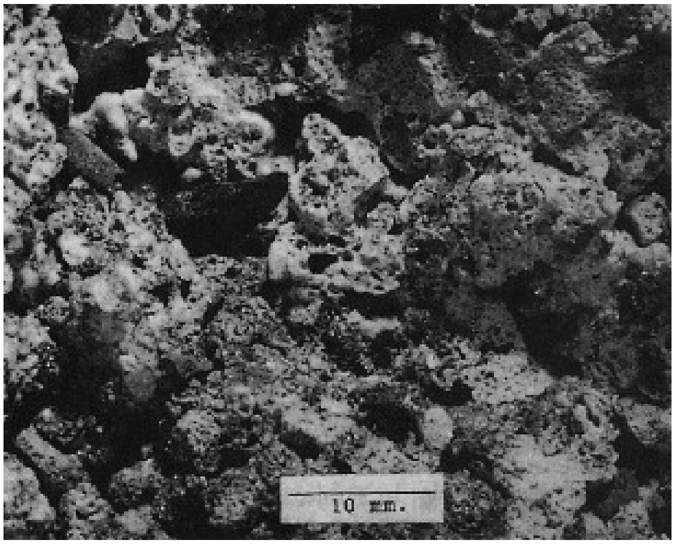

Fig. 2—Coal in sintered clay before crushing (courtesy of Bureau of Reclamation, U.S. Department of the Interior).

identifying and determining quantitatively the proportion of crystalline phases such as periclase (MgO), free lime (CaO), and clays whose crystal structure was not destroyed by the calcination. Differential thermal analysis can be used to estimate the effective temperature to which clays, shales, slate, and similar materials have been fired; the trace of the differential thermal curves for the raw and fired product will coincide above the effective temperature achieved in the firing operation.

Performance of the Petrographic Examination

Samples for Petrographic Examination

Samples of aggregate for petrographic examination should be representative of the source. Recommended procedures for sampling and for preparation of the sample for analysis are covered by ASTM Practices for Sampling Aggregates (D 75), ASTM C 295, and other technical literature [12].

Examination in the Field

Petrographic analysis of samples in the field is usually qualitative or only semiquantitative because lack of facilities makes detailed work difficult. However, detailed examination in the field may be warranted if aggregate retained on the 75 mm (3 in.) sieve is to be used in the work, inasmuch as a representative sample of this size fraction may weigh a 100 kg or more and transportation of the sample to the laboratory is costly. Chips from cobbles not adequately identified in the field should be taken to the laboratory for further study.

If the deposit or rock exposure is variable, samples should be selected from each zone and detailed notes made at the site should relate each sample to a particular zone and portion of the deposit or rock ledge. The relative proportion of unsound, fractured, or chemically deleterious materials should be estimated from measurements made at exposures. These notes and the results of the tests on the samples will be the basis for operation of the deposit inasmuch as it may be desirable to waste or avoid zones or portions containing inferior, unsuitable, or alkali-reactive materials.

Examination in the Laboratory

Petrographic examination of aggregate in the laboratory may be brief or detailed. Brief examination indicates the relative merit of materials from alternate sources and supplies justification for abandonment or continued investigation of undeveloped deposits or other exposures. The preliminary examination should not replace the quantitative analysis included in the program of acceptance tests.

Samples supplied to the laboratory comprise (*a*) granular materials such as gravel, sand, crushed stone, slag, or synthetic aggregate; (*b*) stone in quarried blocks and irregularly shaped pieces or drilled cores; and (*c*) fragments, sawed pieces, or drilled cores of concrete that contain the aggregate (coarse, fine, or both) that is of interest. For fully graded granular materials, the examination should be performed on at least three size fractions included in the aggregate. The sample of each size fraction should comprise at least 150 particles. For natural sand and gravel and crushed stone, the minimum representative samples are shown in Table 1.

TABLE 1—Minimum Representative Sample Size

Size Fraction, mm (in.)	Mass of 150 Particles
90–38 (3–1½)	13 kg (20 lb)
38–19 (1½–¾)	4.3 kg (9.5 lb)
19–9.5 (¾–⅜)	0.6 kg (1.3 lb)
9.5–4.7 (⅜–3⁄16)	0.17 kg (0.38 lb)
4.75–2.36 (No. 4–8)	7.5 g
2.36–1.18 (No. 8–16)	1.1 g
1.18–0.60 (No. 16–30)	0.14 g
0.60–0.30 (No. 30–50)	0.017 g
0.30–0.15 (No. 50–100)	0.0033 g

Samples of the aggregate for petrographic examination are obtained by sieving in accordance with ASTM Method for Sieve Analysis of Fine and Coarse Aggregates (C 136) and ASTM Test Method for Material Finer Than 75-μm (No. 200) Sieve in Mineral Aggregates by Washing (C 117). The fractions are quartered or, for fine aggregate, split repeatedly on an appropriate riffle.

Details of the procedure are outlined by Mather and Mather [3] and in ASTM C 295.

During the analysis, helpful information on identity and physical condition can be obtained by recording such features as (1) friability or pulverulence in the fingers; (2) resonance when struck; (3) ease of fracturing; (4) nature of the fracture surface and fracture fillings; (5) odor on fresh fracture; (6) color and its variation; (7) internal structure, such as porosity, granularity, seams, and veinlets; (8) reaction to water, such as absorption of droplets on fresh fracture, evolution of air on immersion, capillary suction against the tongue, slaking, softening, or swelling; (9) differential attack by acids or other chemicals; and (10) reaction with various stains.

Fractions retained on the 600-μm (No. 30) sieve are best identified, examined, and counted using a stereoscopic microscope. The analysis can be made conveniently by traversing a representative portion of each fraction by means of a mechanical stage and microscope assembly such as that employed in the point-count procedure according to ASTM Practice for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete (C 457) (Fig. 3). The mineralogical composition of finer fractions usually is determined most easily and accurately in immersion oils or

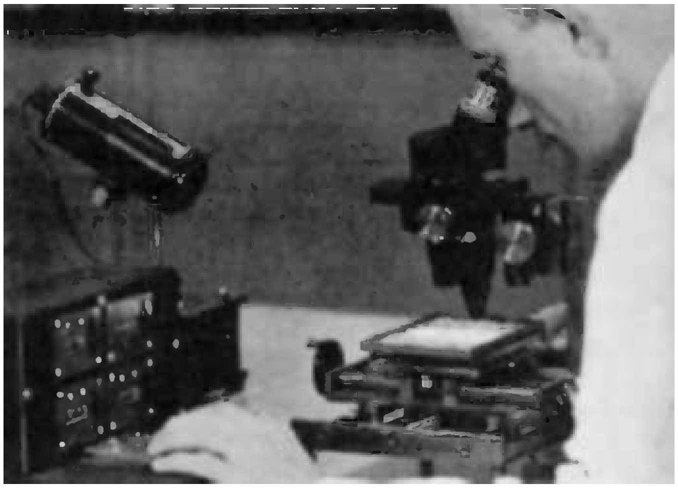

Fig. 3—Performing petrographic examination of fine aggregate with stereoscopic microscope, mechanical stage, and tally counter.

thin sections using a petrographic microscope. However, better continuity in description of physical characteristics of the particles is obtained if analysis of fractions passing the 600-μm (No. 30) sieve and retained on the 150-μm (No. 100) sieve is performed using a stereoscopic microscope.

Thin sections occasionally are necessary in examination of natural aggregate. They usually are employed in the study of quarried stone. However, preparation of plane sections by sawing and lapping, with or without etching or staining, commonly is preferable because of the large area made available for examination or analysis. Such examination should be supplemented by microscopical examination of grain mounts in immersion oils. Thin sections or polished surfaces, supplemented by use of oil immersion mounts of granular material, usually are used in the analysis of blast-furnace slag aggregates. X-ray diffraction and differential thermal analysis may be required to identify or determine quantitatively constituents that are very finely divided or dispersed through particles of the aggregate (Fig. 4).

Any of the previously noted types of coarse or fine aggregate can be examined while enclosed in hardened concrete, using broken fragments, sawed and lapped sections, and microscopical thin sections, as required and appropriate.

Ordinarily, petrographic analysis of concrete aggregates is performed on at least three coarse fractions and five fine fractions. The results may be used to compute the lithologic composition of the aggregate in any gradation comprising the analyzed size fraction. The bulk composition of a concrete sand may vary widely with fineness modulus. Occasionally, one analysis may be performed on a graded aggregate. For such analysis, the composition by count may differ greatly from the composition by mass.

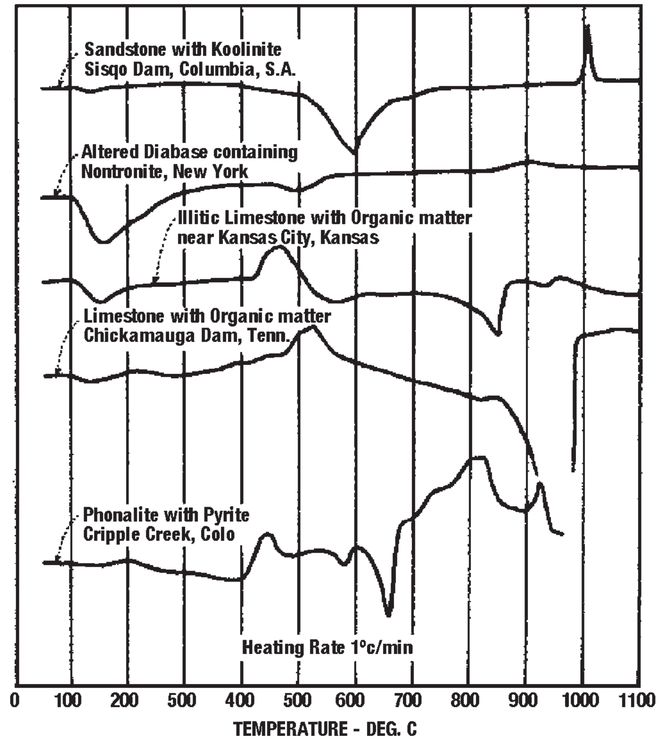

Fig. 4—Typical differential thermal analysis (DTA) records obtained on concrete aggregates. Kaolinite is indicated by the endotherm (downward shift) at 990–1025°C. Nontronite is revealed by endotherms at 100–350°C and 450–550°C. Illite produces the small endotherms at 100–200°C and 500–615°C. Organic matter produces large exotherms (upward shifts) at 430–500°C or 440–600°C. Pyrite (ferrous sulfide, FeS_2) develops a marked exotherm at 400–485°C (courtesy of the Bureau of Reclamation, U.S. Department of the Interior).

Numerical results of the analysis may be expressed by mass or count of particles retained on the 600-μm (No. 30) sieve, but for the finer fractions, the results are based only upon the count of grains unless a correction factor is applied. Consequently, consistent analyses of all fractions of fine aggregate and coarse aggregate can be reported only by count. Analysis by count is technically the more appropriate determination because the influence of particles of a given type upon performance of concrete in test or service depends primarily upon their frequency and distribution in the mass. When applied to coarse aggregate only, the petrographic analysis is more rapid if the relative proportion of the several rock types or facies is determined by mass. However, the accuracy and precision of analysis by mass are affected adversely by unavoidable loss of portions of the particles when they must be broken to allow identification of the composition and description of the physical condition. Specifications on lithologic composition are preferably expressed as percentage by count of particles.

Material passing the 75 μm (No. 200) sieve does not usually enter into the overall estimation of the quality of the aggregate for use in the production of portland concrete since the volume is limited. The examination of the fines will reveal some information on the breakdown and mineral constituents of the aggregate. Occasionally the amount of fines and its constituents can affect the workability of concrete. X-ray diffraction analysis is commonly used in the analysis of fine grain particles to determine the presence of mineral phases such as clays and zeolites. The presence of expansive clays can easily be detected by X-ray diffraction examination of oriented particle samples that have been treated with glycerol or ethylene glycol observing peak shift to about 17 Angstroms.

Details of calculating and reporting of results of petrographic examination and analysis of concrete aggregates are summarized in ASTM C 295. The results of the analysis of composition of aggregates enclosed in hardened concrete, such as by point-count analysis on sawed and lapped surfaces, are expressed as percent by volume of the concrete or percent by volume of total aggregate. Analysis of aggregate enclosed in concrete usually is confined to determination of the proportional volume of individual selected types of particles relative to the proportional volume of the total aggregate (see subsequent discussion).

Observations Included in the Petrographic Examination

In reporting the results of the petrographic examination, the petrographer should supply information on the following subjects as necessary for evaluation of the aggregate for service under the anticipated conditions of exposure in the concrete:

Mineralogical and lithologic composition
Particle shape
Surface texture
Internal fracturing
Coatings
Porosity, permeability, and absorption
Volume change, softening, and disintegration with wetting-drying

Thermal properties
Strength and elasticity
Density
Hardness
Chemical activity
  Solubility
  Oxidation
  Hydration
  Carbonation
  Alkali-silica reactivity
  Alkali-carbonate reactivity
  Sulfate attack on cementitious matrix
  Staining
  Cation-exchange reactions
  Reactions of organic substances
  Effects of contaminants

The significance of these properties is discussed by Rhoades and Mielenz [7,8], Dolar-Mantuani [11], Swenson and Chaly [9], Hansen [13], Mielenz [14], and others [15–19]. Sarkar and Aitcin [20] emphasize the need for petrographic examination of aggregates for very high-strength concrete, especially regarding internal texture and structure of the particles.

A simple test to detect forms of pyrite and similar ferrous sulfides that are likely to oxidize while enclosed in concrete was devised by Midgley [21] and is discussed more generally by Mielenz [14]. Shayan [22] reported popout formation and deterioration of the surface of a concrete floor by oxidation and hydration of pyrite included in exposed particles of coarse aggregate.

Soles [23] found that pyritiferous dolomite used as coarse aggregate produces distress when the concrete is heated to about 150°C (300°F) but less than 300°C (575°F). The deterioration is a result of expansion of the particles as oxidation of the pyrite and crystallization of iron and calcium sulfates take place. The present author has observed similar effects when compact pyritiferous shales constitute appreciable proportions in gravel and sand coarse and fine aggregate. These observations are pertinent to the evaluation of aggregates to be incorporated in concrete that will be subjected to sustained high temperatures in the indicated range, such as structural members and slabs to be included in or to support furnaces, kilns, and the like.

Stark and Bhatty [24] concluded that significant amounts of alkalies (sodium and potassium) can be released from certain alkali-bearing rocks and minerals when in contact with a saturated solution of calcium hydroxide and that such released alkalies engage in expansive alkali-silica reaction in concrete. The release appears to involve ion-exchange phenomena and possibly dissolution of silicates within the aggregate. Sources of alkalies include glassy volcanic rocks, plagioclase feldspars, potassium feldspar (microcline), and feldspathic sand. The release of potassium from phlogopite mica [25] and from particles of bentonite clay in natural sand [26] has been shown to promote an alkali-carbonate reaction in concrete. Similar effects can be produced by montmorillonite-type clays and zeolites from which sodium or potassium released by cation exchange may engage in alkali-silica reaction involving other siliceous constituents of the aggregate [27].

Schmitt [28] recently summarized information on micas as constituents of fine aggregate in concrete, citing work done in England. He concluded that for each 1 % of mica in fine aggregate by mass of total aggregate, the compressive strength of the concrete is reduced as much as 5 %. Frost resistance is reduced and drying shrinkage is increased by increasing content of biotite. He commented also on applicable specifications on allowable quantities of mica in coarse and fine aggregate. Occurrence of mica in concrete sands from numerous sources in the continental United States is reported by Gaynor and Meininger [29].

Higgs [30] reported a correlation between methylene blue adsorption and the amount of smectite (montmorillonite type) clay in natural sand that was the cause of undue slump loss and increased drying shrinkage of concrete, citing work by Davis et al. [31]. Elsewhere, he described the effect of chlorite in aggregate relative to its effect on the freeze-thaw durability of concrete [32]. The critical criteria required for poor durability were found to be chlorite content of volcanic rocks in excess of 20 % and distribution of the mineral in seams, clots, and fissures in contrast to a disseminated condition in the rock.

Petrographic criteria useful in detecting dolomitic rocks that are potentially susceptible to a deleterious degree of the alkali-carbonate reaction in concrete are described by Dolar-Mantuani [11], Hadley [33], Swenson and Gillott [34], and others [35–38].

Dolar-Mantuani [39] described the alkali-silica reactivity of argillites and graywackes. Bachiorrini and Montanaro [40] reported the occurrence of alkali-silica reaction in non-dolomitic carbonate rocks containing disseminated, finely divided silicate minerals. The alkali reactivity of strained quartz and quartzose rocks has been described and discussed by Buck [41] and others [6,11,42]. Investigators of concrete affected by alkali-silica reaction in many structures in Nova Scotia have concluded that alkali-silica reaction in which strained quartz is the primary alkali-reactive constituent has been augmented substantially by an alkali-aggregate reaction involving vermiculite that is present in phyllites, argillites, and graywackes, which are widespread in natural aggregates of that region [43].

It is intended that the preceding properties be determined qualitatively by observation of mineralogical composition and texture and internal structure of the particles by petrographic techniques and simple tests, such as those noted, as appropriate. When other tests are available, or if the particles can be compared petrographically with previously tested materials, the properties can be evaluated semiquantitatively or quantitatively. The petrographer should participate in the formulation of any supplementary studies that are contemplated.

Condition of the Particles

The following classification of properties is useful in cataloging the physical and chemical condition of particles constituting an aggregate. Physical condition is defined by three terms: (1) satisfactory, (2) fair, and (3) poor. Chemical stability in concrete is designated by two terms: (1) innocuous and (2) potentially deleterious.

Satisfactory

Particles are hard to firm and relatively free of fractures, capillary absorption is very small or negligible, and the surface texture is relatively rough.

Fair

Particles exhibit one or two of the following qualities: firm to friable, moderately fractured, capillary absorption small to moderate, surface relatively smooth and impermeable, very low compressibility, coefficient of thermal expansion approaching zero or being negative in one or more directions.

Poor

Particles exhibit one or more of the following qualities: friable to soft or pulverulent, slaking when wetted and dried, highly fractured, capillary absorption high, marked volume change with wetting and drying, a combination of three or more qualities listed under "fair."

Innocuous

Particles contain no constituents that will dissolve or react chemically to a significant extent with constituents of the atmosphere, water, or hydrating portland cement while enclosed in concrete or mortar under ordinary conditions of service in constructions. Particles are stable at high temperature or decompose without expansion.

Potentially Deleterious

Particles contain one or more constituents in significant proportion that are known either to react chemically under conditions ordinarily prevailing (or applicable in the present instance) in portland-cement concrete or mortar in such a manner as to produce significant expansion, interfere with the normal course of hydration of portland cement, or supply substances that might produce harmful effects upon mortar or concrete. By extrapolation, this category is extended to include individual constituents that produce notable expansion under conditions that are expected in the proposed work, such as in concrete to be exposed to high temperatures as a part of the planned service–for example, quartz and highly quartzose rock types that are subject to disruption as the quartz crystal expands about 2.4 % by volume while inversion takes place from the α- to the β-polymorph at 573°C (1063°F) [44].

Coatings should be reported and evaluated separately because coatings usually are confined to portions of a deposit and, for crushed stone, the nature and abundance of coatings vary with processing methods and equipment. Schmitt [28] recently summarized information concerning petrographic features of aggregate coatings and their significance.

Similarly, particle shape should be considered apart from other aspects of physical quality because particle shape is commonly subject to control or modification by processing of the aggregate. Unless otherwise defined by applicable specifications, particles whose length is five or more times their width should be designated as "elongated pieces," and those having a ratio of width to thickness greater than five should be designated as "flat pieces." The measurement should be made in accordance with definitions of these terms as they appear in ASTM Terminology Relating to Concrete and Concrete Aggregates (C 125) or as defined by the specifying agency. Particle shape relative to coarse aggregate is discussed by Sarker and Aitcin [20], who note in particular the unfavorable effect of elongated or flaky pieces on the workability of concrete. See Gaynor and Meininger [29] and Mass [45] for helpful discussions of the significance of the particle shape of concrete sands.

Petrographic Examination of Aggregates in Hardened Concrete

General

Aggregates enclosed in hardened concrete from structures or pavements in service or from test specimens can be subjected to petrographic examination for many reasons, such as initially to identify the nature of the aggregates in older structures under investigation or to determine the involvement of the aggregates in either satisfactory performance or deleterious activity. The following features can be investigated during such an examination:

1. Analysis of composition
   - Identification and qualitative or quantitative determination of proportions of constituents
   - Proportions of physically unsound or chemically reactive constituents
   - Contamination by foreign substances
   - Identification of sources of coarse and fine aggregate
   - Proportioning of blended aggregates
2. Proportions of coarse and fine aggregates
   - Sand-aggregate ratio
3. Segregation
   - Homogeneity of distribution of coarse and fine aggregates
   - Differential distribution of lightweight and heavyweight particles, coarse and fine aggregates
   - Concentration of unsound particles at exterior surfaces
4. Effects of attrition during handling of aggregates and mixing of concrete
   - Rounding of edges and corners
   - Coatings of dust of fracture
   - Aggregate fines within the cement-paste matrix
5. Coatings on aggregate particles
   - Composition and physical characteristics
   - Frequency and extent
   - Deleterious effects
6. Unsoundness of constituents during service or test exposure
   - Relationship of lithology and particle size to popouts
   - Involvement in scaling
   - *D*-cracking
   - Thermal expansion at high temperature in service exposure or fire
   - Decarbonation of carbonate aggregates in high-temperature service or fire
7. Chemical reactivity of constituents during service or test exposure
   - Alkali-silica reaction
   - Alkali-carbonate reaction
   - Dissolution of soluble constituents
   - Oxidation and hydration of ferrous sulfides in normal service
   - Oxidation and hydration of ferrous sulfides in high-temperature service
   - Hydration of free lime (CaO) or magnesia (MgO) in slag aggregates or contaminants
   - Staining of cement paste matrix by organic matter, sulfides, or other substances
   - Presence of gypsum or anhydrite

Proportions of Coarse and Fine Aggregates

The proportions of coarse and fine aggregates within hardened concrete can be determined fairly readily on sawed and lapped sections by means of the microscopical point-count method or the linear-traverse method in general accord with the requirements of ASTM C 457. The task is straightforward if the lithology of the coarse and fine aggregates is unambiguously distinctive such that no mutually common constituents are included in the respective materials. The particles of coarse or fine aggregates are accordingly identified by composition and relegated to the appropriate category, regardless of particle size. The area to be traversed should be at least three times that shown in Table 1 of ASTM C 457 for the respective nominal maximum size of aggregate; the method is of doubtful practicality for concrete containing aggregate whose nominal maximum size is larger than 37.5 mm (1½ in.) [46].

For concrete containing natural or crushed gravel coarse aggregate and manufactured sand or natural sand or a crushed stone coarse aggregate and a natural sand, wherein one or more constituents are found in both the coarse and fine aggregates, other considerations are required. For example, in southern Michigan, gravels and sands contain high proportions of dolomites and calcitic dolomites originating in geologic formations that are operated as sources of crushed stone coarse aggregate for use in structural concrete and pavements. Although the natural aggregates characteristically are rounded because of the only moderate hardness of the rock, the particles of crushed stone typically are rounded at edges and corners by attrition incidental to the processing and handling of the aggregate and the mixing of concrete. In this instance, the distinction between coarse and fine particles can be approximated by classifying as coarse aggregate all particles whose cross section on sawed and lapped surfaces includes one or more dimensions greater than 3⁄16 in., the opening of the U.S. Sieve No. 4 (4.75 μm), the remainder being classified as fine aggregate.

The measurement is made conveniently if the reticle of one eyepiece of the stereoscopic microscope includes a scale that, when calibrated, will demark the 4.75-μm (3⁄16-in.) dimension in the field of view. Of course, the dimensions of the cross section so revealed are not a measure of the size of the particle. Sections of coarse aggregate adjacent to edges and corners of the particle may be less than 4.75 μm (3⁄16 in.) in diameter and the maximum dimension of flat or elongated particles of fine aggregate may be greater than 4.75 μm (3⁄16 in.). However, these effects tend to be self-compensating. Completely angular and irregularly shaped particles of requisite lithology whose size is less than 4.75 μm (3⁄16 in.) would appropriately be relegated to the coarse-aggregate fraction if the aggregate is a combination of a crushed stone and natural sand.

These determinations permit estimation of the sand-aggregate ratio, an important factor in the proportioning of concrete for optimum workability.

Alkali Reactivity of Aggregate Constituents

Examination of hardened concrete from service or test exposure allows determination of alkali reactivity of siliceous or dolomitic rock types through observations made on sawed and lapped sections or fracture surfaces. The manifestations of reactivity include such features as rim formation, microcracking within the particles and adjacent cement-paste matrix, and the presence of secondary deposits. Darkened or clarified rims within the peripheral border of aggregate particles are the initial and most frequent indication of cement-aggregate reaction. The presence of such rims requires determination that the rims are, in fact, a consequence of processes occurring within the concrete rather than a result of prior weathering.

A decision on this matter is made readily if a bonafide sample of the unused aggregate is available for separate study. Peripheral rims occurring adjacent to fractured faces of crushed stone or crushed natural aggregate can be taken as being a product of cement-aggregate reaction. Also, rims produced by cement-aggregate reaction will be seen to thin or disappear where the aggregate particle is bordered by a void space [47]. A condition that is more frequent and more readily discovered than the relationship between peripheral rims and adjacent void space is the thinning or disappearance of the peripheral reaction rim along the bottom side of the aggregate particle as cast in the concrete, a circumstance related to bleeding and settlement of the fresh concrete, presumably producing a real or incipient separation at this location or a more dilute cement paste of higher water-cement ratio and lower alkalinity.

Summarizing the Petrographic Examination

Tables 2–6 exemplify a variety of forms in which the petrographic analysis may be reported. All are based upon samples received as a part of engineering investigations. The tabulations are always accompanied by appropriate discussions and supplementary descriptions. Table 2 is in the form that has been employed by the Bureau of Reclamation [48]. It departs somewhat from the format recommended in ASTM C 295, but the inclusion of pertinent descriptions facilitates interpretation of the analysis. The tabulation could be simplified by cataloging the rock types into major and secondary classifications; for example, "granite: fresh, moderately weathered and internally fractured, and deeply weathered." The summary of physical and chemical quality is included in a separate tabulation.

Table 3 is the analysis of the sand produced with the gravel whose composition is summarized in Table 2. The format conforms with ASTM C 295. Table 4 is similar except that the designations of quality are not used because they are inappropriate for the description of synthetic lightweight aggregates.

Table 5 is an analysis of a sample representing a commercial crushed stone coarse aggregate passing the 37.5-mm (1½-in.) sieve. The analysis was obtained because the aggregate apparently delayed or prevented development of specified strength by the concrete under certain conditions. In the tabulation, the denotations "innocuous" and "deleterious" are restricted to potentially deleterious alkali-silica reactivity because the significance of the sulfides and organic matter in the stone was not evaluated.

Table 6 is an example of petrographic analysis of aggregates in hardened concrete, namely, a crushed stone coarse aggregate and a natural sand containing particles of shale.

Petrographic Examination of Natural Aggregates

Examination of Natural Aggregates in the Field

Sand and gravel result from weathering, natural abrasion, and impacting of rock and the deposition of the resulting particles along streams, in lakes or marine basins, or by wind or glaciers on the earth's surface. Consequently, sand and gravel are more or less complex mixtures of different kinds of rocks and minerals. Moreover, deposits of sand and gravel usually vary vertically by stratification and laterally because of the lenticular nature of zones and strata, or because of facies changes. The concrete-making qualities of the aggregate are influenced by these changes. Examination in the field also should reveal the variability of the sand and gravel with reference to unsound or deleterious particles, interstitial clay, organic matter, or secondary mineralization.

Deposits of sand and gravel are commonly changed by deposition of mineral matter from ground water or by weathering of particles. Examination in the field should indicate the lateral and vertical extent and the physical nature and

TABLE 2—Example of Tabulation of a Petrographic Analysis of Gravel

________________ Plant, ______________ Company, Near Denver, Colorado
Sample No. ____________

Size Fraction, % by mass[a]					Physical Quality	Chemical Quality
Rock Types	1½–¾ in.	¾–⅜ in.	⅜–³⁄₁₆in.	Description of Rock Types		
Granite	29.5	40.0	48.6	medium- to fine-grained, rounded to fragmental	satisfactory	innocuous
Weathered granite	12.0	17.7	17.2	fractured, weathered, rounded to fragmental	fair	innocuous
Deeply weathered granite	...	0.5	...	fractured, slightly friable, rounded to fragmental	poor	innocuous
Coarse-grained granite	6.4	6.1	8.4	pink, rounded, includes some free quartz	satisfactory	innocuous
Fractured coarse-grained granite	...	0.9	...	pink, rounded, includes some free quartz	fair	innocuous
Rhyolite porphyry	0.8	0.2	1.1	microcrystalline, porphyritic, white to brown	satisfactory	innocuous
Andesite porphyry	2.2	1.2	0.1	microcrystalline, porphyritic, tan to green	satisfactory	innocuous
Weathered andesite porphyry	...	0.3	...	as above, fractured and weathered	fair	innocuous
Basalt	0.2	...	0.6	weathered, fractured, black, microcrystalline	fair	innocuous
Diorite	0.4	0.4	0.1	medium- to fine-grained, hard, massive	satisfactory	innocuous
Granite gneiss	32.2	14.3	15.8	hard, banded, fine- to medium-grained	satisfactory	innocuous
Weathered gneiss	10.3	7.8	2.7	as above, fractured to slightly friable	fair	innocuous
Deeply weathered gneiss	0.2	...	...	as above, intensely fractured to friable	poor	innocuous
Schist	2.2	2.3	2.2	hornblende schists, hard, rounded	satisfactory	innocuous
Fractured schist	...	0.4	0.2	as above, fractured	fair	innocuous
Quartzite	2.8	6.2	2.0	fine-grained, hard, massive to schistose	satisfactory	innocuous
Milky quartz	0.6	1.0	0.5	massive, hard, brittle, dense, smooth	fair	innocuous
Quartzose sandstone	...	0.1	...	fine-grained, massive, firm to hard	satisfactory	innocuous
Ferruginous sandstone	...	0.4	...	porous, brown, platy, quartzose	fair	innocuous
Shale	...	0.2	0.5	soft, absorptive, rounded, gray	poor	innocuous
Rhyolite	0.2	...	...	cryptocrystalline, porphyritic, pink to gray	satisfactory	deleterious

NOTE: Conversion factors—1 in. = 25.4 mm and 1 lb = 0.45 kg.

[a] Based upon analysis of 19.0 lb of 1½–¾-in., 2.7 lb of ¾–⅜-in., and 0.80 lb of ⅜–³⁄₁₆-in. aggregate.

TABLE 3—Example of Tabulation of a Petrographic Analysis of Natural Sand

________________ Plant,______________ Company Near Denver, Colorado
Sample No.________________

	Amount, as Number of Particles, %												
	In Size Fractions Indicated[a]							In Whole Sample[b]					
								Physical Quality			Chemical Quality		
Constituents	No. 4–8	No. 8–16	No. 16–30	No. 30–50	No. 50–100	No. 100–200	Passing No. 200	S[c]	F[c]	P[c]	I[c]	D[c]	T[c]
Granite and granite gneiss	34.9	33.6	21.0	4.8	...	...	...	13.5	1.0	0.6	15.1	...	15.1
Pegmatite	34.2	28.7	2.0	...	...	...	...	8.2	0.8	0.2	9.2	...	9.2
Rhyolite tuff	0.8	0.9	0.3	...	...	...	...	0.3	...	...	...	0.3	0.3
Basalt	...	1.0	...	...	...	...	...	0.2	...	...	0.2	...	0.2
Sericite schist	2.4	0.7	...	9.5	11.2	1.1	...	4.7	...	...	4.7	...	4.7
Quartz and quartzite	24.6	41.2	59.2	65.0	43.0	48.8	28.8	51.6	...	...	51.6	...	51.6
Feldspar	2.6	3.3	16.7	11.1	18.7	23.8	10.5	...	11.4	...	11.4	...	11.4
Claystone	0.5	...	...	...	...	1.2	...	...	...	0.2	0.2	...	0.2
Chalcedonic chert	...	0.6	...	3.3	...	...	...	1.2	...	...	...	1.2	1.2
Mica	...	...	0.8	6.3	10.2	8.3	51.1	...	3.8	...	3.8	...	3.8
Hornblende, garnet, zircon, etc.	...	...	...	...	16.9	16.8	9.6	2.3	...	...	2.3	...	2.3
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	82.0	17.0	1.0	98.5	1.5	100.0

[a] Based on count of 500 particles in each sieve fraction.

[b] Based on gradation of the sample received and on the distribution of constituents by size fractions shown at the left above.

[c] S = satisfactory; F = fair; P = poor; I = chemically innocuous; D = potentially chemically deleterious; T = total of constituent in whole sample.

composition of the coatings. Areas of the deposit that are free from coatings and zones that are so heavily coated as to preclude processing as aggregate should be delineated.

Weathering of gravel and sand after formation of the deposit is common on terraces and at lower levels along existing stream channels, glacial deposits, and outwash. The examination should define the extent and distribution of such weathering.

Close observation of gravel and sand exposed on the surface of the deposit commonly will reveal unsound particles that slake or fracture with freezing-thawing or wetting-drying (Fig. 5). Identification of such particles will aid evaluation of the petrographic examination performed in the laboratory. In interpreting the effects of natural freezing and thawing, the consequences of particle size should be evaluated [49]. Representative specimens of particles affected by freezing and thawing should be packaged separately and transmitted to the laboratory with samples of the gravel and sand. Water-soluble salts in coatings or ground water also may be revealed by efflorescence at or near the surface of the deposit. Their presence forewarns of the need for their identification and quantitative determination of their concentration in the aggregate.

Examination of Natural Aggregates in the Laboratory

Samples and data from the field should be examined to determine (1) the abundance of individual lithologic or mineralogical types; (2) the abundance of particles in various conditions of alteration and internal texture and structure and degrees of chemical reactivity; (3) the composition, frequency, abundance, and physical nature of coatings; (4) particle shape and surface texture; and (5) the possible qualitative contribution of particles of the several types to properties of concrete (Tables 1 and 2).

Natural aggregate may contain more than 20 rock and mineral types. Consequently, petrographic examination is commonly time consuming. Based upon similarity in composition and probable performance in concrete, two or more rock types commonly can be combined into a single category with considerable savings in time and without loss in the validity of the analysis. For example, granites, quartz diorites, granodiorites, and quartz monzonites—or rhyolites, dacites, and latites—of similar composition and physical condition might be combined, thus eliminating the need for tedious examination sufficient to effect a separation.

Soft, friable, and altered particles may be original constituents of sand and gravel, or may be developed by weathering in the deposit. Weathering in the deposit is especially significant because the alteration affects most or all particles, possibly causing softening and absorptivity in the superficial portion of the particles. This action decreases both the bond with the cementitious matrix and the strength, durability, and volume stability of the concrete.

Coatings on gravel and sand vary from minute spots and films to a cement that produces zones of sandstone or conglomerate in the deposit. Coatings usually are composed of fine sand, silt, clay, and calcium carbonate; but organic matter, iron oxides, opal, manganiferous substances, alkali- and alkali-earth sulfates, and soluble phosphates have been identified [8,28]. The petrographic examination should reveal the composition, abundance, physical properties, probable potential chemical reactivity, and the ease with which the coatings are removed by impact and abrasion.

TABLE 4—Example of Tabulation of a Petrographic Analysis of Expanded Clay Aggregate

________________ Plant, ____________ Company
Sample No. ________________

Amount, as Number of Particles, percent

In Size Fractions Indicated[a]

Constituent	¾–⅜ in.	⅜–3⁄16 in.	No. 4–8	No. 8–16	No. 16–30	No. 30–50	No. 50–100	Passing No. 100	In Whole Sample[b]	Remarks
Black to gray, vesicular particles	44.6	30.2	36.2	44.1	46.5	55.0	62.2	64.4	42.4	vesicular, hard
Black to gray, vesicular particles	13.1	1.7	...	...	...	...	...	...	1.2	vesicular, friable
Red to tan, vesicular particles	26.1	29.5	20.2	25.0	15.4	24.0	21.8	20.6	23.5	vesicular, hard
Red to tan, vesicular particles	7.7	1.8	...	...	...	...	...	...	0.8	vesicular, friable
Red to brown, brick-like particles	4.9	23.6	32.3	22.0	25.6	12.4	7.8	4.9	21.3	not vesicular, firm to fragile
Gray to pink, brick-like particles	2.8	9.8	7.7	8.8	9.2	3.0	1.6	1.1	7.1	not vesicular, fragile, many slake in water
Gray to pink, brick-like particles	0.6	1.0	0.4	...	...	...	...	...	0.4	not vesicular, friable, many slake in water
Gray to black, friable particles	...	1.7	2.0	...	0.8	0.3	...	...	1.0	not vesicular, contain coal
Granite	0.2	0.4	0.4	...	1.4	2.8	4.7	7.0	1.4	hard, dense
Sandstone	...	0.2	...	...	...	...	...	...	0.1	hard, fine-grained
Coal	...	0.1	0.8	0.1	1.1	2.5	1.9	2.0	0.8	hard to friable
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	

NOTE: 1 in. = 25.4 mm.

[a] Based on count of 500 particles in each size fraction.

[b] Based on gradation of the sample received, and on the distribution of constituents by size fractions shown at left above.

Gravel and sand potentially susceptible to the alkali-silica reaction occur along many important rivers in the United States and have been responsible for serious distress in many concrete structures of all types, including highway pavements (Fig. 6). The location of many known deposits of alkali-reactive natural aggregate in the western United States was reported by Holland and Cook [50]. The known alkali-reactive substances are the silica minerals, opal, chalcedony, tridymite, cristobalite, and strained or very finely divided quartz; glassy to cryptocrystalline rhyolites, dacites, latites, and andesites, and their tuffs; at least certain artificial siliceous glasses; and certain phyllites, slates, argillites, and graywackes (Fig. 7). Particles of volcanic glass or sometimes devitrified volcanic glass in aggregates may be reactive, depending on composition. Acid glasses (those with a silica content above 65 %) and intermediate glasses (with silica contents between 55 and 65 %) are commonly reactive; more basic glasses (silica content below 55 %) are less so. Reactive glasses may be identified by refractive indices below 1.57. Occasionally basalts contain glass whose index of refraction is less than 1.535 and palagonite may be partly altered to opal; such types are potentially alkali reactive. Fraser et al. [51] have provided a detailed study of the composition of chalcedonic cherts in relation to their reactivity with alkaline solutions. Alkali-reactive dolomitic rocks may occur as constituents of natural aggregates. A number of instances of alkali silica reactivity leading to serious distress have been observed with limestone aggregate that contains small amounts of dispersed silica, often skeletal remains of small organisms. The fact that limestone aggregates may not have the characteristic dolomitic composition and impurity content that results in alkali-carbonate reaction does not preclude the possibility of alkali-silica reaction if disseminated reactive silica exists in the material. Any aggregate containing a significant proportion of these substances is potentially deleteriously alkali reactive in concrete in service.

TABLE 5—Example of Tabulation of a Petrographic Analysis of Crushed Stone Coarse

Aggregate.[a]________________ Plant,____________________ Company
Sample No.________________

		Amount, % by weight[b]					
		Physical Quality			Chemical Quality		
Rock Type or Facies	**Description of the Rock Type or Facies**	**S[c]**	**F[c]**	**P[c]**	**I[c]**	**D[c]**	**T[c]**
Dense dolomitic limestone	gray to buff, contains sparse organic matter with pyrite and marcasite	56.2	...	...	56.2	...	56.2
Soft dolomitic limestone	gray to buff, soft to friable, slightly porous, sparse organic matter with pyrite and marcasite	...	33.9	...	33.9	...	33.9
Soft, organic dolomitic limestone	same as above except containing one or more seams of iron sulfides and organic matter	...	4.2	...	4.2	...	4.2
Limestone	white to gray, coarse- to medium-grained	2.4	...	...	2.4	...	2.4
Laminated limestone	laminated, fine-grained, iron sulfides and organic matter abundant	...	2.1	...	2.1	...	2.1
Chalcedonic limestone	white to gray, particles of zones of chalcedony evident	1.0	...	...	...	1.0	1.0
Chalcedonic chert	conchoidal fracture, dense	...	0.1	...	...	0.1	0.1
Sandstone	includes also grains of quartz and feldspar	0.1	...	...	0.1	...	0.1
Total		59.7	40.3	...	98.9	1.1	100.0

[a] Sample graded in accordance with specifications of the _____ State Highway Dept. for 11/2 in.–No. 4 aggregate for concrete highway pavement (1 in. = 25.4 mm).

[b] Based upon analysis of 25.9 lb of aggregate split from the sample (1 lb = 0.45 kg).

[c] S = satisfactory; F = fair; P = poor; I = not deleteriously alkali reactive; D = potentially deleteriously alkali reactive; T = total of constituent in the sample.

TABLE 6—Microscopical Analysis of Aggregate in Hardened Concrete[a]

Core No. 1: Street pavement in front of _________________________.Washington Street, ______________________, Michigan
Core No. 2: Sidewalk, SW corner of __________ Street and _________ Street, __________, Michigan

Item	Core No. 1	Core No. 2
Coarse aggregate, % by volume of total aggregate	54.99	54.70
Sand fine aggregate, % by volume of total aggregate	45.01	45.30
Shale particles, % by volume of sand fine aggregate	11.35	8.96

[a] Microscopical point-count method, ASTM C 457, based on the following criteria:

Core No.	Number of Counts	Area Traversed, in.^{2}[b]
1	1944	35.9
2	2838	24.9

[b] Maximum area available in submitted samples (1 in. = 25.4 mm).

Fig. 5—Cobbles of argillaceous limestone disrupted by freezing-thawing in the deposit, near Charlesvoix, Michigan. Observation of natural disintegration forewarns of possible difficulty in service as concrete aggregate (courtesy of Michigan Department of Transportation).

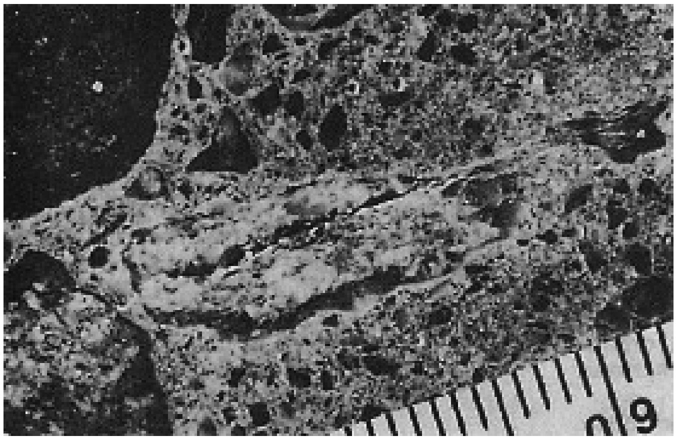

Fig. 7—Metasubgraywacke exhibiting advanced alkali-silica reaction in mass concrete, including darkened peripheral rim, internal microcracking, and radial cracking of enclosing mortar matrix. The internal microcracks are characteristically open in the interior of the particle and closed at the periphery. Note white deposits in external microcracks. A millimetre scale is shown.

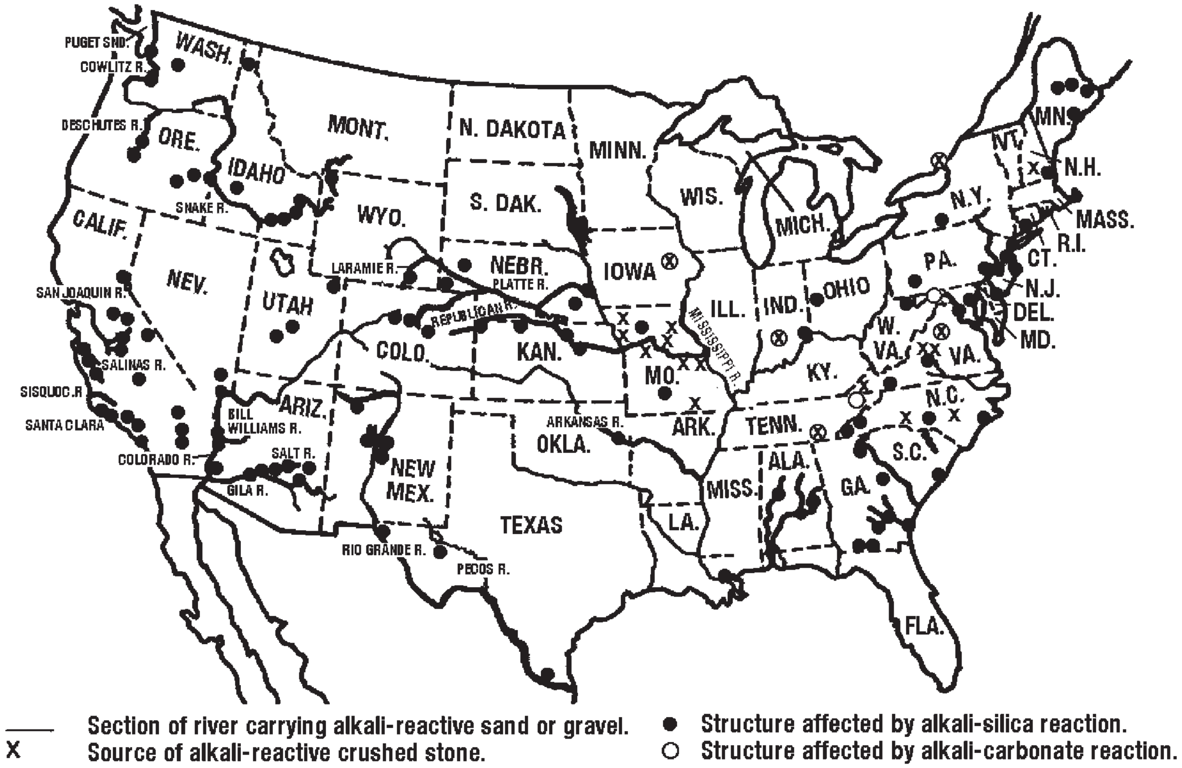

Fig. 6—Location of structures in the United States (other than Alaska and Hawaii) known to be affected by alkali-aggregate reaction in concrete, sources of sand or gravel subject to alkali-silica reaction, and sources of crushed stone subject to alkali-carbonate reaction.

Petrographic Examination of Crushed Stone

Examination of Stone in the Field

Rock formations are massive or stratified, the strata can occur in any attitude relative to the horizontal, and rock may vary widely in porosity, hardness, toughness, degree of fracturing, or chemical reactivity. For example, the alkali reactivity of dolomitic limestones typically varies widely even within the same quarry when deleterious facies are present [33,34].

Rock formations commonly contain zones of faulting, jointing, or local shearing, within which the materials are fractured or chemically decomposed. Certain zones may contain deleterious or unsound substances, such as chalcedonic or opaline chert and clay or shale in limestone or dolomite, or zones of hydrothermal alteration in igneous or metamorphic formations in which sulfides, zeolites, clays, or clay-like minerals are prominent. Especially in warm and humid areas, rock formations commonly are fractured, leached, and partially decomposed by weathering near the surface to varying depths, depending on localized differences in fracture, porosity, or lithology.

These features should be discovered by geologic and petrographic examination of natural exposures, quarried faces or other excavations, and drilled core. Petrographic examination in the field should include exposures of geologic formations and stone used in the area for fill, ballast, riprap, and masonry. Excavation of trenches or test pits may be required. This survey may reveal lithologic varieties that have failed during natural exposure to freezing-thawing or wetting-drying or to oxidation and hydration (Fig. 8). The significance of the size of fragments should be recognized in any evaluation of such observations [49]. Disintegration of rock on natural exposures commonly does not coincide with results of the sulfate soundness test ASTM Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88) [52].

Shale, claystone, and argillaceous rocks, including deeply altered basalts and diabases (traprock) containing nontronite, may slake and fracture during brief exposure even though they appear sound when excavated. Similar effects occur if the zeolites leonardite-laumontite constitute a significant proportion of the rock or occur as seams or veinlets in the formation; they are especially common in granodiorites, quartz diorites, quartz gabbros, and anorthosites. Soluble salts usually will be revealed by efflorescence at exposed surfaces.

Note that the term “crushed stone” covers aggregates produced by the processing of boulders, cobbles, and the like as they may be derived from deposits of gravel and sand, where substantially all of the faces of the particles have resulted from the crushing operation (ASTM C 125). Field examination of such sources is analogous to that applied to natural aggregates as discussed previously.

Examination of Crushed Stone in the Laboratory

As indicated earlier, crushed stone aggregates are commonly complex petrographically and not only when they are derived from the crushing of boulders and cobbles recovered from gravel. Consequently, they should be examined in the same detail as required for natural aggregates (Table 5).

Petrographic examination of stone in the form of quarried blocks or irregularly shaped pieces should include inspection of the entire sample. The examination should establish the relative abundance of individual rock types or facies (Fig. 9). Specimens representative of each type should be obtained by the sawing or coring of typical pieces; these specimens provide for special tests, detailed petrographic examination, and reference. Procedures for obtaining representative samples are described by Abdun-Nur [12].

Quantitative petrographic analysis can be performed on calcareous rocks by preparing plane surfaces by sawing and lapping, followed by etching. Thirty seconds of immersion in 10 % hydrochloric acid (HCl) will provide adequate differential attack on calcite and dolomite and will expose non-calcareous constituents, such as clay, chert, pyrite or marcasite, and siliceous sand or silt. The etched surface can be analyzed by point-count or linear traverse in general accordance with procedures given in ASTM C 457. This technique can be applied to calcareous aggregates in hardened concrete (Fig. 10).

Quantitative analysis can be performed similarly on prepared surfaces of quartzose and feldspathic rocks following etching by 1½ to 2 min immersion in hydrofluoric acid (HF) (Fig. 11).

Fig. 8—Disintegration of argillaceous facies of limestone used as riprap at Chickamauga Dam, Tennessee. Observation of such disintegration indicates unsoundness of stone in at least portions of a quarry (courtesy of the Bureau of Reclamation, U.S. Department of the Interior).

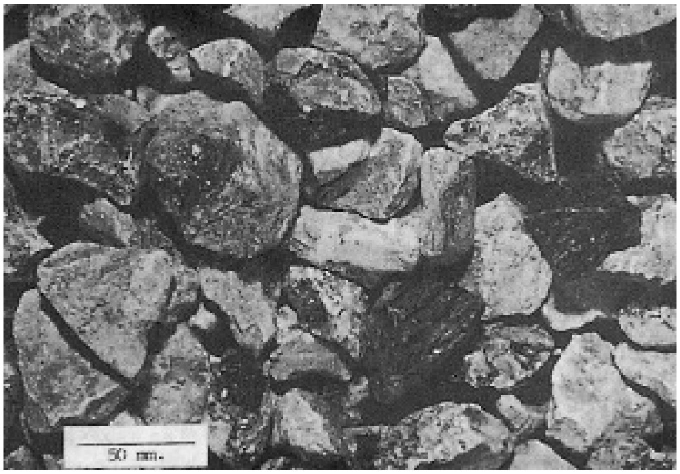

Fig. 9—Crushed limestone aggregate containing seams and pieces of illite shale, Webster Dam, Kansas. The physical quality was indicated as follows: satisfactory, 61 %; fair, 34 %; poor, 5 % by mass (courtesy of the Bureau of Reclamation, U.S. Department of the Interior).

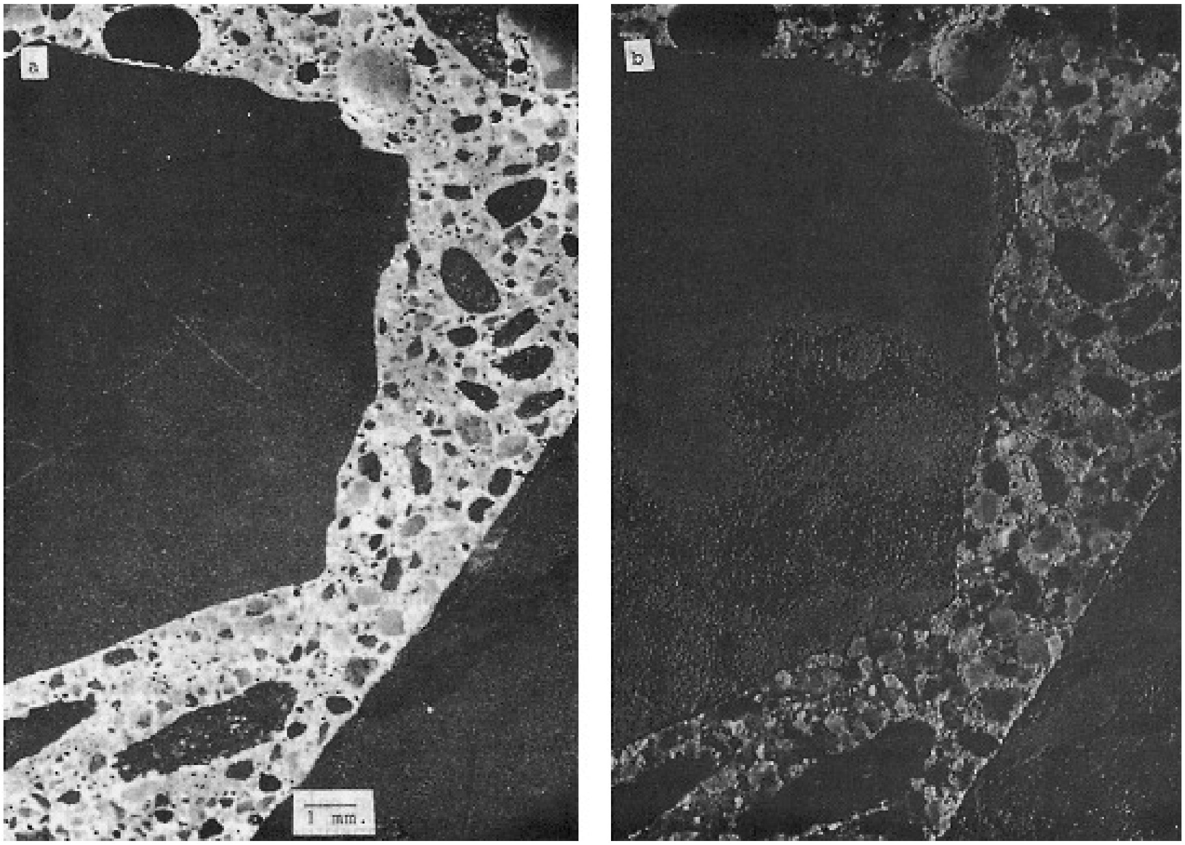

Fig. 10—Concrete containing dolomitic limestone coarse aggregate (*a*) before, and (*b*) after etching for 30 s in 10 % HCl. Scattered crystals in the etched areas on the lapped surface of the aggregate particles are dolomite.

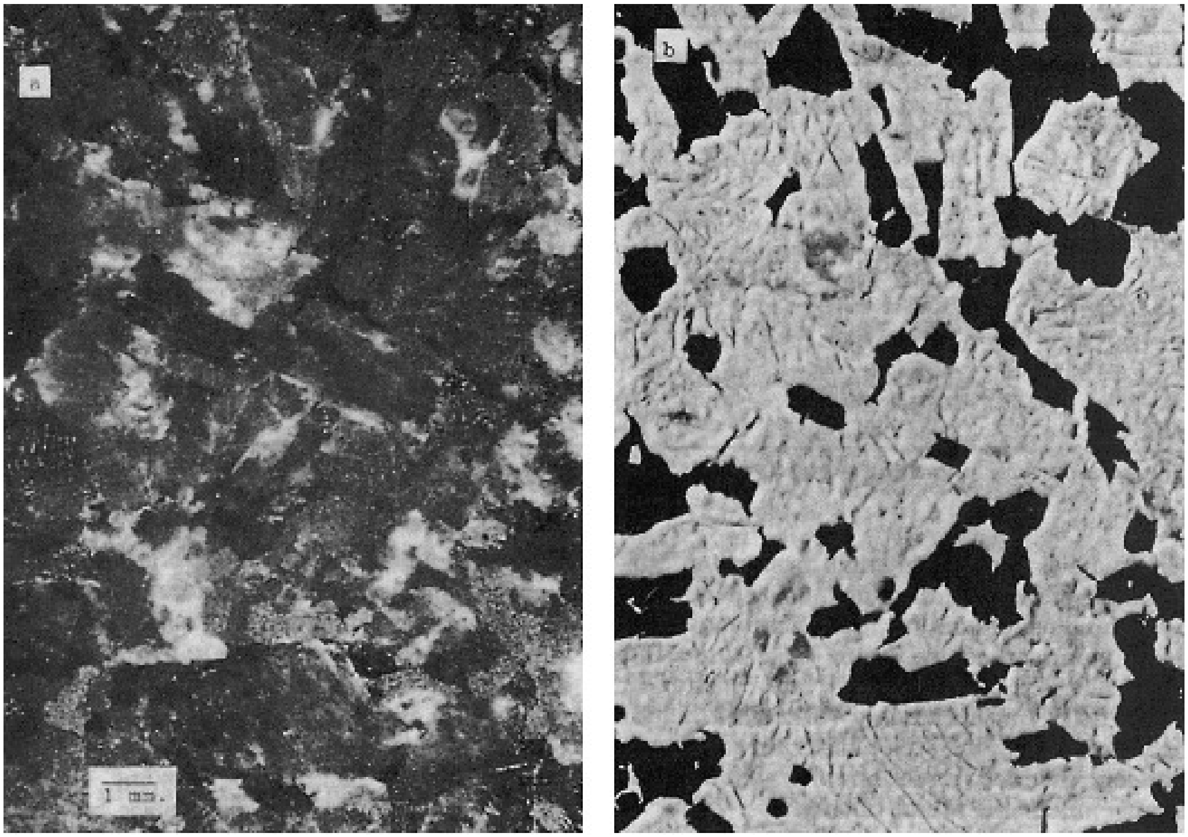

Fig. 11—Augite gabbro (*a*) before, and (*b*) after 11/2 min etching in concentrated HF. White areas are plagioclase feldspar, dark areas are augite in the etched surface.

If the sample of stone was submitted for crushing tests in the laboratory, the manufactured aggregate produced should be examined to determine the effects of natural fracturing, jointing, and internal texture on particle size and shape; frequency of fractures and seams within the particles; distribution of unsound or deleterious substances in the size fractions; and the abundance and composition of crusher dust. These features should be correlated with the processing equipment and methods employed.

Inspection of the stone prior to processing is important because only thus can the examination of the finished aggregate be interpreted fully. For example, if unsound or deleterious particles constitute 10 % of the finished aggregate, was this proportion derived from approximately one piece in ten of the original sample or does a typical piece of the stone contain approximately 10 % of unsound material? The former possibility suggests that the quarry should be examined to determine whether the unsound zones can be avoided or wasted; the latter suggests that the material should not be used as aggregate in concrete for permanent construction.

If the samples are in the form of drilled core, the entire length of the core should be examined and compared with logs available from the driller and geologist. Special attention must be given to sections in which core loss was high or complete, inasmuch as such zones commonly represent fractured, altered, or otherwise unsound rock. The core should be examined by means of the hand lens, stereoscopic microscope, and petrographic microscope, as necessary, to establish variations in lithology; frequency and intensity of fracturing; content of clay or other soft materials, regardless of rock type; and presence of chemically reactive substances. The examination is facilitated by sawing and lapping of the core along the length, with or without etching or staining. These observations should be correlated from hole to hole so that the variation in lithology or quality of the rock, both in depth and laterally, is established.

The quality of the rock to be expected from the formation represented by the cores also will be revealed by petrographic examination of aggregate produced from cores in the laboratory.

Petrographic Examination of Blast-Furnace Slag

Blast-Furnace Slag

Blast-furnace slag is the nonmetallic product, consisting essentially of silicates and aluminosilicates of calcium and other cations, that is developed simultaneously with iron in a blast furnace (see ASTM C 125). Properties of blast furnace slag as concrete aggregate have been reported by Gutt et al. [53]. Three general types of blast-furnace slag are used for concrete aggregate; namely, air-cooled slag, granulated slag, and lightweight slag [54]. Petrographic examination of lightweight or expanded slag will be discussed in a later section.

Performance of the Petrographic Examination of Blast-Furnace Slag

The procedure for petrographic examination of blast-furnace slag is not included specifically in ASTM C 295. However, the instructions provided for examination of ledge rock, crushed stone, and manufactured sand are applicable. In addition to the indicated microscopical methods, examination of polished and etched surfaces in reflected light is a valuable technique, preferred by some petrographers because the surfaces are easy to prepare in sizes larger than standard thin sections, the two-dimensional aspect simplifies quantitative estimation of composition, and microchemical tests can be used to identify various phases in the surface under study.

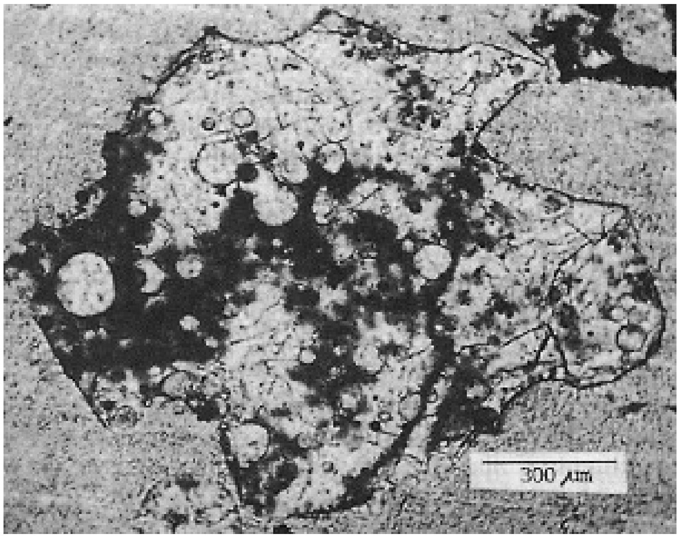

Fig. 12—Photomicrograph of granulated blast-furnace slag. Note the vesicles (bubbles) in glass phase (white). The dark areas are concentrations of microcrystalline melilite and merwinite (courtesy of the Bureau of Reclamation, U.S. Department of the Interior).

Air-cooled slag is more or less well-crystallized, depending primarily upon the method of disposal employed at the steel plant. Such slag crushes to angular and approximately equidimensional pieces, the surface textures of which are pitted, rough, or conchoidal. Crystals range from submicroscopic to several millimetres in size. Abrasion resistance relates to glass content and the condition of internal stress [54].

Well-granulated slag is substantially all glass. Crystals occur individually or in clusters scattered through the glass matrix. Incipient crystallization produces brown or opaque areas in thin sections (Fig. 12).

The petrographic examination of blast-furnace slag aggregate should include descriptions of the various types of slag as well as of contaminating substances. The slag constituent usually can be segregated into two or more varieties, depending upon particle shape, surface texture, color, vesicularity, crystallinity, or the presence of products of weathering. Evaluation of effects of weathering is especially important when old pits or deposits or fill are being exploited. Each type should be studied in sufficient detail to assure identification of potentially deleterious compounds.

More than 20 compounds have been identified in blast-furnace slag (Table 7), but even well-crystallized slag rarely contains more than five compounds (Table 8). The typical constituent of blast-furnace slag is melilite, a compound of variable composition between akermanite ($2CaO{\cdot}MgO{\cdot}2SiO_2$) and gehlenite ($2CaO{\cdot}Al_2O_3{\cdot}SiO_2$). Pseudowollastonite and anorthite are also common occurrences. High-lime blast-furnace slags usually contain one or more forms of dicalcium silicate (α, β, or γ forms of $2CaO{\cdot}SiO_2$). Magnesium-containing blast-furnace slags commonly include monticellite, forsterite, or merwinite. Calcium sulfide (CaS) is almost always present in small proportion. Sulfides of manganous manganese and ferrous iron

TABLE 7—Compounds Occurring in Blast-Furnace Slag[a]

Compound	Chemical Formula	Compound	Chemical Formula
Gehlenite	$2CaO \cdot Al_2O_3 \cdot SiO_2$	oldhamite	CaS
Akermanite	$2CaO \cdot MgO \cdot 2SiO_2$	ferrous sulfide	FeS
Pseudowollastonite	$\alpha CaO \cdot SiO_2$	manganous sulfide	MnS
Wollastonite	$\beta CaO \cdot SiO_2$	spinel	$(Mg, Fe)O \cdot Al_2O_3$
Bredigite	$\alpha' 2CaO \cdot SiO_2$	anorthite	$CaO \cdot Al_2O_3 \cdot 2SiO_2$
Larnite	$\beta 2CaO \cdot SiO_2$	periclase	MgO
γ-dicalcium silicate	$\gamma 2CaO \cdot SiO_2$	lime	CaO
Olivine	$2(Mg, Fe)O \cdot SiO_2$		
Merwinite	$3CaO \cdot MgO \cdot 2SiO_2$	cristobalite	SiO_2
Rankinite	$3CaO \cdot 2SiO_2$	calcium aluminate	$CaO \cdot Al_2O_3$
Monticellite	$CaO \cdot MgO \cdot SiO_2$	cordierite	$2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$
Pyroxene			
Diopside	$CaO \cdot (Mg, Fe)O \cdot 2SiO_2$	sillimanite	$Al_2O_3 \cdot SiO_2$
Enstatite	$MgO \cdot SiO_2$	mullite	$3Al_2O_3 \cdot 2SiO_2$
Clinoenstatite	$MgO \cdot SiO_2$	madisonite	$2CaO \cdot 2MgO \cdot Al_2O_3 \cdot 3SiO_2$

[a] Compiled from several sources, primarily Nurse and Midgley [56], McCaffery et al. [62], and American Concrete Institute Committee 201 [60].

are common. Properties and techniques for identification of these compounds are summarized by Rigby [55], Nurse and Midgley [56], Insley and Frechette [57], and Snow [58], and in standard works on mineralogy. X-ray diffraction methods are necessary if crystalline phases are submicroscopic and are a great aid to a petrographer who is developing experience independently in this field.

Several constituents of blast-furnace slag may be deleterious to the performance of concrete. Sulfides released into the cement-paste matrix produce innocuous green staining of the interior of the concrete. Occasionally, a mottled aspect is produced on the surface of damp concrete [14], but the coloration fades to a homogeneous shade of gray on drying and exposure to the atmosphere. Presence of colloidal sulfides is suggested by yellow or brown coloration of the glass phase [53]. Gypsum (calcium sulfate dihydrate, $CaSO_4 \cdot 2H_2O$) commonly forms in blast-furnace slag by weathering and may result in sulfate attack on the cement-paste matrix of concrete in service. Granulated slag is most susceptible to the formation of gypsum during weathering exposure.

Inversion of β-dicalcium silicate to the γ-dicalcium silicate, with accompanying 10 % increase in volume of the crystal, causes "dusting" or "blowing" of slag [54,59,60]. The inversion ordinarily takes place before the slag has cooled, and the disintegrated material is removed by screening during the production of coarse aggregate. In less severe occurrences, the disintegration takes place slowly, producing pieces that are partly or wholly weak and friable, and thus unsuitable for concrete aggregate. This action of dicalcium silicate can be avoided by maintaining a ratio of CaO to SiO_2 in the

TABLE 8—Most Frequently Occurring Combinations of Compounds of $CaO \cdot MgO \cdot Al_2O_3 \cdot SiO_2$ Produced by Crystallization of Blast-Furnace Slag[a]

	Combination of Compounds[b]												
Flux Stone	C_2AS	C_2MS_2	C_2S	CS	C_3S_2	C_3MS_2	MA	CMS_2	CMS	CAS_2	M_2S	MS	MgO
	X	X	X		X								
	X	X	X			X							
Limestone	X	X				X	X						
	X	X					X			X			
	X	X		X						X			
		X		X				X		X			
		X						X		X	X		
		X					X		X	X	X		
Dolomite		X					X		X		X		
		X			X	X	X		X				
					X								X
								X		X	X	X	

[a] After Nurse and Midgley [56].

[b] Key: C_2AS = gehlenite } melilite
C_2MS_2 = akermanite
C_2S = dicalcium silicate
CS = wollastonite or pseudowollastonite
C_3S_2 = rankinite
C_3MS_2 = merwinite
MA = spinel
CMS_2 = diopside
CMS = monticellite
CAS_2 = anorthite
M_2S = forsterite
MS = enstatite
MgO = periclase

slag sufficiently low to prevent formation of the compound, or by chilling the molten slag so that the compound does not crystallize [53]. If air-cooled slag is poured in thin layers, rapid cooling ordinarily arrests the compound in the β-modification. Dicalcium silicate can be identified microscopically in slag by special techniques [59]. Scattered crystals of β-dicalcium silicate commonly are stable in blast-furnace slag and therefore are innocuous.

Free lime (CaO) and magnesia (MgO) are extremely rare as constituents of air-cooled blast-furnace slag and are not likely to form either as a primary phase or devitrification product in granulated blast-furnace slag [56,61].[2] Nevertheless, their absence or presence and abundance should be established by petrographic examination. These compounds are deleterious because of the increase in solid volume resulting from hydration and carbonation in place.

Cristobalite (SiO_2) has been reported as a constituent of blast-furnace slag [62]. This compound is potentially subject to the alkali-silica reaction in portland-cement concrete; if identified, its abundance should be determined.

The glass phase of blast-furnace slag is not deleteriously reactive with cement alkalies.

Contaminating substances whose presence or absence should be determined by petrographic methods are metallic iron, iron carbide, coke, and incompletely fused fluxstone. The last is important because hydration and carbonation of free lime and magnesia may produce expansion of the concrete and popouts. Metallic iron and iron carbides rust by oxidation and hydration if exposed at the surface of the concrete.

Petrographic Examination of Lightweight Concrete Aggregates

Expanded Clay, Shale, and Slate

Petrographic examination of lightweight aggregate should include segregation of the particles into as many categories as required to describe the sample adequately (Table 4). Particles of expanded material may be segregated on the basis of particle shape, surface texture, development of a coating or "skin," and vesicularity. Incompletely expanded particles and particles not expanded should be distinguished from vesicular ones. Such particles usually represent sandstone or siltstone that was intercalated within the clay, shale, or slate. They should be separated on the basis of porosity, absorptivity, density, friability, softness, occurrence of efflorescence, and reaction to water (softening, slaking, or swelling). The petrographic examination supplements standard tests in distinguishing clayey particles from "clay lumps" determined in accordance with ASTM C 142. Other materials to be identified and determined quantitatively are underburned or raw material, coal, and miscellaneous rock particles. The composition and content of raw materials commonly can be established most easily and accurately by X-ray diffraction or differential thermal analysis.

The individual types of particles also should be analyzed petrographically to establish the presence of free lime or magnesia. These compounds result mainly from decomposition of calcite and dolomite in the feed during firing. They may produce distress or popouts in concrete or concrete products unless the aggregate is water- or steam-cured prior to use [63]. Hard-burned magnesia may not be neutralized by such treatment.

Petrographic examination also assists in the selection of raw materials, development of manufacturing equipment and methods, and process control.

Industrial Cinders

Industrial cinders used as concrete aggregate are the residue from high-temperature combustion of coal and coke in industrial furnaces. Petrographic examination should determine the physical nature of the cinder particles on the basis of composition, friability, softness, particle shape, and surface texture. Particular attention should be given to identification of sulfides, sulfates, coal, and coke.

Expanded Blast-Furnace Slag

Expanded blast-furnace slag is produced by carefully controlled intermingling of molten slag and water or steam in one of several ways [54]. The petrographic examination should describe the aggregate in terms of the nature of the expanded particles, including their particle shape, surface texture, friability or softness, effects of weathering in stockpiles, and content of contaminating substances, such as dense slag. The petrographic examination is especially important when fill or waste deposits are later contemplated for use as a source of concrete aggregate.

Pumice, Scoria, Tuff, and Volcanic Cinders

Pumice, scoria, tuff, and volcanic cinders used for lightweight aggregate are naturally occurring porous or vesicular lava and ash. Pumice is a very highly porous and vesicular rock composed largely of natural glass drawn into approximately parallel or loosely intertwined fibers and tubes. Scoria is a highly porous and vesicular rock in which the vesicles typically are rounded or elliptical in cross section, the interstitial glass occurring as thin films (Fig. 13). Tuff is a general term designat-

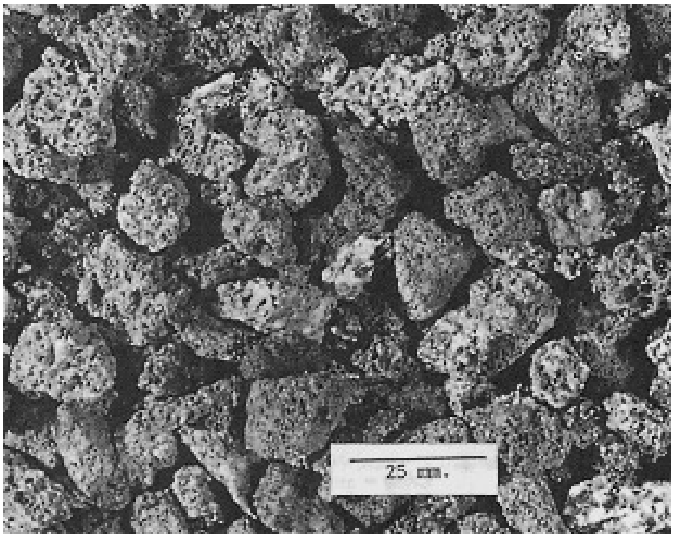

Fig. 13—Typical basaltic scoria aggregate. The pieces are black, gray, and reddish brown. Note the rounded vesicles (courtesy of the Bureau of Reclamation, U.S. Department of the Interior).

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[2] Unlike blast-furnace slags, basic open-hearth slags commonly contain free oxides that are subject to hydration and carbonation in portland-cement concrete, namely, free lime (CaO) and magnesiowustite, a solid solution of magnesium oxide (MgO), manganese oxide (MnO), and ferrous oxide (FeO).

ing consolidated volcanic ash of any lithologic type or physical character. Volcanic cinder is a loose accumulation of highly vesicular (scoriaceous) fragments of lava, predominantly ranging from 4 to 32 mm (0.16 to 1.26 in.) in diameter.

Petrographic examination of these types of aggregate includes segregation of the particles on the basis of particle shape, surface texture, porosity or vesicularity, fracturing, friability or softness, weathering, specific gravity, secondary deposits in voids, coatings, and potential alkali reactivity. Extraneous or contaminating substances are primarily dense particles of volcanic rock and organic matter. In production of lightweight aggregate, two types of volcanic materials occasionally are intermixed for economy or to control gradation or unit weight. The type and relative proportion of the materials can be established by petrographic examination.

Volcanic glass with an index of refraction less than 1.535 is potentially deleteriously reactive with cement alkalies; glass whose index of refraction is in the range of 1.535–1.570 probably is alkali reactive. Opal, chalcedony, tridymite, and cristobalite are also common alkali-reactive constituents of volcanic aggregates. These minerals are especially common as alteration products of volcanic rocks of acidic to intermediate composition. However, opal may occur as an alteration product of palagonite in basaltic lavas.

In spite of alkali-silica reaction and formation of alkalic silica gel in highly porous vesicular lavas and tuffs, expansion and cracking of the enclosing concrete usually is prevented by the abundant voids into which the hydrating gel can escape without development of excessive stress in the mortar. For example, a highly porous rhyolite tuff from Hideaway Park, Colorado, containing abundant tridymite caused only 0.041 % expansion of a high-alkali cement mortar during one year of moist storage in accordance with ASTM Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method) (C 227), yet the specimen contained abundant alkalic silica gel [64]. A similar but dense rhyolite tuff from near Castle Rock, Colorado, produced an expansion of 0.400 % under the same conditions.

Perlite

When heated rapidly to fusion, certain obsidians and pitchstones release gases that, being trapped within the molten glass, vesiculate the rock and cause disruption into small pieces. The product is known commercially as perlite.

Petrographic examination should indicate the composition of the aggregate in terms of particle shape, surface texture, rock classification, density, friability or fragility, and potential alkali reactivity. Perlite may contain particles of dense volcanic rock or individual crystals.

Being composed of rhyolitic volcanic glass, typical perlite is potentially reactive with cement alkalies, although significant expansion may not occur because of the porosity of the particles. However, laboratory tests demonstrate that certain perlites produce significant expansion of mortar stored in accordance with ASTM C 227 in combination with either high-alkali or low-alkali cement [65,66]. Such volume change will not necessarily cause structural distress if appropriately accommodated in the design of the constructions or concrete product, such as masonry units or precast panels.

Exfoliated Vermiculite

Exfoliated vermiculite is produced by rapid heating of the micaceous mineral, vermiculite. Release of combined water expands the crystals—like an accordion—increasing the volume to as much as 30 times its original size. The degree of expansion varies widely, depending upon mineralogical properties and purity and the conditions of firing.

During petrographic examination, the particles of vermiculite are segregated by degree of expansion, elasticity or brittleness of the flakes, and fragility of the expanded crystals. These differ significantly within individual samples from some sources, especially from marginal deposits where the vermiculite grades into hydrobiotite or biotite. Also to be reported is the intermixture of the vermiculite with particles of rocks and minerals occurring with the vermiculite in the ore deposit.

Diatomite

Crushed and sized natural diatomite typically is soft, porous, absorptive, and ranges from firm to pulverulent. Finely divided opal and opaline skeletons of diatoms are the predominant constituents. Fine sand, silt, clay, and volcanic ash are present in widely differing proportions. At least certain diatomites produce significant expansion of mortars stored in accordance with ASTM C 227, with both high- and low-alkali cement [65,66].

Petrographic Examination of Recycled Concrete for Use as Concrete Aggregate

Recycled Concrete

Recycled concrete is defined in the report of the American Concrete Institute (ACI) Committee 117 on Cement and Concrete Terminology [67] as “hardened concrete that has been processed for reuse, usually as aggregate,” and in ASTM C 33 as “crushed hydraulic-cement concrete.” Such reclaimed concrete from structures and pavements was used widely in Great Britain and Germany following World War II and, more recently, it has been investigated in the United States, Great Britain, Canada, and elsewhere as a partial or complete replacement for conventional aggregates for purposes of economy, substitution for natural or crushed stone aggregates in short supply, and environmental benefits.

Buck [68] concluded that use of recycled concrete as aggregate in new concrete is feasible and may become routine. Forster [69] reported on extensive studies of such applications by state departments of transportation. Committee 37-DRC, Reunion Internationale des Laboratoires D’Essais et de Recherches sur les Materiaux et les Constructions (RILEM), reported on the state of the art in this technology for the period of 1945–1985 [70].

The following summary of the properties of concrete containing recycled concrete as aggregate may assist the petrographer in decisions on (1) features to be considered in examination of constructions to be demolished with the intent that the concrete will be reclaimed for production of concrete aggregate and (2) observations that should be included in petrographic examination of samples of such materials.

Compressive Strength

Hansen and Narud [71] concluded that the compressive strength of concrete containing recycled concrete is controlled largely by the water-cement ratio of the original concrete when other conditions are essentially identical. When the water-cement ratio of the original concrete is equal to or lower than that of the new concrete containing the recycled aggregate as coarse aggregate only, they observed that the strength of the new concrete is equal to or higher than that of the original concrete, and vice versa, provided the fine aggregate is a natural sand or manufactured sand of suitable quality.

In evaluating the probable influence of recycled concrete from a given source on the strength of new concrete, the compressive strength of the concrete to be recycled is a more practical measure of quality than is the water-cement ratio because the water-cement ratio usually is not known and is not readily determined in practice.

A substantial reduction in compressive strength may result when the conventional fine aggregate is replaced in whole or in part by fine aggregate derived from the recycled concrete. The RILEM report [70] concludes that all material below 2 mm in recycled concrete should be screened out and wasted.

Water Requirement

Use of recycled concrete decreases workability of fresh concrete, increases the water requirement for a given consistency, increases drying shrinkage at a given water content, and reduces the modulus of elasticity at a given water-cement ratio [72]. These effects are greatest when the recycled concrete is used as both coarse and fine aggregate.

Freeze-Thaw Resistance

Widely varying results have been obtained in tests of freeze-thaw resistance of concrete containing recycled concrete as aggregate. The results relate to many factors, including use of recycled concrete as coarse aggregate alone or as both coarse and fine aggregate, the quality of the original concrete in terms of water-cement ratio and parameters of the air-void system, frost resistance of the aggregates included in the recycled concrete, and presence or absence of purposeful air entrainment in the new concrete containing the recycled concrete. In any event, there is no reason to believe that the recycled concrete should not be required to meet generally accepted standards for quality of the cementitious matrix, air entrainment, and soundness of aggregates when the new concrete is to be exposed to severe conditions of weathering.

Presence of Chemical or Air-Entraining Admixtures

Available data [73] indicate that, when used at generally recommended rates in the original concrete, plasticizing, set-retarding, or air-entraining admixtures in recycled concrete have no significant effect on slump, air content, or setting time of the new concrete or compressive strength of the new concrete after hardening. High concentrations of water-soluble chloride ion in recycled concrete have been shown to contribute to accelerated corrosion of steel embedments included in the new concrete. It is not expected that the chloride-containing admixtures used at ordinary rates as normal-setting or accelerating admixtures in the recycled concrete will influence the setting time of the new concrete.

Presence of Mineral Admixtures

Mineral admixtures such as fly ash, natural pozzolans (raw or calcined), or silica fume included in recycled concrete are unlikely to modify the properties of the new concrete.

Unsound Recycled Concrete

Prospective sources of recycled concrete may be unsound or have been rendered unsound in service as a result of the presence of finely porous aggregate that is susceptible to disruption by freezing and thawing, deterioration by sulfate attack, damage by fire or service at high temperatures, presence of alkali-silica or alkali-carbonate reaction, and so on. Tests and experience show that concrete disrupted by *D*-cracking can be successfully recycled as concrete aggregate, presumably in part because of the reduction in maximum size of the offending coarse aggregate, use of recycled concrete as coarse aggregate only, air entrainment of both the original and the new concrete, reduction of the water-cement ratio, and introduction of fly ash as a partial replacement of the portland cement [69].

Concrete affected by a harmful degree of alkali-aggregate reactivity should be rejected as a source of recycled concrete unless it can be shown that the expansive processes have terminated, the new concrete will serve under conditions in which the internal relative humidity will be maintained at very low levels, say 70 % or less, or that no deleterious effects are found in appropriately extended tests of the proposed concrete mixtures. The possibility of the introduction of additional alkalies into the particles of recycled concrete during the service exposure of the new concrete should be recognized, such as from the cementitious binder of the new concrete or by penetration of alkaline solutions from an external source, such as sea water, alkaline soils, deicing chemicals, or industrial media.

The utility of concrete damaged by fire or high-temperature service must be examined on an individual basis. Portions of the constructions may be suitable for such use. Of particular concern is recycled concrete that contains aggregates including disseminated pyrite (ferrous sulfide, FeS_2), such as certain dolomites and firm shales, where the concrete in service was maintained for long periods at temperatures in the range of 150–300°C (370–575°F). As noted previously [23], pyritiferous dolomite or shales in aggregate can produce distress when the concrete is heated in that range due to expansion of the particles by oxidation and hydration of the pyrite and subsequent crystallization of iron and calcium sulfates. These observations are pertinent to evaluation of recycled concrete derived from concrete exposed to temperatures in this intermediate range, such as structural members adjacent to kilns and furnaces or constituting the frame of such facilities. They pertain also to the use of such recycled aggregates in new concrete intended for these applications.

Chemical Contamination or Radioactivity

Concrete contaminated by noxious, toxic, or radioactive substances should be rejected for use as recycled aggregate. An otherwise desirable source may be salvaged by elimination of excessively contaminated portions of the concrete if they can be isolated and disposed of separately, such as portions of pavements or marine structures in which high concentrations of chloride or water-soluble sodium are found.

Contamination by Bituminous Materials

Recycled concrete containing asphaltic materials, such as bituminous concrete, may or may not affect air entrainment of the new concrete [69]. Enhanced entrainment of air may require use of an air-detraining admixture, but elimination of the contaminant is preferable. The proportions of such materials can be brought to acceptable levels by specifications on removal of overlayments, joint fillers, and the like during the course of demolition and processing of the recycled aggregate.

Metallic Contaminants

Metallic particles or fragments that survive the crushing and screening operations and magnetic separation usually will be in acceptable proportions in the finished recycled concrete. Ferrous particles may cause staining where they lie adjacent to exposed surfaces, especially in the presence of appreciable concentrations of water-soluble chloride salts. Small particles of aluminum metal or galvanized iron may produce blisters or shallow cracking on surfaces of fresh or "green" concrete as a result of the release of hydrogen formed by the interaction with the alkalies within the cementitious matrix.

Glass as a Contaminant

Fragments of plate glass, bottle glass, or glassware may be present in demolished concrete structures and difficult to avoid or to extract during production of recycled aggregate. As scattered particles lying adjacent to exposed surfaces, they can produce popouts and local cracking as a result of the alkali-silica reaction, and unsightly and annoying bulges may develop on floors where the eruptions are confined by flexible coverings. The effects of the alkali-silica reaction may develop in the presence of either low- or high-alkali cements.

Contamination by Brickwork

The report of RILEM Committee 37-RDC [70] concludes that up to 5 % by mass of fragmented brick masonry usually can be tolerated in building rubble for production of recycled-concrete aggregate. On the other hand, highly porous, fired-clay brick may be susceptible to disruption when particles of such brick lie adjacent to surfaces that are exposed to freezing and thawing while wet. The report recommends that particles of brick rubble having a density less than 1.95 g/cm^3 be rejected as lightweight particles in accordance with ASTM C 33.

Refractory bricks having a high content of crystalline magnesia (MgO, periclase) present special problems, such as when recycled concrete is obtained from facilities that include furnaces or kilns lined by such masonry. Large-size fragments of such bricks included in conventional aggregates are known to create cracking, spalling, and popouts as deep as 50 mm (2 in.) in portland-cement structural concrete, pavements, and tunnel linings.

Miscellaneous Contaminants

A Japanese standard for use of recycled concrete limits various impurities as shown in Table 9 [70].

TABLE 9—Recommended Limits on Contaminants for New Concrete to be Subjected to Wetting and Drying or Freezing and Thawing as Reported by Committee 37-RDC, International Union of Testing Laboratories for Materials and Structures

Material	Maximum Allowable % by Volume of Recycled Concrete[a]
Lime plaster	7
Gypsum plaster	3
Soil	5
Wood	4
Asphalt	2
Vinyl acetate paint	0.2

[a] Amount that reduced compressive strength 15 % compared to the control concrete containing recycled concrete.

Stringent limits are required on the allowable concentration of water-soluble sulfates in recycled concrete, such as that derived from gypsum plaster or plasterboard. The RILEM Committee report [70] recommends that the sulfate content of the new concrete be limited to 4 %, expressed as the SO_3 content by mass of the entire concrete, and that the same limit be applied to the recycled-concrete aggregate.

The Committee recommended further that stringent limits be placed on allowable amounts of the contaminants listed in Table 9 for new concrete to be subjected to wetting and drying or freezing and thawing. Noting that organic substances, such as paint, may entrain excessive amounts of air, they recommend a limit of 0.15 % by mass of the recycled-concrete aggregate for organic particles.

Examination of Prospective Sources of Recycled Concrete in the Field

The petrographer can assist in evaluation of prospective sources of recycled concrete by examining the intact structures or pavements at the site of demolition prior to initiation of procedures for aggregate production. This evaluation might constructively designate those locations or portions of concrete placements that are unsuitable or of questionable quality for use as concrete aggregate, such as portions highly contaminated by sea water, industrial chemicals or wastes, or radioactivity, and where extensive fenestration, installations of gypsum plaster or wallboard, or applications of paints or other coatings or overlayments will make difficult the securing of aggregate containing acceptable levels of deleterious substances. Similar avoidance of difficulties may relate to installations of lightweight and porous brickwork or potentially deleterious refractory bricks.

Such inspection should reveal the existence of portions of the installations that are seriously affected by the alkali-silica or alkali-carbonate reactions or severe corrosion of steel embedments. In each instance, use of the concrete in production of recycled concrete aggregate would be brought into question.

Performance of Petrographic Examination of Recycled Concrete

ASTM C 295 does not contemplate petrographic examination of recycled-concrete aggregate. Nevertheless, the procedures for examination of ledge rock, crushed stone, lightweight aggregates, and manufactured sand may be employed as appropriate.

The petrographic examination should be carried out with recognition of the possibility that the aggregate was derived from rubble that comprises two or more classes of concrete of differing composition and condition, possibly from more than one source, and may include contaminants of divergent types.

Objectives of the examination may include evaluation of processing procedures and facilities as well as determination of composition, quality, and anticipated performance of the aggregate in one or more concretes to be subjected to differing levels of attrition, impact, and load and to various types of environmental exposure.

The following features should be observed, recorded, and reported relative to the effectiveness of operations that constitute the production of the recycled concrete:

Grading
Particle shape
Surface texture
Angularity
Coatings of dust of fracture

Variability among nominally similar production lots

Contamination by foreign materials

The following features should be observed, recorded, and reported relative to the composition and condition of the particles:

Hardness (hard, moderately hard, weak, soft)

Relative proportion of mortar and coarse aggregate

Bond of mortar to coarse aggregate

Characteristics of the cement-paste matrix (firmness, absorptivity, bond to fine aggregate, luster on fresh fracture, presence of a mineral admixture, estimated water-cement ratio or water-to-cementitious materials ratio, microcracking)

Air-void content and parameters of the air-void system

Alteration of the cementitious matrix (carbonation, leaching, sulfate attack, staining)

Secondary chemical deposits in included aggregate, air voids, cracks, and cement-aggregate interface

Nature of coarse and fine aggregates (natural, crushed natural, crushed stone, slag, calcined or sintered lightweight aggregate, other)

Lithologic composition of aggregate and foreign materials

Quality of aggregates (sound, unsound, alkali reactive, thermal stability, cement-aggregate reactivity other than alkali-aggregate reaction)

Damage to original concrete during service (freezing before setting, freeze-thaw, leaching, sulfate attack, in situ hydration of iron sulfides, fire damage, high-temperature exposure, other)

Techniques of the petrographic examination directed to these objectives are covered by ASTM C 295, C 457, and C 856. Where the examination, other information, or instructions by the purchaser or supervisor show the need for partial chemical analysis of the recycled concrete or portions thereof, the chemical analytical work and petrographic examination should be closely coordinated and the data mutually exchanged.

Because of its great effect on the properties and performance of concrete containing recycled concrete, Hansen and Narud [71] determined the volumetric ratio of coarse aggregate and mortar in the recycled concrete used in their tests by casting specimens of a mixture of the aggregate and a binder identified by them as “red cement.” Presumably, satisfactory specimens could be prepared with the portland cement blended with a suitable red iron-oxide pigment so as to allow easy distinction between the particles of recycled concrete and the binder. After hardening of the specimen, the relative proportions of coarse aggregate and mortar in the recycled concrete were determined on sawed and lapped surfaces by the linear traverse method. No details were provided as to the explicit criteria applied to distinguish sections of coarse aggregate from those of larger particles of fine aggregate.

This writer has employed the point-count method of ASTM C 457 in analysis of ordinary portland-cement concrete, distinguishing coarse aggregate from fine aggregate by classifying as coarse aggregate all particles that include a dimension greater than 4.75 mm (3/16 in.) in the plane of the section (see the previous section on Petrographic Examination of Aggregates in Hardened Concrete).

In any event, the method of Hansen and Narud can be employed to determine the relative proportions of coarse aggregate and mortar and of coarse aggregate, fine aggregate, and cementitious matrix. The same type of specimen can be used to determine air-void content and other parameters of the air-void system of the recycled concrete in accordance with ASTM C 457, these being features important to the performance of new concrete subjected to moderate or severe conditions of weathering exposure.

As noted previously, detection of rock types or other particles susceptible to the alkali-silica reaction or the alkali-carbonate reaction in recycled concrete presents two general problems: (1) if pre-existing manifestations of one or both of these reactions are found, will they proceed sufficiently to damage the new concrete? and (2) if no evidence of these reactions is seen, will the composition and environmental exposure of the new concrete be such as to initiate and propagate these reactions to a deleterious extent? These questions require careful analysis of all factors concerned, including the kind and amount of alkali-reactive aggregate, the concentration of available alkalies in the recycled concrete, the previous extent of development of distress, the availability of alkalies in the new concrete (including those derived from the cement, mineral admixtures, and other sources), and the availability of moisture to the new concrete during the service exposure.

Conclusion

Petrographic examination should be included in the investigation and testing of concrete aggregate for use in permanent construction and in the manufacture of concrete products and precast structural elements. Applied in the field, the method aids exploration and sampling and permits preliminary evaluation of materials from alternative sources. Detailed examination of aggregates in the laboratory supplements the standard acceptance tests, especially by (1) detecting adverse properties, (2) comparing the aggregate with aggregates for which service records or previous tests are available, (3) explaining results of tests and justifying special tests as required, (4) detecting contamination, and (5) determining the efficiency and relative merit of processing and manufacturing methods for aggregate production.

Validity of the results depends upon the training and experience of the petrographer. However, with proper training and the adoption of uniform techniques and nomenclature, subjective elements in the examination are not significant.

The method can be applied effectively to sand, gravel, crushed stone, slag, natural and synthetic lightweight aggregates, and aggregates produced from recycled concrete.

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Alkali-Silica Reactions in Concrete

David Stark[1]

Preface

THIS CHAPTER IS THE FOURTH OF A SERIES DEALING with chemical reactions of aggregates in concrete. The first version, entitled "Chemical Reactions," was written by W. C. Hansen and was included in *ASTM STP 169A,* published in 1966. It dealt with several types of chemical reactions known at the time to occur in concrete, including alkali-silica reactivity. The second version was written in 1978 by Sidney Diamond for *ASTM STP 169B* and was entitled "Chemical Reactions Other Than Carbonate Reactions." It specifically excluded alkali-carbonate reactivity and emphasized alkali-silica reactivity. The third version, written by the present author, covers only "Alkali-Silica Reactions in Concrete." This fourth edition reviews and updates the topics as addressed in cement and concrete articles and introduces new technology that has been developed.

The shifting emphasis in this chapter since 1966 reflects the predominating concern with alkali-silica reactivity in present-day construction and reflects our updated knowledge of how to deal more effectively with the problem. Accordingly, more detail and emphasis than previously included are provided for the practicing technologist responsible for providing long-term durable concrete.

Introduction

The selection of aggregates for use in concrete was based primarily on physical characteristics such as grading, particle shape, hardness, density, and "cleanness." Such characteristics were the major, if not the only, consideration of the technologist in achieving long-term specified concrete strengths and volume stability. Virtually no attention was given to the chemical or mineralogical composition of the aggregate, despite the known fact that concrete is a highly alkaline system in which pore solutions usually exceed pH values of 13.

In the late 1930s, Stanton, of the California Division of Highways, detected a previously unknown deleterious chemical reaction involving pore solutions in the concrete and certain compositions of siliceous aggregates [1]. He determined that resulting expansion could lead to abnormal cracking, reduction in strength, and early in-service failure of the concrete structure. This phenomenon became known as alkali-silica reactivity (ASR).

By the 1940s, ASR was known to have developed in widespread areas of the United States, and through the following decades, became identified in most areas of the world. The fact that aggregates must be evaluated for chemical stability in concrete as well as for physical competence thus became evident with costly realization. In response to this situation, ASTM developed standards to identify and evaluate construction materials to avoid deleterious ASR. Salient features of ASR, together with a summary of methods of preventing ASR, are given in this chapter.

Symptoms of Alkali-Silica Reactivity

ASR may develop in hardened concrete and may appear with one or more characteristics [2]. Abnormal cracking may develop in a variety of patterns, depending on the design of the structure, reinforcement, detailing, restraints, and exposure conditions, as shown in Figs. 1–3. Figure 1 illustrates typical fine pattern cracking due to ASR. Here, cracks are generally random in many directions and with little, if any, preferred directional restraint to expansion. Figure 2 illustrates well-defined longitudinal cracking along a pavement highway, caused by ASR. Here, maximum restraint to expansion extends parallel to the longitudinal pavement direction, thereby resulting in greater expansion in the transverse direction of the pavement. Figure 3 reveals cracking in a generally horizontal plane in which little restraint develops in the vertical direction. The important point is that the greatest cracking due to ASR can develop in the direction of least resistance, provided uniform restraint on the concrete does not exceed the tensile strength in the concrete structure.

Characteristic features resulting from ASR are shown in Fig. 4. They include reaction rims in peripheries of aggregate particles, microcracks in the concrete, and ASR gel in the microcracks. It should be noted that rims may be due only to weathering in natural gravels and, as such, are not necessarily related to ASR.

The best evidence on which to confirm the development of ASR is the presence of alkali silica gel. It is a reaction product of alkaline solutions in the concrete and reactive forms of silica or siliceous components of the aggregate, which swells and expands with the uptake of moisture. It should be emphasized that simply the presence of gel deposits does not confirm the occurrence of associated distress. Rather, it denotes only that the reaction had developed.

The presence of alkali silica gel can be confirmed by determination of refractive indices using the petrographic microscope, together with applying uranium acetate solution and

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[1] CTL Group, 5400 Old Orchard Road, Skokie, IL.

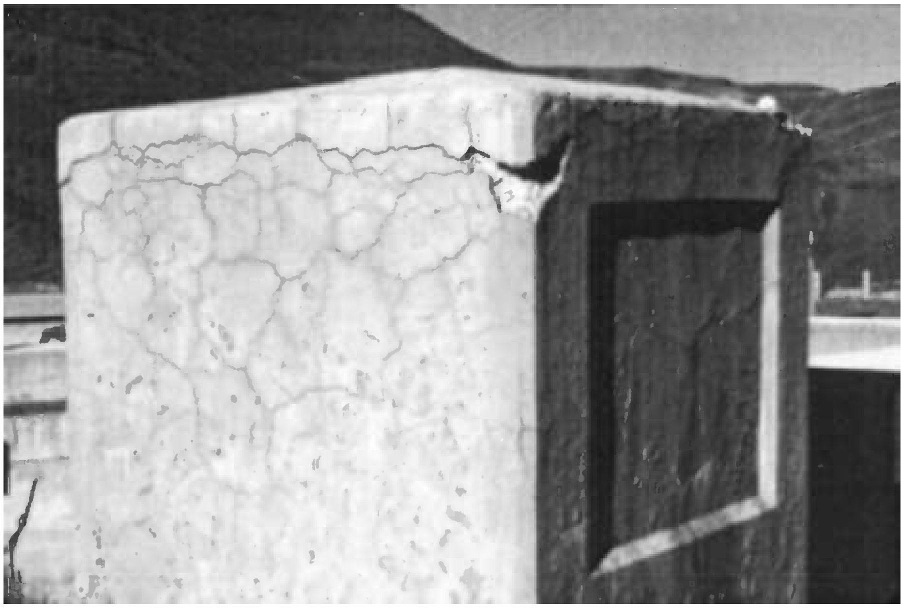

Fig. 1—Fine pattern or random cracking initiated by ASR in a parapet wall of a dam. There is little, if any, cracking relating to differential restraint to expansion. It may be somewhat accentuated by drying shrinkage.

viewing using ultraviolet light in the stereomicroscope, as described in the annex of C 856 [2].

Mechanism of Reactions and Distress in Concrete

Voluminous literature [3,4] has been produced dealing with all aspects of ASR. In its simplest and most straightforward form, the reaction can be described as a two-step process as follows:

Step 1–Aggregate particle + alkali hydroxide solution → Gel reaction product

Step 2–Gel reaction product + Water → Swelling and possible expansion

Each of these factors variably can affect concrete performance as noted below:

1. Hydroxyl ion concentration in the concrete solution is the crucial factor in potential for ASR, not the amount of sodium or potassium in, for example, cementitious materials.
2. Numerous siliceous aggregate materials are not equally deleteriously reactive. For example, opal and volcanic tuff materials are often highly reactive while certain chert or silicate minerals are essentially innocuous.
3. ASR gel may exert variable pressure and expansion in the concrete, depending on composition, viscosity, and moisture availability.

Fig. 2—Longitudinal cracking initiated by ASR in a jointed, slab-on-grade pavement. A longitudinal crack pattern prevails in this pavement, together with minor random cracking, particularly near transverse joints.

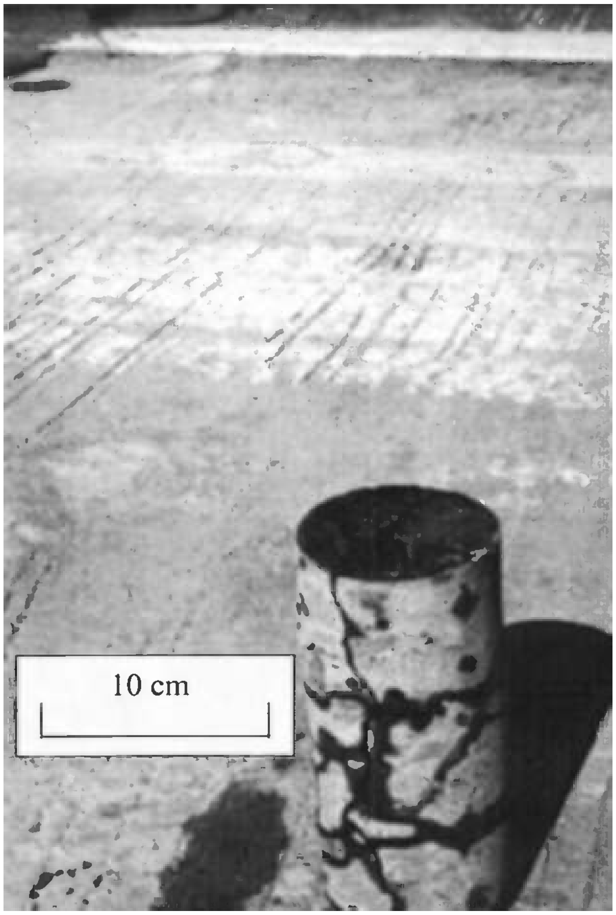

Fig. 3—Vertical concrete core from jointed pavement in which major cracking due to ASR developed primarily in a horizontal orientation. Lesser cracking is evident vertically and diagonally. The horizontal cracking developed normal to the general direction of least resistance to cracking.

Overall, the Powers and Steinour [5] model best fits published results. They proposed that safe reactions are those in which sufficient calcium ion is available in alkaline pore solutions to produce limited-swelling high-calcium-content gel. Conversely, when less calcium ion is available, a higher alkali content gel forms, which has a greater capacity to swell with absorption of moisture.

The initial surface reaction in this model is assumed to occur in contact with the pore solution in the cement paste, which is saturated with respect to calcium hydroxide. It therefore produces essentially a non-swelling reaction product. For a safe reaction to continue, it is assumed that consumption of alkali in the reaction results in greater solubility and availability of calcium at subsequent reaction sites, thereby continuing to produce a high calcium non-swelling gel reaction product. These safe reactions were believed to begin and continue without expansion if the initial alkali concentration is not greater than that produced by cements of about 0.6 % equivalent sodium oxide (Na_2O), or about 0.4 normal sodium hydroxide (0.4N NaOH) concentration in pore solutions at 0.50 water-cement ratio (w/c) by mass.

According to Powers and Steinour, an expansive reaction occurs when alkali concentrations in pore solutions are higher, thus reducing the concentration of calcium ion. Accordingly, a low-calcium, high-alkali unlimited-swelling gel reaction product is formed. In this case, the reacted layer in the aggregate becomes too thick, and the calcium ion cannot diffuse to the reaction site at a sufficient rate to prevent formation of a swelling gel. The diameter of the hydrated calcium ion is substantially larger than that of the hydrated sodium or potassium ion, with the result that they diffuse more readily than the calcium ion to the internal reaction sites within a particle of reactive aggregate. The lower solubility of calcium in solution and the lower diffusion rate of calcium to reaction sites within the particle both serve to reduce its participation in the reaction. However, if the amount of reactive silica, or its fineness, is increased

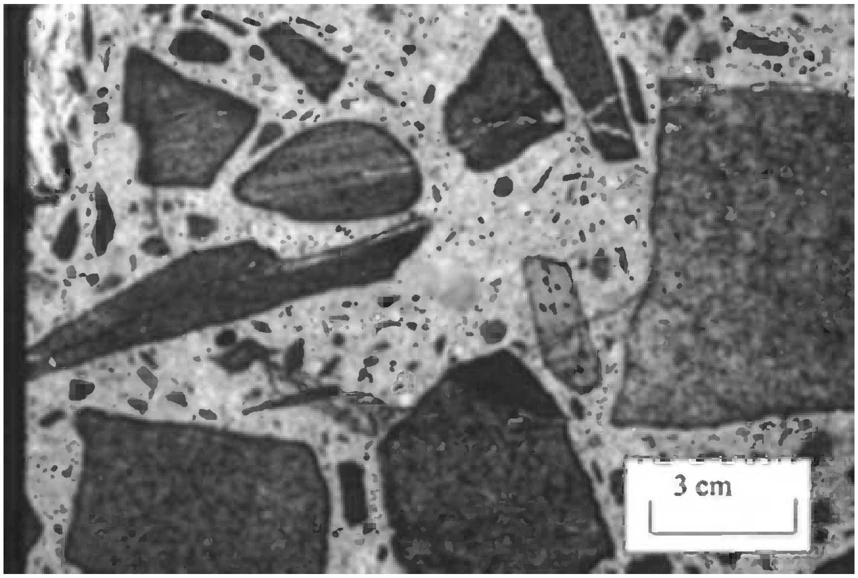

Fig. 4—Evidence of ASR in sample of concrete from gravity dam. Coarse aggregate particles were quarried and composed of shistose to mylonitic composition with the largest aggregate particle having a maximum size of 2 in. ASR-related features include well-defined, dark reaction rims and microcracks through aggregate particles and cement paste matrix that contain white-colored ASR gel deposits.

sufficiently, the alkali content of the solution is rapidly reduced to a safe level so that a higher calcium, limited-swelling gel can form. Obviously, intermediate-alkali and corresponding calcium ion concentrations, diffusion rates, permeabilities of reactive aggregates, and moisture availabilities exist and result in intermediate conditions in which ASR develops.

Another aspect of the Powers and Steinour model is the diffusion of silica away from reaction sites and out of reacted particles. They proposed that silica was able to diffuse out through the reaction layer in the aggregate particle and that expansion-controlling processes were diffusion rates of alkali and calcium to reaction sites. Chatterji [6] suggested that calcium hydroxide plays an additional role in ASR in that it can cause or increase expansions by impeding the escape of dissolved silica from reactive particles. In this circumstance, calcium reacts to form a calcium-alkali silica gel that blocks the escape of other reaction products out of the particle.

ASR gels vary widely in composition, depending not only on circumstances just indicated, but on other factors such as distance from the reaction site and movement along cracks [7]. Electron microprobe and X-ray studies suggest that ASR gels are two-phase composites consisting of the swelling alkali-silicate hydrate phase of relatively narrow composition and a limited swelling calcium-alkali silicate hydrate phase, which varies in both proportion in the gel and composition. Being calcium-bearing, it provides some rigidity to the paste structure. Viscosity of the gel is a major factor affecting swelling pressures generated in concrete.

Types of Reactive Aggregate

Potentially deleteriously reactive rock types probably exist in every state in the United States and in most countries throughout the world. Many natural siliceous rock types are known to have reacted deleteriously with the highly alkaline solutions in concrete. Probably the most widely used of such aggregates are those that contain noncrystalline or poorly crystalline silica. These include opaline and chalcedonic silica that are found in such rock types as chert and flint, as secondary fillings in voids in, for example, basalt, and as interstitial and interlayer material in rock types such as sandstone. Reactive silica of this type also is found as microscopic veinlets or finely disseminated in otherwise innocuous rock types such as limestone and dolomite. The most highly reactive rock types known to the writer are shales that contain opaline diatoms, which have reacted deleteriously and produce popouts within one day of placing the concrete [8].

Deleteriously reactive silica also is present in metamorphic rocks where it is classified as strained quartz and microcrystalline quartz. In these cases, crystal structures are variously distorted, thus rendering them susceptible to deleterious ASR. This imperfectly crystallized quartz has been recognized as the reactive component of such metamorphic rocks as gneiss, schist, metagraywacke, and quartzite. These rock types are slowly reactive and are known to have exhibited deleterious reactivity with high-alkali cements only after five or more years of service.

The other general reactive component of siliceous aggregates is the glassy to cryptocrystalline matrix of volcanic rocks of approximately rhyolitic to andesitic composition. This material, especially tuff, constitutes a major proportion of these rock types and is commonly altered by weathering that facilitates penetration of alkaline solutions into the aggregate particle in the concrete. Weathered volcanic rock is particularly troublesome in that it has produced ASR-associated distress when used with cements with less than 0.60 % equivalent Na_2O. Obsidian and pumice, which are very dense or very porous volcanic glasses, respectively, also are known to have reacted deleteriously in concrete. Slightly metamorphosed volcanic rocks, such as metarhyolite, are also known to produce expansive ASR.

Moisture Availability and Environmental Effects

As indicated, moisture must be sufficiently available in the concrete to permit expansive ASR to occur. Laboratory data indicate that relative humidity (RH) values in the concrete must exceed about 80 %, referenced to 23 ± 1°C, before expansion due to ASR can develop [9]. This condition is easily met in outdoor slab-on-grade concrete, regardless of climate, including desert conditions. Interior portions of elevated concrete members, such as bridge decks, support columns, and girders, also exceed the 80 % RH level, at least on a cyclic basis, even where prolonged severe drying conditions prevail. Except for near-surface concrete in elevated structures, concrete climates can be expected to be sufficiently damp to support expansive ASR on an almost continuous basis. Near-surface concrete experiences major RH fluctuations that support expansive ASR on an intermittent basis, regardless of climate. Where high water-cement ratios are used, residual mixing water may be sufficient to support expansive ASR in mass concrete, even in interior air-conditioned exposures.

Methods of Identifying Potentially Reactive Aggregate

ASTM has five standards available to identify potentially reactive siliceous aggregates. They are:

1. ASTM Standard Guide for Petrographic Examination of Aggregates for Concrete (C 295)
2. ASTM Standard Test Method for Potential Alkali-Silica Reactivity of Aggregates (Chemical Method) (C 289)
3. ASTM Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method) (C 227)
4. ASTM Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar Bar Method) (C 1260)
5. ASTM Standard Test Method for Determination of Length Change of Concrete Due to Alkali-Silica Reaction (C 1293)

Another procedure, ASTM Standard Test Method for Potential Volume Change of Cement-Aggregate Combinations (C 342), was intended to identify expansive cement-aggregate reactions involving so-called sand-gravel aggregates that occur primarily in eastern Colorado, Kansas, Nebraska, Iowa, Missouri, and southeastern Wyoming. The basis for this test was the belief that the cause of distress observed in concrete made with these aggregates was more or less unique to those areas and so was given a separate test procedure. The author believes that alkali-silica reactivity is the major cause of distress in those areas and that the test is of little additional value. This standard was discontinued in 2001.

ASTM C 295–Petrographic Examination of Aggregates

ASTM C 295, together with ASTM C 294, “Standard Descriptive Nomenclature for Constituents of Concrete Aggregates,” is a guide recommended for identifying and describing constituents of aggregate by petrographic examination of concrete. It can be done rapidly but is somewhat subjective in nature, depending on the experience and capabilities of the petrographer examining the aggregate. Basically, results depend on the judgment of the petrographer in contrast to fixed measuring procedures for classifying aggregate. A major use is for alerting materials engineers to the presence of potentially reactive rock and mineral types, and is highly recommended for use in conjunction with other tests and field performance evaluations.

ASTM C 289–Chemical Method

One procedure used to identify whether an aggregate is potentially deleteriously reactive is ASTM C 289, commonly referred to as the Quick Chemical Test. This method was originally developed by Mielenz and co-workers at the U.S. Bureau of Reclamation [10]. In this procedure, the aggregate in question is sized to the 300–150 micron sieves, then three 25-g samples of the material are immersed in *1N* NaOH solution in sealed containers held at 80 ± 1°C for 24 h. The resulting suspensions are then filtered, and the filtrates are analyzed for reduction in the original hydroxyl ion concentration caused by reaction with the test sample and for the amount of silica dissolved in the solution. The result is plotted on a graph where dissolved silica (Sc) in millimoles per liter is plotted logarithmically on the abscissa and reduction in alkalinity (Rc) is plotted on a linear scale on the ordinate. A curve is drawn on the graph in which "Aggregate Considered Innocuous" is located on one side of the curve, and "Aggregate Considered Deleterious" or "Potentially Deleterious" is located on the other. Innocuous aggregates are considered to be those that produce little or no reduction in alkalinity, or those for which a significant reduction in alkalinity is accompanied by relatively little dissolution of silica. Location of the innocuous-deleterious curve on the graph presumably is based on field performance and on ASTM C 227 mortar-bar test results, with the mortar bar criterion being 0.10 % or greater linear expansion.

ASTM C 289 appears to have some serious limitations and is seldom recommended by various organizations and countries in their practices and standards. It fails to identify slowly reactive aggregates, such as gneiss, schist, or quartzite, where reactivity results from strained or microcrystalline quartz. Furthermore, reports have shown that mineral phases, such as calcium, magnesium, iron carbonates, gypsum, and zeolites, interfere in the results of the test, thereby underestimating Sc values or overestimating Rc values, and in the classification of known reactive aggregates as innocuous. On the other hand, the test was found to be severe for several aggregates with good field service records where it was attributed to the fine particle size of the material (150–300 μm) and high test temperature (80°C).

Overall, the test could be used as a quality control tool for aggregate procedures only if results of the test are confirmed by more reliable laboratory tests or field performance records.

ASTM C 227–Potential Alkali Reactivity of Cement-Aggregate Combinations

ASTM C 227 has been an ASTM mortar bar standard since 1950 and was initially conceived by Stanton as a means of identifying safe or unsafe cement-aggregate combinations. As such, it simulated what might actually occur in concrete [1]. Testing was carried out using the aggregate in question, which was sized to a fine aggregate grading. If a coarse aggregate was to be evaluated, it had to be crushed, sized, and washed to the prescribed grading as well. A high-alkali cement, greater than 0.60 % as equivalent Na_2O, or the contemplated job cement, was to be used in the test. Four companion mortar bars were made, then stored over water in a sealed container held at 38 ± 2°C for the duration of the test period. A zero reference comparator reading was made the day following casting, then biweekly to bimonthly to a suggested test period of six months.

The Appendix of the ASTM Standard Specification for Concrete Aggregates (C 33) states that in C 227, cement-aggregate combinations showing expansions greater than 0.10 % at six months "usually should be considered capable of harmful reactivity." The Appendix also states that combinations showing expansions of at least 0.05 % at three months should be considered "potentially capable of harmful reactivity." During the 1950s, the 0.05 % and 0.10 % expansion levels were applied to test ages of six and twelve months, respectively. This then was a stricter test criterion. Today, the 0.05 % criterion at three months is considered innocuous if the six months criterion is less than 0.10 %.

Many years of experience with C 227 have revealed shortcomings that, in some cases, have resulted in misleading conclusions with eventual deterioration of concrete. The suggested expansion criteria appear to be valid for rock or mineral types arbitrarily defined as rapidly reactive aggregates, such as opaline-bearing materials and weathered glassy to cryptocrystalline volcanics of rhyolitic to andesitic composition, when they are tested with cement with 1.0 % alkali. C 33 leaves open to the engineer's discretion the option of extending the test period to, for example, 12 or more months. However, extending the test period to more than 12 months still may result in failure to define so-called slowly reactive aggregates as potentially deleteriously reactive in field structures. These aggregate types include certain gneisses, schists, graywackes, and metavolcanics. Even when used with cements with 1.0 % alkali, expansions for these rock types may reach no more than 0.05 % after two to three, or more, years [11]. Such a timeframe is not practical in these cases.

A further limitation from C 227 results is a recommendation to use low-alkali cement (less than 0.60 % equivalent Na_2O) if an aggregate is judged to be potentially deleteriously reactive. Field and laboratory studies [11] have revealed that certain aggregates, such as glassy volcanics or tuffs, are deleteriously reactive with low as well as high-alkali cements.

Leaching of alkalies from the concrete also has been found to be a serious limitation on expansion [12,13] in C 227. An aggregate may be judged to be innocuous when used with cement of a certain alkali level when, in fact, it may be deleteriously reactive.

Overall, C 227 may be useful for identifying certain aggregates as deleteriously reactive. However, many cases exist where the procedure falls short of expectations.

ASTM C 1260–Potential Alkali Reactivity of Aggregates

It is evident that limitations exist in ASTM C 227 and C 289 for identifying known slowly reactive aggregates such as gneiss, graywacke, some siliceous limestones, schist, and quartzite. Thus, a test method was developed in South Africa at the National Building Research Institute [14] and was designed to rapidly identify slowly and highly reactive aggregates, based on field performance record. With some modifications in the late 1980s, it became an ASTM standard in 1990 and is known as the Potential Alkali Reactivity of Aggregates (Mortar Bar Method).

For this method, the aggregate is sized and washed to meet the specified grading, then used at a proportion of one part of cement and 2.25 parts of aggregate, then cast into mortar bars of 0.47 water-cement ratio by mass, and stored in a moist cabinet at 23 ± 2°C. One day after casting, the specimens are removed from the molds, a comparator reading is obtained, then it is stored for 24 h in water brought to 80 ± 2°C. An additional comparator reading is obtained, then the bars are transferred to *1N* NaOH solution and again stored at 80 ± 2°C in sealed containers for 14 days. Periodic comparator readings, for example, at 1, 3, 7, 10, and 14 days, are made, while being careful to prevent cooling and drying while taking the readings. Total time required between making the mortar bars and obtaining the final measurement of length change is 16 days. Test criteria suggested in the Appendix of C 33 are as follows:

- Expansions in NaOH solution greater than 0.20 % at 16 days indicate potentially deleterious expansion.
- Expansions between 0.10 % and 0.20 % are uncertain but are known to include aggregates that can be deleterious or innocuous in field concrete.
- Expansions not greater than 0.10 % represent innocuous aggregate.

Several important points must be emphasized in this procedure. First, the alkali content of the cement used in making the mortar bars may be comparatively low because a much greater NaOH concentration is used in the immersion solution and diffuses into the mortar bars, thus exerting overwhelming control on reactions. Second, the test result indicates only that the aggregate may be potentially reactive, not that it will react deleteriously in field concrete. Cement alkali level and environment exert control on field performance. Third, the test has successfully identified, in conformance with field observations, the deleterious nature of reactivity with most slowly reactive aggregates, such as certain gneisses, schists, and quartzites. Overall, this procedure is rapid but may overstate the shortcomings found in ASTM C 227 and C 289.

ASTM C 1293–Length Change of Concrete Due to Alkali-Silica Reaction

ASTM C 1293 is considered by some to be the most reliable procedure among ASTM test methods to evaluate aggregates for alkali silica reaction. It allows for the evaluation of aggregates as they are used in concrete. Coarse aggregate can be evaluated without crushing them, or fine aggregates can be tested without sieving and recombining according to prescribed particle size distributions. It also is the only method where combinations of coarse and fine aggregates proposed for use in job applications can be tested.

In general, the test is effective in recognizing the potential alkali silica reactivity of both the rapidly reactive aggregates, such as opaline-bearing, glassy cryptocrystalline volcanic rocks, as well as the slowly reactive aggregates such as granite gneiss, graywackes, schists, and some sandstones and limestones.

In this procedure, equivalent alkali in the cement plus NaOH solution added to the mixture should be 1.25 % as Na_2O, and the w/c should range between 0.42 and 0.45. Three companion concrete prisms are cast, cured, and stored in a moist environment in a sealed container at 38 ± 2°C for up to 12 months or more as needed. C 33 indicates the concrete would be considered deleterious if expansion reaches 0.040 % or more at 12 months.

Several limitations exist in the C 1293 standard. First, it may require a year or more to obtain a meaningful result, depending on the nature of the aggregate in question. Second, an alkali level of 1.25 % as equivalent Na_2O of the cement plus NaOH solution is much greater than most alkali levels in field concrete and therefore cannot provide realistic safe cement alkali levels for the aggregate tested.

To help resolve any of these issues, recent work has shown the potential for an accelerated version of the test to be performed at 60°C at greater than 95 % relative humidity over a three-month period. Earlier, Stark et al. [15] used fine and coarse aggregate, received as concrete aggregate. Tests of concrete prisms up to 42 days in 1*N* NaOH solution at 80°C showed some success. Other specimens were cast with the same alkali level as that used for the immersion test, also with some success.

Overall, none of the existing ASTM test methods provide cement alkali levels and environments that can reproduce given cement-aggregate combinations that exactly prevent or allow deleterious ASR. Existing tests are either too lenient, too severe, or too time-consuming. One rapid procedure that has been developed, and can include mineral admixtures, is not yet an ASTM Standard [16].

Avoiding Expansive Alkali-Silica Reaction

Three methods are used today to prevent abnormal expansions due to ASR. They are:

1. Avoid the use of reactive aggregate.
2. Limit the cement alkali level.
3. Use pozzolans or other admixtures in the fresh concrete.

The most feasible method for a particular job will depend on economics and the local availability of suitable materials. In some cases, combinations of the various methods have been used. Each of the three methods is discussed below.

Use Innocuous Aggregate

At first glance, it would seem a simple matter to use innocuous aggregates to avoid expansive ASR. However, this presumes that the innocuous aggregate has a proven service record and has been evaluated properly using pertinent ASTM standards. If a field service record is to be used, assurance must be made that the prospective aggregate from a commercial or lithologic source is of similar petrographic character as that on which the service record is based. If quarried stone is being considered, for example, one must be careful to evaluate the same rock strata, particularly where bedding is convoluted or inclined. If water-laid deposits from a given source of sand or gravel are being considered, one must be sure to evaluate materials from, for example, the same river terrace level. Petrologic character of the source material can vary significantly not only vertically from bed to bed, but also laterally. Variations often develop in mineralogic composition, proportion, and particle size of individual rock types in the prospective aggregate source. Use of an alternate source of a durable aggregate may result in major transportation costs for shipment to the job site.

The use of field service record also requires that cement alkali level be known for the existing concrete, regardless of whether low or high alkali cement was used previously and will be used in new construction. An aggregate that is known to be slowly reactive, when used with a more rapidly reactive aggregate, may then become innocuous due to the remaining lower concentration of alkali in solution in the concrete.

A second major factor in evaluating field service record is the environment to which the concrete is exposed. This includes temperature, moisture accessibility, and restraint. Elevated temperatures and ready availability of moisture are known to exacerbate alkali-silica reactivity, whereas restraint or confining pressure reduces expansion and reactivity-induced distress.

Deleterious ASR has been observed in concrete structures in all habitable climates, including hot and cool damp regions where the concrete is exposed directly to the ambient outdoor conditions. Moisture usually is sufficiently available, either continuously or cyclically, to support expansive ASR, and that temperature change, in a practical sense, serves only to change the rate of reaction. Commonly, increases in ambient outdoor temperature are accompanied by reductions in relative humidity with possible offsetting effects on ASR. For example, increase in temperature may increase the rate of chemical reaction, but drying would remove water that might otherwise be available for absorption by and resulting expansion of ASR gel. Water-cement ratio also will affect reactivity, with residual mixing water possibly being the only source of moisture available to support expansive reactions.

A third factor that has been attributed to field performance is extraneous sources of alkali, specifically, hydroxyl ion. Examples include sodium chloride (NaCl) deicer salts, alkali salts occurring naturally in soils [e.g., sodium sulfate (Na_2SO_4)], industrial solutions (e.g., NaOH), and seawater. Calculations suggest that many times the quantity of alkali present in the cement can diffuse into concrete from external sources, possibly increase hydroxyl ion concentration, and perhaps convert an otherwise innocuous cement-aggregate combination into a deleterious one. Inspection must consider all such factors in any assessment of field performance relating to potential for deleterious ASR.

Results of laboratory research have alluded to so-called pessimum (intermediate) proportions of reactive aggregate at which maximum expansion rates and levels develop [5]. This appears to be largely of academic interest except to explain some case histories in which all of the aggregate was reactive but no damage resulted, while in related cases only a relatively small proportion of reactive material was used and damage resulted. Attempting to specify aggregates for field concrete on the basis of avoiding pessimum ratios should be avoided.

Limit Cement Alkali Level

As noted earlier, cement alkali level is a major factor determining expansion due to ASR. In general, the higher the cement alkali level, the greater will be expansion due to reactivity. Simply specifying low-alkali cement (less than 0.60 % equivalent Na_2O) to avoid expansive ASR has not always been successful. In some cases, cement alkali levels as low as 0.45–0.50 % or less have failed to prevent expansive reactivity where partially weathered glassy to cryptocrystalline volcanic rock of rhyolitic to andesitic composition constitute no more than 5 % of the aggregate [11]. In this regard, alkalies from, for example, feldspars or noncrystalline volcanic aggregates can be released by water or calcium hydroxide solutions, thus resulting in the development of expansive ASR. The best indicator presently available to determine a safe cement alkali level is field performance record where all pertinent factors are properly considered.

Deleterious ASR also can occur in concrete in which cathodic connection or stray current systems develop [17,18]. It appeared that current densities of at least $25 mA/m^2$ caused the greatest expansion when the cement alkali level was high. However, there was no expansion when the cement alkali level was low.

Use Admixed Materials

Certain finely divided ground materials may be added to concrete to permit the safe use of otherwise potentially reactive cement-aggregate combinations. These materials are intended to react in the normal course of cement hydration by complexing alkalies to reduce hydroxyl ion concentrations in concrete, or by altering diffusion rates of alkali and lime to reaction sites. This results in the formation of harmless nonexpansive reaction products. The most widely used material for this purpose is fly ash obtained from power plants burning coal. Other, less commonly used materials include ground granulated blast-furnace slag, finely ground volcanic glass, and silica fume. In addition, lithium solutions, which have been found to be extremely effective in reducing expansions in experimental work, are contemplated for the future. The use and testing of these materials are discussed below.

Fly Ash

Two categories of fly ash are recognized in ASTM Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete (C 618): Class F and Class C. For purposes of preventing expansive ASR, the most important distinction is the minimum permissible proportion of $SiO_2 + A1_2O_3 + Fe_2O_3$, which for Class F ash is 70 % and for Class C ash is 50 %. Class C ashes usually have greater lime (CaO) contents, often exceeding 10 % and even 20 %. High lime contents adversely affect the capability of a fly ash to prevent expansive reactivity [19]. An optional requirement of fly ash is a 1.5 % maximum limit on "available" alkalies as determined by methods described in Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland Cement Concrete (C 311). The latter restriction is important because total alkali content of cement + fly ash may result in the equivalent of very high alkali levels that may convert an otherwise innocuous cement-aggregate combination into a deleterious one [20].

ASTM provides several testing procedures to determine the suitability of a fly ash for controlling expansive ASR in concrete. The Standard Test Method for Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction (C 441) involves replacing 25 % of the cement, by mass, with an amount of fly ash equal to the volume of cement replaced. The alkali content of the cement should be about 1.0 %. Highly reactive Pyrex glass is used as fine aggregate in a specified grading, and the water content is gauged to meet a flow requirement. Companion mortars are made with and without the fly ash. Test storage is over water in sealed containers held at 38 ± 2°C. Expansion using the fly ash is determined at 14 days, and the absolute expansion level of the test mortar is evaluated.

ASTM C 618 requires expansion of the test mixture to not exceed 100 % at 14 days when using low alkali cement.

C 441 has been criticized for a number of reasons. First, specific Pyrex glass is used as the standard reactive aggregate. It contains appreciable alkali that, it has been argued, interferes with proper evaluation of the pozzolan. Of much greater importance is the fact that Pyrex glass reacts significantly faster than many fly ashes, while in field concrete containing natural aggregate, the reverse is probably true. For this same reason, meeting the C 618 expansion criterion at 14 days for the test mortar will be difficult, if not impossible, to meet, even though the fly ash might effectively suppress expansive alkali-silica reaction.

Raw or Calcined Natural Pozzolan

Remarks presented for fly ash apply generally to raw or calcined natural pozzolans, such as some volcanic ashes, opaline shales, and diatomaceous earths. However, C 618 recommends 75 % reduction in expansion after 14 days under C 441 test conditions, as noted earlier.

Ground Granulated Blast-Furnace Slag (GGBFS)

Ground granulated blast-furnace slag can be used to prevent expansive ASR. C 441 is used to evaluate slag as well as fly ash, except that 50 % by mass of portland cement is replaced by slag equal to the volume of cement replaced. ASTM Standard Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortar (C 989) suggests that reduction in expansion should be at least 75 %.

The mechanism by which slags inhibit expansive reactivity is purported to be different than that by which fly ashes prevent expansion. From laboratory investigations [21], it was concluded that slag hydration in portland cement-water systems serves to reduce diffusion rates of alkali to aggregate reaction sites, thereby minimizing the rate of ASR. This would have the effect of producing a potentially less expansive or nonexpansive gel reaction product. This explanation needs further confirmation.

Silica Fume

Silica fume is occasionally used to prevent expansive ASR. It appears to function in much the same manner as fly ash in reducing expansions due to reactivity. That is, the calcium silicate hydrate reaction product complexes alkali, thereby reducing the hydroxyl-ion concentration in the pore solution in the concrete. Standard Specification for Use of Silica Fume as a Mineral Admixture in Hydraulic Cement Concrete, Mortar, and Grout (C 1240) is used. To prevent ASR, 10 % silica fume by mass of cementitious material will be used [22,23].

Metakaolin

Metakaolin is a relatively recent material developed to prevent deleterious ASR in concrete [27]. It is derived from the weathering of feldspar in granitic rock types and manufactured by calcining the kaolin to a temperature range of 700–800°C. After grinding to pozzolan fineness, it is used to replace up to 10 % of the portland cement with metakaolin [24].

Lithium Salts

McCoy and Caldwell [25] first reported the exceptional capability of lithium salts to inhibit expansive ASR. They found that small threshold quantities of any of several such salts could prevent expansion when used with high-alkali cement and Pyrex glass. Later work [26,27] verified these findings using highly reactive commercially available aggregate. Here, it was recommended that lithium hydroxide (LiOH) salt be used because it is soluble in tap water, unlike lithium fluoride or lithium carbonate, thereby permitting better dispersal in large concrete batches. It also has been included in experimental field concrete pavement. Currently, there are no specification guidelines for its use, but the experimental laboratory work suggests a dosage rate of about 1:1 $LiOH \cdot H_2O$: equivalent Na_2O in the portland cement. Today, lithium nitrate is often used to reduce potential hazardous effects from the user [27].

An important advantage of lithium salts over other admixed materials is the fixed composition of the salt, thus requiring no testing for individual concrete mix materials [21]. ASTM presently does not address this means of preventing expansive ASR. A very beneficial but, today, not practical use for lithium solution would be its penetration to uncracked hardened concrete to prevent or mitigate distress due to ASR [27].

Concluding Remarks

The concrete technologist has at his disposal broad knowledge and guidelines to produce concrete that is not susceptible to deleterious ASR. ASTM provides various test procedures and recommendations that, if used prudently with other information noted in this chapter, should ensure that deleterious reactions can be avoided in future construction.

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35

Alkali-Carbonate Rock Reaction

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Preface

THE SUBJECT OF ALKALI-CARBONATE ROCK REACTIVITY (ACR) was discussed for the first time as a distinct and separate chapter-length topic in *ASTM STP 169B* by H. N. Walker [1]. Prior to that, W. C. Hansen, in *ASTM STP 169A* in 1966 [2], discussed the reactivity of dolomitic limestones, along with the reactivity of other rocks and minerals, under the general heading of chemical reactions. Previous editions of *Significance of Tests and Properties of Concrete and Concrete-Making Materials (ASTM STP 169,* 1956) and its predecessors *ASTM STP 22A,* in 1943, and *ASTM STP 22,* in 1935, did not treat the subject of deleterious carbonate rock reactions because such reactions were not recognized as a distinct problem until identified as such by Swenson in 1957 [3], based on his investigations of expanded, cracked, and buckled concrete in the vicinity of Kingston, Ontario, Canada.

In addition to the now numerous occurrences of reactive carbonate rock in concrete in Ontario, in the United States there have been affected concretes in Virginia, West Virginia, Kentucky, Missouri, Tennessee, Iowa, Illinois, Indiana, and New York; and elsewhere in the world in Iraq, Bahrain, England, and China.

Introduction

When a portland cement concrete mixture is designed and placed, it is expected that the coarse and fine aggregate will not participate in any chemical reactions with components of the cement paste that will cause the aggregate particles to crack or to become otherwise dimensionally unstable.

Historically, there was a time when that expectation was satisfactorily fulfilled, or at any rate believed to have been satisfactorily fulfilled, because for certain instances of concrete deterioration, the reasons for that deterioration were ascribed to causes other than deleteriously reactive aggregates.

The satisfactorily innocuous, or "inert," behavior of aggregates was, at that time, favored by such factors as:

1. Continued availability of aggregate materials from "old" sources, those materials that had performed without problems over years of consumption.
2. The general availability of low-alkali cement that was furnished as a matter of course during the years when kiln dust was not included in the product.

As demand for concrete increased, in large part to meet the requirements of transportation-related construction, new sources for aggregates were necessarily developed. It should be noted that new sources include previously unused or undeveloped strata or portions of old operating limestone quarries as well as new "greensite" quarry sources. And, at around the same time, in the late 1960s and early 1970s, in response to economic and environmental constraints, cement kiln dust, which is richer in alkalies than the cement product, began to be returned to the product. Thus, the preceding Factors 1 and 2 no longer favored so much the satisfactorily innocuous behavior of aggregates with respect to alkalies in the cement, and instances of deleterious behavior became more numerous.

The following ASTM documents are directly applicable to the subject of alkali-carbonate reactivity and are listed with a notation as to their particular relevance.

ASTM Standard Specification for Concrete Aggregates (C 33)

Directs that deleteriously reactive aggregates shall not be used in moisture-exposed concrete made with cement containing more than 0.60 % total alkalies and, in the Appendix, describes the petrographic characteristics of potentially reactive carbonate rocks and, in part, how to evaluate them.

ASTM Guide for Petrographic Examination of Aggregates for Concrete (C 295)

States that petrographic examinations should identify and call attention to potentially alkali-carbonate reactive constituents, determine their amounts, and recommend additional tests to determine whether those amounts are capable of deleteriously affecting concrete. Reference is made to examination of drilled core and ledge rock. These topics are of particular importance since it is more often the case that alkali-carbonate reactive rock comprises a relatively small stratigraphic portion of a producing quarry.

ASTM Descriptive Nomenclature for Constituents of Natural Mineral Aggregates (C 294)

Describes the characteristics, texture, and composition of carbonate rocks that are potentially alkali-reactive, in essentially the same language as is in ASTM C 33.

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ASTM Test Method for Potential Alkali Reactivity of Carbonate Rocks for Concrete Aggregates (Rock Cylinder Method) (C 586)

Directly measures the expansion of small cylinders of rock during exposure in sodium-hydroxide solution. The rock sample is secured in accordance with the applicable requirements of ASTM D 75. Positive test results cannot, by themselves, be used as an accurate predictor of the damage that the rock will cause in concrete. ASTM C 1105 (see next paragraph) should be used to evaluate the behavior of the rock in concrete.

ASTM Test Method for Length Change of Concrete Due to Alkali-Carbonate Rock Reaction (C 1105)

Directly measures the expansion produced in concrete prisms, stored at 23°C and 95 % RH, by the alkali-carbonate reaction, using up to 19.0 mm (3/4 in.) maximum size aggregate, and the job cement.

ASTM Practice for Examination and Sampling of Hardened Concrete in Constructions (C 823)

Provides general and specific guidance for evaluating constructions in the field and for obtaining samples for laboratory examination as by ASTM C 856 (see next paragraph), with which it may be used in an interactive way, with ASTM C 856 providing preliminary information for detailed and rigorous application of ASTM C 823 to provide a second set of samples for petrographic examination by ASTM C 856. ASTM C 823 is of particular relevance when evaluating an aggregate source for alkali-carbonate reactivity based on its service record in previous constructions. In that connection, it is important to have information on the alkali content of the cement used, and the cement content of the concrete in the constructions.

ASTM Practice for Petrographic Examination of Hardened Concrete (C 856)

Provides the general method for how to go about examining field and laboratory concrete chiefly microscopically to establish whether alkali-carbonate reactions have taken place, and gives some specific criteria for recognizing the effects of alkali-carbonate reactions.

ASTM Practice for Sampling Aggregates (D 75)

Gives the procedure for obtaining samples for examination and testing by ASTM C 295, C 1105, C 586, and any other tests and examinations from which general conclusions regarding the aggregate may be drawn. It should be noted that ASTM C 586 is best, and most conveniently, done on ledge rock samples that will permit the preparation of three mutually perpendicular small rock cylinders. Information regarding obtaining ledge rock samples is in ASTM D 75. Alternatively, in the event that ledge rock samples cannot be obtained, it is possible that the small cores (1.38 in., 35 mm) needed for ASTM C 586 can be drilled out of larger particles—for example, 2 in. (50 mm) or larger—of aggregate obtained from stock pile or conveyor belt.

ASTM Method for Reducing Field Samples of Aggregate to Testing Size (C 702)

Samples taken in accordance with ASTM D 75 may be reduced in size by quartering or by use of a mechanical splitter as described in ASTM C 702.

Types of Carbonate Rock Reactions

Although various reactions involving carbonate rock, both dolomitic and nondolomitic, in concrete or mortar have been recognized, only one, the expansive dedolomitization reaction, which is the principal subject of this chapter, is of importance with respect to having produced significant damage in concrete constructions. This reaction may or may not produce rim zones on particles of reacting rock in concrete.

Other reactions, causing the production of rim zones and other internal changes in carbonate rocks of various compositions are of scientific interest. Some other reactions are discussed in Refs 1, 4, and 5. Katayama [6] gives a detailed review of rim formation.

Expansive Dedolomitization Reaction

The expansive dedolomitization reaction occurs between alkaline solutions (almost always sodium and potassium hydroxides derived from the cement[2]) in the cement paste matrix and limestone aggregate particles of particular but nevertheless somewhat variable compositions and textures. However, a feature common to all such limestones is that there will be small dolomite crystals present in the matrix of the rock that will react with the alkaline solutions in a manner such that the dolomite crystals, of original composition $\mathrm{CaMg\ (CO_3)_2}$, become other minerals and compounds; hence, the term dedolomitization, as represented, simply, here, and as discussed in greater detail in subsequent sections.

calcium magnesium carbonate (Dolomite)
+ alkali hydroxide solution → magnesium hydroxide
+ calcium carbonate + alkali carbonate

The chief physical result of dedolomitization, which distresses and damages concrete, is that the reaction causes the affected aggregate particles to expand and crack and thereby to extend the cracks into the enclosing mortar matrix.

Manifestations of Distress

Concrete that is affected by the alkali-carbonate rock reaction expands and cracks. Depending on the perfection of the petrological characteristics of the rock that promote expansion, and the convergence of the environmental and materials-related factors that promote expansion, important among which are availability of moisture and alkali content in the concrete, the concrete may in a matter of months exhibit severe surface cracking accompanied by closing of joints, as was reported in Canada by Swenson [3]. Pattern cracking after three years of exposure is shown in Fig. 1.

When all of the factors favoring alkali-carbonate reaction combine to produce near-ideal conditions for causing the reaction, “spectacular expansion and deterioration,” as reported by

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[2] Alkalies from deicing materials such as sodium chloride (NaCl) may augment the alkalies supplied by the cement.

Fig. 1—Typical cracking of concrete slab by alkali-carbonate reaction after three years, Ontario, Canada [7].

Rogers [8], can occur within three years. In that instance, the concrete in sidewalks, curbs, and gutters expanded about 1.2 % (Fig. 2) with consequent compressive failures, blow-ups, and shoving of adjacent asphalt pavement. It is reported that the alkali content of the cement was about 1.0 % Na_2O equivalent, and the cement content of the concrete was about 300 kg/m^3 (500 lb/yd^3).

Fig. 2—Concrete slab affected by alkali-carbonate reaction (upper portion of photo) that has moved 9 cm to the right relative to the concrete in the lower portion of the photo that has nonreactive aggregate. Expansion was measured at 1.2 % in three years, Ontario, Canada [48].

In ACR-affected field concrete, there is no definitive feature of the concrete or of the geometry of the cracking that precisely identifies the cause of the cracking as being alkali carbonate-rock reaction. (In contrast, gel deposits, if present, may provide a strong indication that cracking is due to alkali-silica reaction but not exclude the possibility that both reactions can be present.) The crack geometry, or pattern, produced is the response of the concrete to internal expansive force based on its particular size and shape and the direction of maximum moisture availability. Generally, expansion of ordinary, plain, cast-in-place concrete as in sidewalks, floors, decks, slabs, and footings where there is a moisture gradient from top to bottom or from side to side results in pattern cracking similar to that shown in Fig. 1. Where moisture conditions are uniform or where the concrete elements are thick, or of low surface area-to-volume ratio, bulk expansion of the concrete is evidenced by closing of joints, extrusion of filler material, shoving or buckling of adjacent materials, or crushing of adjacent weaker concretes. Cracks may be prominent or faint–visible only following wetting of the surface to produce some contrast–and concrete between the cracks can be hard and intact.

Conclusive evidence that the observed distress has been caused by alkali-carbonate reaction is best obtained by detailed examination of the concrete following ASTM C 856, during which it will be observed that the aggregate exhibits the characteristic microscopic texture and composition of alkali-reactive carbonate rock (see the section on Characteristics of Alkali-Reactive Carbonate Rocks), and that internal cracking in the aggregate extends into the mortar.

Reinforced and post-tensioned concrete, precast and prestressed concrete, and other kinds of prefabricated concrete can develop individualized cracking responses to internal expansion that may not be similar to those produced in slabs on grade. For example, elongate prestressed or post-tensioned pieces typically develop long cracks parallel to the direction of tensioning or prestressing.

In at least one particular type of exposure condition, the manifestation of distress due to ACR does not involve gross expansion of the concrete and widespread cracking, but instead, the damage is confined to the surface region and is the result of the application of deicing salts. For example, a parking deck investigated by the author containing reactive carbonate aggregate throughout was deeply scaled exposing numerous cross-fractured coarse aggregate particles in the scaled surface. Only the coarse aggregate particles in the near-surface region had cracked, extending cracks into the mortar to connect with similar cracks from other reactive coarse aggregate particles, with the result that the surface concrete was lost. Although the deck has reactive aggregate throughout, the alkali content of the concrete, in the absence of deicing salts, was insufficient to sustain the reaction and to develop the overall expansion and tensile cracks producing the map, or pattern cracking, that is usually observed.

Smith [9] has presented experimental data showing the exacerbating effect of sodium chloride (NaCl) on alkali-carbonate reactivity. Using concrete prisms, made with a 1.1 % alkali cement and a known reactive aggregate, stored in saturated NaCl solution at 70°F (21°C), he recorded about 0.19 % expansion at 300 days. The same concrete stored in pure water at the same temperature showed 0.12 % at 300 days.

Using reactive aggregate from the same quarry (Pittsburgh quarry at Kingston, Ontario) about 27 years later, Alasali et al. [10] recorded comparable results, measuring 0.23 % expansion at 275 days on concrete prisms made with 1.13 % alkali cement stored in 5 % NaCl at 23°C. A similar prism stored in water at 38°C measured 0.155 % expansion at 275 days.

Gillott and Rogers [11] showed that alkali contributed by certain rare types of mineral components in the coarse aggregate (e.g., dawsonite) can exacerbate the ACR.

Comparison of Alkali Carbonate Rock Reaction with Alkali-Silica Reaction as Regards Generation of Crack Damage

In alkali silica reaction (ASR), the first damage to the concrete can be produced by both (a) internal cracking of aggregate particles extending cracks into the paste and mortar, and (b) extension of existing cracks and production of new cracks as a consequence of the migration and subsequent expansion of the ASR gel, initially produced by the reacting particles, away from the particles and into cracks and voids in the concrete.

In contrast, direct damage due to alkali carbonate reaction is produced by the cracking, and extension of cracks into the mortar, generated within the reactive coarse aggregate particle itself.

The initial, or direct, damage produced in both ASR and ACR may then be exacerbated by the action of cyclical freezing and thawing. And, it should be recognized that, although bulk expansions of concrete produced solely by the alkali-carbonate reaction may be lower than those produced in severe alkali-silica reaction, the ultimate damage to the concrete can be of the same order as produced by ASR if the concrete is exposed in an environment where cyclical freezing and thawing occurs [12]. That is, the initial cracks produced by the alkali-carbonate reaction are then exploited by saturation of the cracks and subsequent freezing.

Characteristics of Alkali-Reactive Carbonate Rocks

Petrographic

The alkali-carbonate reactive limestones that were first studied in Canada, and shortly thereafter in the United States, have characteristic microscopic textures and mineralogical compositions which, by precedent, have been regarded as prototypical. However, as will be noted subsequently, there are expansive, dedolomitizing, alkali-carbonate reactive rocks that do not conform to the historical prototype [13,14]. The classic, or prototypical, microscopic texture and structure is characterized by relatively larger, rhombic crystals of dolomite [$CaMg(CO_3)_2$] set in a finer-grained matrix of calcite [$CaCO_3$], clay, and (commonly) silt-sized quartz. The characteristic composition is that in which the carbonate-mineral components of the rock consist of substantial amounts of both dolomite and calcite, and the dilute hydrochloric acid (HCl) insoluble residue contains a significant amount of clay.

Examples of typical microstructures of dolomitic limestones known to be alkali-carbonate reactive are shown in Fig. 3.

The rhombic crystals of dolomite occurring in the matrix of the reactive rock may be relatively sparsely distributed and appear to be "floating" in the background (Fig. 3, Field A) or may be more crowded together with dolomite rhombs adjacent or touching (Fig. 3, Field E).

The grain sizes of the calcite and clay matrix in which the dolomite rhombs are set is typically 2 to 6 μm for the calcite with smaller clay particles disseminated throughout. It is often the case that silt-sized quartz grains are also disseminated throughout the matrix.

The typical texture that is described here is found in those reactive carbonate rocks that are identified as early expanders, showing rock prism expansions (ASTM C 586) of a few tenths percent in a matter of weeks, and field manifestations in concrete within perhaps one year after construction.

A modification of the typical texture is found in reactive carbonate rocks identified as late expanders that may not show noticeable rock prism test expansions until approximately 25 weeks (rarely, not before one year) and that may not show manifestations in concrete after five years in moist storage. In those rocks, the typical feature of dolomite rhombs in a clayey fine-grained matrix is consistent, but the matrix is coarser-grained and is composed of interlocking dolomite grains together with calcite, clay, and, more commonly than not, silica minerals. Those differences between early and late expanders are reflected in the bulk compositions, as shown in Table 1, and in differences in internal textural restraint, or rigidity, as investigated by Hilton [15,16] and discussed subsequently. Whether the possibility of late (rock prism) expanders causing distress in field concrete after many years of exposure should be disregarded is an open question.

The above description of the alkali-carbonate reactive texture derives from the "type locality" in Ontario, Canada where the rocks were first identified and described. And, the description has historically served well as a prototype for recognizing the ACR texture in North America and elsewhere. However, extensive studies on ACR rocks in China [13] concluded that the prototypical texture was less frequently observed, due to the variable character of the matrices, and that there are only two necessary petrographic conditions: the presence of fine-grained dolomite crystals and a pore structure allowing alkali solution to penetrate the rock. Milanesi et al. [14] report on a very expansive, porous, fine-grained (0.01–0.03 mm) dolomite with 10–20 % clay minerals but with no calcite.

The best technique for identifying reactive texture is by use of thin sections with a petrographic microscope.

Alternatively, the reactive texture can be identified in opaque section in concrete or rock samples, using a highly polished specimen and manipulation of the illumination to get the

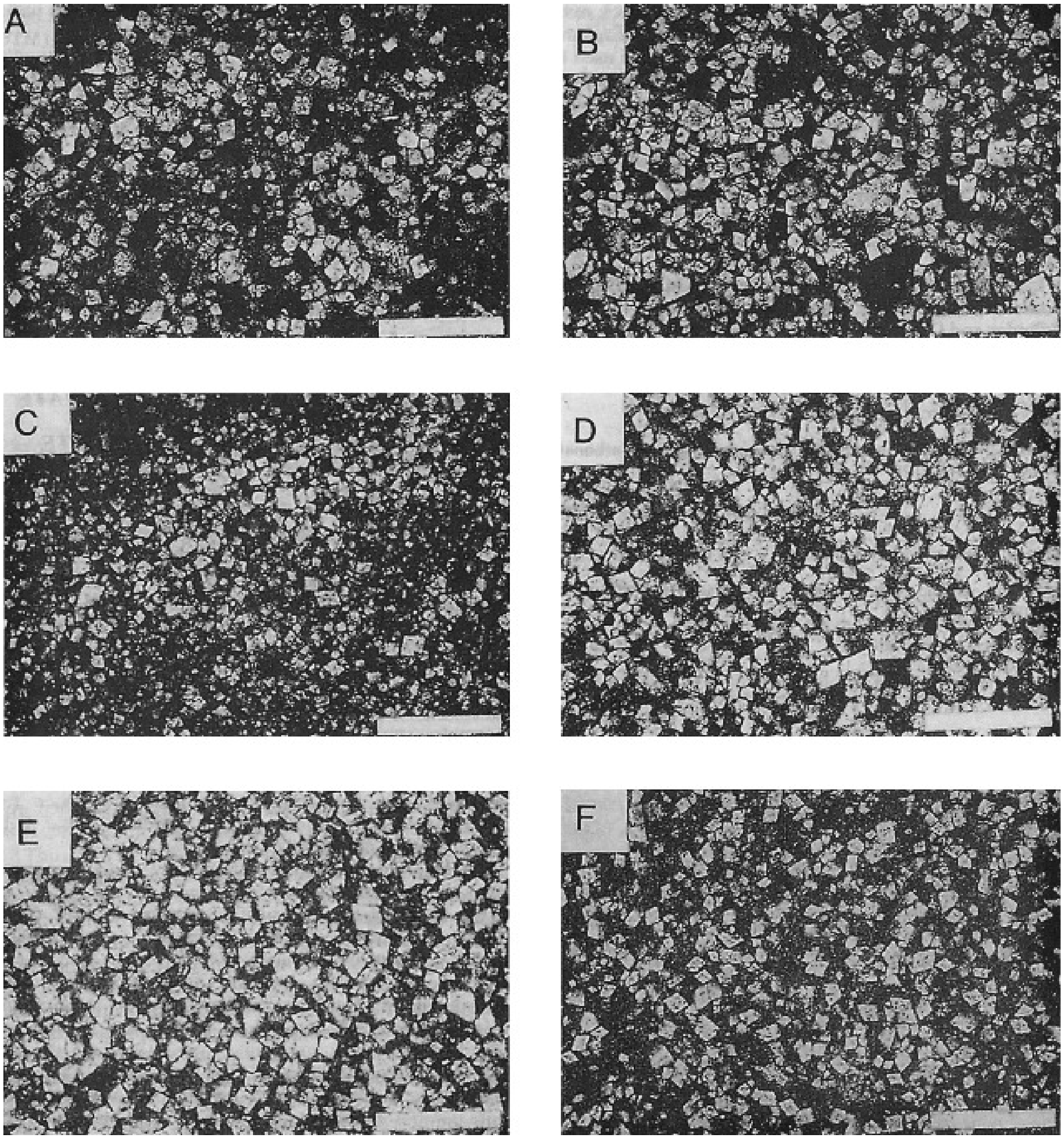

Fig. 3—Photomicrographs of thin sections of alkali-carbonate reactive limestones from quarries in eastern Canada. Scale bar is 0.25 mm. Fields A, B, and C are from different quarries. Fields D, E, and F are from the same quarry, D and F from the same bed [48].

TABLE 1—Composition of Early and Late Expansive Carbonate Rocks (Adapted From Ref 1)

	Acid Insoluble Residue, %	Dolomite % of Total Carbonate
Kingston, Ontario, early expanders [3,17,18]	5 to 15	about 50
Iowa, Illinois, and Indiana early expanders [19,20]	10 to 20	40 to 60
Virginia early expanders [21,22]	13 to 29	46 to 73
Gull River, Ontario, late expanders [18]	21 to 49	75 to 87
Virginia late expander [23]	33	>90

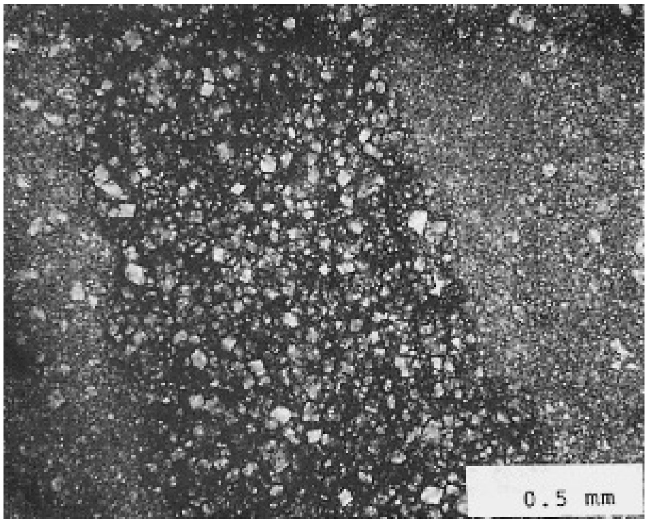

Fig. 4—Vein of reactive carbonate texture in limestone displaying alternating bands of reactive and nonreactive texture. Northern Virginia, field is approximately 2 mm across. The same type of structure can also exist on a larger scale, from bed to bed, or within a bed, with the alternating bands being several inches thick.

proper contrast and reflectance. A light etching of the specimen surface with dilute HCl can be very helpful.

Carbonate rocks displaying reactive texture can comprise a substantial or a small portion of a bed in a limestone quarry. Or, the reactive texture may occur as veins within a limestone bed that does not otherwise display the reactive texture (Fig. 4).

The occurrence of the reactive carbonate rock texture is a reliable diagnostic guide. Rocks which exhibit the texture should be tested further to determine their expansive potential with the contemplated job cement.

Alkali-carbonate reactive coarse aggregate particles in concrete may or may not display reaction rims. The formation of reaction rims on ACR rock particles is incidental to their propensity for reaction and generation of crack damage. However, the presence of reaction rims calls for closer inspection of those coarse aggregate particles, since often, but not always, their presence is indicative of alkali-carbonate reaction.

Broadly speaking, it appears to be the case geologically that the conditions for formation of alkali-expansive carbonate rocks are more restricted than for other limestones, and they are not abundant in the volume of limestone that exists on earth. They may occur in carbonate rocks of any age but, thus far, have been found most often in carbonate rocks of Ordovician age.

It is clear from the microtexture of reactive carbonate rocks, with the commonly sharp-edged rhombs, and occurrence as veins of reactive texture, that that texture is of replacement, or possibly diagenetic, origin rather than one resulting from primary deposition. As such, it is reasonable to infer that, since the production of the reactive texture requires special conditions subsequent to deposition and not obtaining as components of the ordinary depositional regime for limestones, there will be less of it in the total limestone section–as field experience thus far seems to indicate.

To put reactive carbonate rocks in perspective with reactive siliceous rocks, reactive siliceous rocks are far more abundant in the overall category of siliceous aggregates (crushed stone together with sand and gravel) that might be used in concrete than are reactive carbonate rocks in the category of all carbonate rocks. And, generally, but not entirely, the carbonate aggregates that participate in alkali-carbonate reaction are crushed stones from limestone quarries. This is in contrast to alkali-silica reactivity where reactive siliceous components can derive from both gravel and crushed stone sources. That is, except near their bedrock source, and under conditions of rapid erosion and deposition, limestone-bearing gravels, considering earth's gravels as a whole, are not as abundant as siliceous gravels because limestones are less able than siliceous rocks to survive the weathering and transportation necessary to put them in gravel deposits as hard and sound particles suitable for use as concrete aggregate. Consequently, alkali-carbonate reactive particles in gravel deposits, because such particles are limestones, are less likely to be found in gravels than alkali-silica reactive particles. However, gravel deposits containing reactive carbonate rock exist and are reported to have been used in concrete in England in the M 50 Motorway[3] and have been found and used in concrete, causing damage, in Ontario, Canada, near their bedrock source.[4]

Chemical and Mineralogical Composition

Typical compositions of early and late expanding alkali-reactive expansive carbonate rocks are shown in Table 1, with their geographic location and publication reference indicated.

In an extended study of the composition of reactive and nonreactive Ontario limestones and their expansion in the Rock Cylinder and Concrete Prism Expansion Tests, Rogers [8] found that the compositions of potentially expansive rocks fell within a fairly distinct field when the CaO:MgO ratio is plotted against either the Al_2O_3 content or the insoluble residue. The plot versus insoluble residue is shown in Fig. 5. Those empirical relationships are the basis for the Canadian Standard Chemical Method screening test for ACR, CSA A23.2-26A.

Factors Affecting Expansion of Concrete with Reactive Carbonate Rock and Factors Affecting Expansion of Reactive Carbonate Rock by Itself

A suite of carbonate rocks that have reactive texture and that exhibit varying degrees of (unrestrained) expansion in the rock cylinder test (ASTM C 586) may exhibit a very different expansion ranking when tested "in restraint" in the rock cylinder test [15,16] or when restrained by incorporation in concrete [23]. Based on Hilton's work [15] the degree to which reactive carbonate rocks will cause expansion in concrete is related, on the one hand, to the restraint imposed by the concrete, and, on the other, to the volume of dolomite in the rock, up to a point, and the internal textural restraint, or rigidity, of the carbonate rock in question.

A reactive carbonate rock with low textural restraint can be highly expansive unrestrained, but when restrained, either experimentally or within concrete, it is more compressible and less expansive.

In Hilton's experimental work [15,16], the most expansive rock, unrestrained in ASTM C 586, expanded 7.8 % at 14 weeks and, restrained in a steel frame in 1N sodium hydroxide

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[3] W. J. French, personal communication.

[4] C. A. Rogers, personal communication.

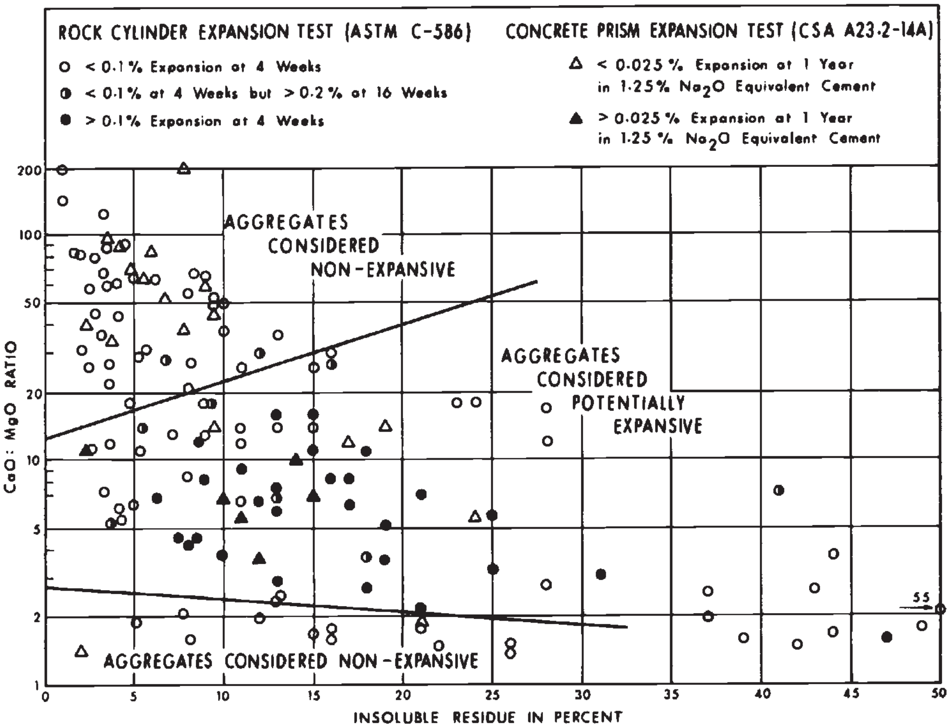

Fig. 5—CaO:MgO ratio versus insoluble residue for a suite of alkali-carbonate reactive and nonreactive carbonate rocks [8].

(NaOH), expanded 0.275 % at 14 weeks. The rock has 58.6 % by volume dolomite, 23.5 % calcite, and 5.63 % porosity. Petrographically, the rock has a high volume of dolomite rhombs tightly packed but with abundant voids between the masses of tightly packed rhombs (Fig. 6).

The least expansive sample did not expand at all after eight weeks in 1N NaOH. It is a typical "crystalline" dolomite composed of a mosaic of equant dolomite grains in a tight virtually nonporous structure (Fig. 7). It had 94 % dolomite by volume, <6 % calcite, and 0.18 % porosity. It did begin to expand after 40 weeks of continuous soaking in the NaOH solution.

Qian et al. [24] obtained similar results using autoclaved concrete microbars (150°C in 10 % KOH solution) incorporating mosaic-textured, almost pure, "crystalline" dolomites. They concluded that these rocks do undergo dedolomitization but that early expansion is very low compared to rocks with the typical ACR texture. However, with sufficiently long reaction time significant expansion could develop.

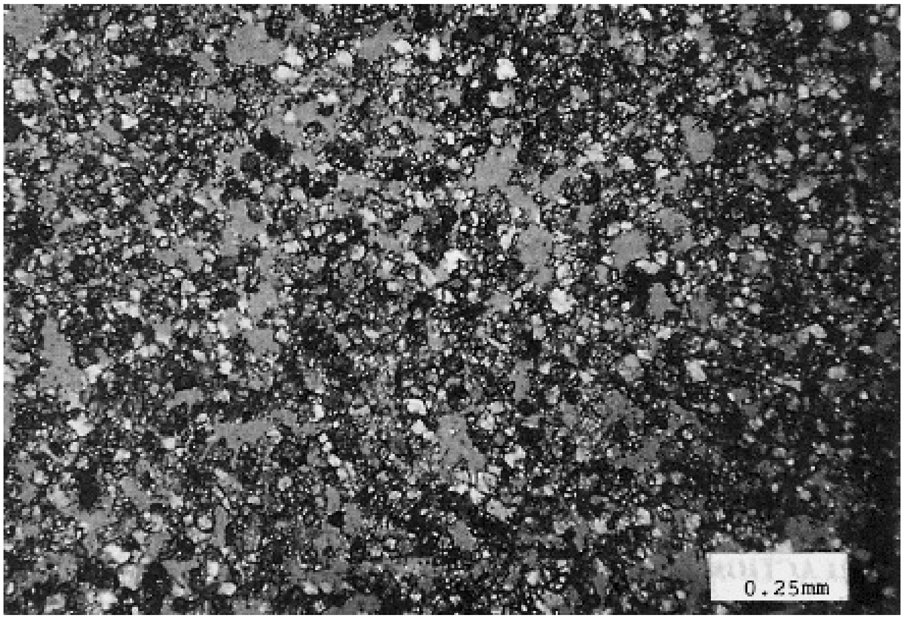

Fig. 6—Expansive rock from Missouri, 7.8 % in 14 weeks in ASTM C 586. High content of dolomite rhombs in porous matrix.

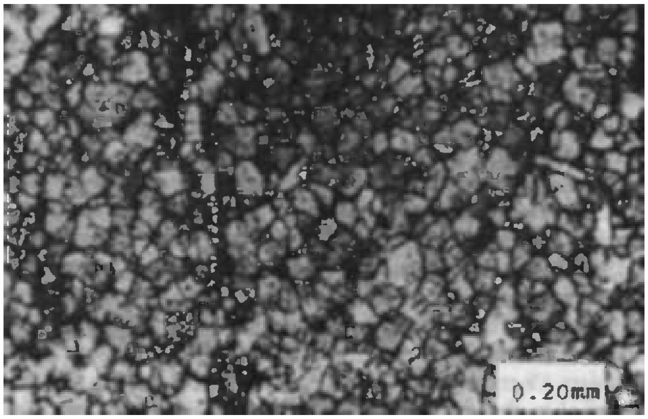

Fig. 7—Nonexpansive rock, in ASTM C 586, from Virginia. Structural framework of equant dolomite grains with no discrete "floating" rhombs and no interstitial matrix.

Further in Hilton's work, a rock, representative of several, with the typical alkali-carbonate reactive texture (Fig. 8) and similar to Fig. 3 (Fields A, B, and F), expanded unrestrained 1.4 % at 14 weeks, and restrained 0.31 %. It has 32.4 % by volume dolomite, 36.7 % calcite, and 1.25 % porosity.

The rock with the lesser value of unrestrained expansion expanded more under restraint than the rock with five times greater unrestrained expansion, and almost two times greater dolomite volume.

Thus, carbonate rocks with different or equivalent chemical/mineralogical composition could have vastly different expansion characteristics in concrete if their structural fabrics differ to such an extent that their elastic properties are quite different.

The petrological factors leading to the optimization of the lithology for producing expansion in concrete are: (1) clay content, or insoluble residue content, in the range of 5–25 %; (2) calcite to dolomite ratio of approximately 1:1; (3) increase in dolomite volume up to the point at which interlocking texture becomes a restraining factor; and (4) small size of the discrete "floating" dolomite rhombs.

The expansion of concrete containing alkali-reactive carbonate rocks is increased by: (1) increasing coarse aggregate size; (2) moisture availability; (3) higher temperature; (4) high alkali content of the concrete and high pH of the liquid phase in cement pores; (5) high proportion of reactive stone in the coarse aggregate; and (6) lower concrete strength [23,25].

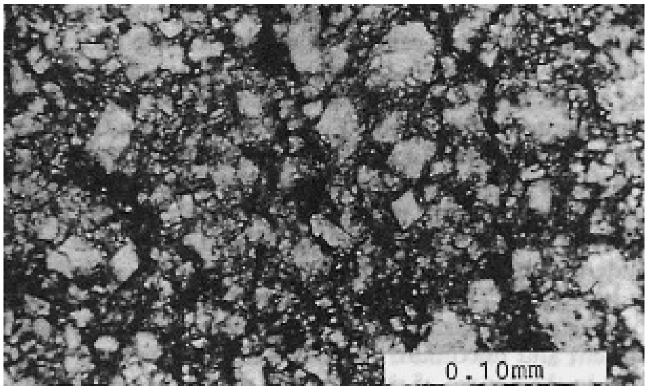

Fig. 8—Expansive rock from northwest Virginia with typical reactive texture. Expansion in ASTM C 586 was 1.4 % at 14 weeks.

Mechanism of Reaction and Expansion

Following the first recognition of the alkali-carbonate reaction by Swenson [3], work by Hadley [4], and later investigators, showed that the main chemical reaction that occurred in the rock was that the dolomite [$CaMg(CO_3)_2$] decomposed, or dedolomitized, to calcite ($CaCO_3$) and brucite $Mg(OH)_2$, as represented by the following reaction in which *M* represents an alkali element, such as potassium, sodium, or lithium:

$$CaMg(CO_3)_2 + 2MOH \rightarrow Mg(OH)_2 + CaCO_3 + M_2CO_3$$

A further reaction that occurs is that the alkali carbonate produced in the initial reaction may then react with the $Ca(OH)_2$, produced as a normal product of cement hydration, to regenerate the alkali hydroxide. For example:

$$Na_2CO_3 + Ca(OH)_2 \rightarrow 2\ NaOH + CaCO_3$$

Although the starting and end products of the reaction have been well documented and establish the overall bulk chemical and mineralogical changes that take place, the dedolomitization reaction, as such, does not translate into a mechanical explanation for the expansion, because the volume of the reaction products is smaller than that of the reactants [26]. The explanation was sought in behavior of the reactive rock and alkali system with respect to indirect mechanisms and secondary reaction products that had been identified in various investigations. That is, the primary dedolomitization reaction was considered to be the prerequisite that enabled other processes to work to produce the expansion.

An idea common to some proposed mechanisms is that, in one way or another, water absorption and incorporation is important in producing the expansion.

Sherwood and Newlon [27] proposed that expansion could be related to the formation of the hydrated, relatively high-volume, low-density, secondary mineral products gaylussite and buetschliite. Feldman and Sereda [28] found trace amounts, in reacted rock, of a material that expands when it absorbs water. Walker [29] and Buck [30] found minerals of the hydrotalcite-sjogrenite group, which may possibly help promote water absorption and consequent expansion.

Swenson and Gillott [31] proposed that dedolomitization exposes "active" clay minerals that are present as inclusions, in the rocks that they studied, within the dolomite euhedra. Exchange sites on the clay surface adsorb sodium ions, and water uptake by the "new" clay results in swelling. Thus, it is the "active" clay released during dedolomitization that contributes to the expansion.

Clay inclusions within the dolomite rhombs of expansive rocks have not been universally reported. They have not been observed, for example, in expansive rocks from Virginia.

Tang et al. [32] conclude that the expansion due to ACR is caused by ions and water molecules migrating into restricted spaces created by the growth and rearrangement of the products of dedolomitization, particularly brucite. Therefore, the expansion results directly from dedolomitization and not from water uptake of clay mineral inclusions within dolomite rhombs. Using STEM and EDAX to observe the locations of the reaction products in expanded samples of Kingston, Ontario, reacted rock, they found that the brucite produced by the reaction occurred in a 2-μm-thick ring of parallel-oriented 25Å crystals, with space between them, surrounding the euhedral dolomite rhombs of the typical reactive texture. The brucite crystals were, in turn, surrounded by the calcite and clay particles of the matrix. The calcite of the dedolomitized rhomb remained within the interior of the dedolomitized crystal.

Later work, discussed below, using compacts of cement and dolomite, or magnesite, to simulate ACR rocks, is considered by Tong, Tang, and others to confirm this expansion mechanism [26,33,34].

The role of the clay minerals in the fabric of the rock may be to provide mechanical pathways to the dolomite rhombs by disrupting the structural framework of the rock, thus weakening the carbonate matrix and, by that means, helping to promote expansion. Thus, the presence of clays is not essential to the chemistry of the dedolomitization reaction but is an enabling factor, mechanically promoting expansion when dedolomitization occurs.

Katayama [6], based largely on a review of the literature, suggests that there is a stronger correlation between expansion and silica (insoluble residue) content of carbonate rocks having ACR texture than with their dolomite content, and that deleterious expansion is confined to impure carbonate rocks that contain more than 5–10 % insoluble residue. Therefore, alkali-carbonate reaction should be reviewed in light of the alkali-silica reactivity of cryptocrystalline quartz hidden in the microstructure of the matrix enclosing the dolomite rhombs.

For several Chinese aggregates, Tong and Tang [35] found that both alkali-reactive dolomite and alkali-reactive silica, as microcrystalline quartz, are present in the same aggregate particle. Both dedolomitization and silica gel formation occurred in rapidly expansive concrete microbars cured at either 60°C or 150°C in 10 % KOH solution. XRD and SEM/EDAX results confirmed the presence of the products of both reactions.

Investigators have noted that brucite may not always be detectable as evidence that dedolomitization has occurred in concrete that has expanded [13,35–37]. The reasons for this are discussed subsequently.

Experiments aimed at establishing and reconfirming the chemistry and products of the dedolomitization reaction, and the expansion associated with the reaction, have been conducted using single crystals of mineral dolomite, dolomite mineral powders, cylinders of dolomitic rocks, and mortar compacts of powdered, high-purity dolostone, and of magnesite, with portland cement.

Randonjic et al. [37] confirmed the basic dedolomitization reaction by examining single crystals of dolomite, before and after immersion in NaOH solution, using environmental scanning electron microscopy (ESEM), atomic force microscopy (AFM), and the surface analytical technique X-ray photoelectron spectroscopy (XPS). After immersion in NaOH they observed the chief reaction products of calcite and brucite and that the first reaction product to form was brucite, followed by calcite. Their results suggest that the brucite would be susceptible to further chemical change by reacting with species from the alkaline environment such as Ca^{2+} and Si^{4+}. And, that this should also be true for magnesium and iron rich phases formed during dedolomitization. The authors propose that since the chemical changes occurring during dedolomitization involve surface reactions, they would be expected to involve expansive volume changes. Processes which may contribute to expansion include the formation of brucite, the uptake of water, and the formation of iron-rich phases from ferroan dolomite.

Tong [13] states that it is feasible for brucite to react with silica in the alkaline environment, to form gel-like materials, and that it is therefore sensible that it would not be detected. He notes also that brucite can be difficult to detect due to the small amount produced and its poor crystallinity.

Prince and Perami [38] showed that dedolomitization is possible in the absence of alkali by experiments on pastes composed of dolomite mineral powder, soda, calcium chloride, and lime, in various proportions. The pastes were cured 24 h at 40°C in a saturated atmosphere and the reaction products monitored by XRD. In paste composed of only dolomite and lime, only brucite and calcite were formed–the dedolomitization occurred in the absence of any alkali. With introduction of soda, dedolomitization was enhanced. The general conclusion was that the particular reaction products depend on the ratio of calcium to sodium ions. High values of the ratio promote the formation of brucite and calcite, while lower values promote brucite and pirssonite [$CaNa_2\,(CO_3)\cdot 2H_2O$].

Milanesi et al. [14] related expansion of concrete prisms and rock cylinders to dedolomitization in both types of samples and in powdered samples of the same three rocks immersed in strong alkaline solutions, using XRD, SEM, and optical microscopy to identify the reaction products. The most expansive rock in both the rock cylinder test (ASTM C 586 but using 19×75 mm cylinders) and the concrete prism test, CSA A23.2-14A, was a porous, fine-grained, dolomite with a minor amount of quartz, feldspar, and rhyolite clasts, but with no evident calcite. Previous work showed the rock to be definitely not alkali-silica reactive. Examination of the cylinder before and after immersion in 1 N NaOH solution showed strong dedolomitization had occurred, producing calcite and brucite. A calcitic dolomite and a dolomitic limestone, both of coarser grain size and lower porosity than the expansive rock, shrank in the rock cylinder test and expanded only about 6 to 7 % of the 5-year prism expansion (0.205 %) of the most expansive sample. Those samples showed no evidence of dedolomitization in the rock cylinder test. Powders of the three rocks completely dedolomitized in 1 N NaOH after four days, forming calcite and brucite with no detectable dolomite. The authors attribute the differences in expansion of the three dolomitic rocks to differences in their microstructural characteristics. They conclude that the equations proposed by Hadley [4] are adequate to explain dedolomitization in concrete under normal service conditions.

Tang et al. [34] and Tong and Tang [26,33] using mortar compacts of portland cement with high-purity, powdered dolostone and high-purity, powdered magnesite immersed in alkaline solutions at elevated temperatures performed experiments aimed at addressing the following questions: (1) whether the volume-reducing alkali-carbonate reaction (the volume of the products being less than the volume of the reactants) can cause expansion; (2) whether absorption of water by clay exposed by dedolomitization causes the expansion; (3) whether the ACR is actually an alkali-silica reaction due to the reactivity of cryptocrystalline quartz hidden in the matrix enclosing the dolomite rhombs; (4) whether the presence of the matrix of calcite, clay, and fine-grained quartz enclosing the dolomite rhombs in the prototypical ACR texture is a necessary condition for expansion by dedolomitization.

The use of compacted cylinders of dolomite or magnesite particles in a matrix of cement paste simulates the spatial relation of dolomite rhombs enclosed in a fine-grained matrix, as is the case in natural alkali-reactive dolomitic limestones, and eliminates the effects of cryptocrystalline quartz in the matrix, clay in, or between, the dolomite rhombs, and calcite, clay, and quartz comprising the matrix, focusing instead on the reaction of the dolomite or magnesite particles in the cement paste.

Magnesite was used as a cross reference material [26,33] because it also reacts to produce brucite in an expansive reaction in which the brucite is the unique solid product, simplifying the expansion mechanism, and it is also a solid volume reducing process.

Compacts were also made with limestone powder [34], high purity alumina, and cement paste by itself, as control samples. In addition to the expansions of the compacts, the expansions of rock prisms and concrete microbars of the same materials were measured. Reaction products were determined by X-ray diffraction.

The control samples produced negligible expansions while the magnesite and dolomite compacts exhibited considerable expansions which correlated well with the dolomite consumption/calcite production and the magnesite consumption/brucite production in the respective compacts. The authors conclude that the results provide a powerful confirmation that the expansion was directly caused by the reactions occurring at the interfaces of the dolomite or magnesite particles with the enclosing cement paste matrix. And that, although it is a solid volume reducing reaction, an expansive force is generated by the growth and rearrangement of the reaction products, together with the migration of alkali and hydroxyl ions, and water, into the confined space of the particle/matrix interface.

It is reasonable that the compacts are a good, functional, proxy, artificial aggregate for the ACR limestone in which dolomite crystals are enclosed in a fine-grained matrix. The conclusion may then be drawn from the experiments that neither calcite, silica, or clay in the matrix, nor clay in, or between, the dolomite rhombs is related to the expansion-causing process. Rather, the expansion is directly proportional to the dedolomitization of the dolomite enclosed in the matrix and must, therefore, be related to the accumulation and positioning of the reaction products in relation to the surface of the dolomite rhombs.

Evaluating the Potential for Alkali Carbonate Reactivity

Field Service Record

Field performance information on concrete incorporating the aggregate under consideration can furnish direct answers to the questions that the laboratory evaluation tests attempt to answer indirectly. That is, tests on the rock, in or out of concrete, are intended to provide information on which, in essence, to base a prediction on what will happen if the rock is used in concrete construction. However, if “good” information is available from the field, then, logically, that is best, and indirect information to predict what can be observed directly is second best.

Provided certain essential information, noted later, is available and certain exposure conditions are fulfilled, an evaluation of the potential for aggregate reactivity based on the investigation of concrete structures in the field can be the most unequivocal and economic way of accomplishing the evaluation.

When investigating and sampling a concrete structure (ASTM C 823) known to contain aggregate from the source in question, for example, by prior petrographic examination, the alkali content of the cement and the cement content per cubic yard must be known. In general, if the cement used in the concrete was low alkali cement, less than 0.6 % total alkalies as per ASTM Specification for Portland Cement (C 150), then the concrete cannot provide information on the propensity for reaction of the aggregate with high alkali cement. However, in view of the facts, (1) the most reactive carbonate rocks can produce deleterious expansion with cement of 0.4 % total alkalies [23], and (2) the more important measure is the alkali content per unit volume of concrete, not the alkali content of the cement per se. Then even if the cement used was less than 0.6 % total alkalies, if the cement content of the concrete was high, the alkali level per unit volume may equal or exceed that of concrete in which the cement content was low but the alkali content of the cement was high. A threshold guideline, based on pounds of sodium oxide (Na_2O) equivalent per cubic yard, must be established for judging whether the aggregate should be considered to have been used in a “high” or “low” alkali concrete.

If the structure shows no distress and it has been established that the alkali content of the concrete would be sufficient to cause reaction if the aggregate is potentially reactive, then the effects of aggregate size, proportion of reactive aggregate, and moisture availability to the concrete must also be considered singly and in combination before concluding that the aggregate is non-deleteriously expansive.

Larger-sized reactive aggregate causes greater expansion than smaller-sized reactive aggregate. A reactive aggregate that has not caused distress with “high” alkali cement in small top size may cause distress when used in the larger size with the same cement.

The greater the proportion of reactive aggregate of the total aggregate, the greater the expansion. A reactive aggregate that has not caused distress as a small portion of the coarse aggregate in a structure with “high” alkali level may cause distress when used in larger proportion with the same cement.

Potentially reactive aggregates will not cause deleterious amounts of expansion if the concrete is protected and in a relatively dry condition. Where exposure to moisture also includes exposure to sodium-chloride-containing deicing salts, which are known to exacerbate ACR, the conclusion that the aggregate is non-deleteriously expansive is strengthened.

Concrete Prism Expansion Test

The surest measure of the potential for deleterious expansion of a carbonate aggregate in concrete is the measurement of the length change (for percent expansion) of concrete prisms, as by ASTM C 1105 or Canadian Standard CSA A23.2-14A.

ASTM C 1105 is intended primarily for evaluating a particular cement-aggregate combination prior to use in concrete, although, as noted, it can be used for investigational or research purposes (Paragraphs 4.5 and 6.3).

When it is used for its primary purpose the alkali content of the cement will be that of the job cement, and the storage conditions will be at 23°C and > 95 % relative humidity. If the alkali content is raised by using a higher alkali cement and/or adding NaOH to the mixing water, and the storage temperature is raised, the test is accelerated and is then generally similar to CSA A 23.2–14A and ASTM C 1293, in which the storage temperature is 38°C (100.4°F) and the cement content is stipulated at 420 kg/m^3 (708 lb/yd^3), with a total alkali content, using cement of 0.9 % alkali augmented by NaOH in the mixing water, of 1.25 % by mass of cement, or, 5.25/kg/m^3 (8.85 lb/yd^3).

C 1105 is closer to field conditions than is CSA A 23.2-14A which is intended to accelerate the expansion by maximizing the conditions for the detection of slowly reactive rocks and for

TABLE 2—Concrete Prism Test, Expansion Limits, and Alkali Content

Test	Na_2O Equivalent Alkali Content	Expansion Limit at Indicated Time, Percent		
		3 months	6 months	1 year
ASTM C 1105	job cement	0.015	0.025	0.030
CSA A23.2-14A	1.25%		...	0.040

facilitating comparisons among aggregates by using exactly the same aggregate grading and alkali level.

In C 1105 the grading of the job-proposed aggregate can be used if it is not coarser than 19 mm (3/4 in). But, C 1105 specifies that if the job-proposed aggregate has material larger than 19 mm (3/4 in.), it shall not be used, based on the assumption that the plus 19-mm material does not differ in composition, lithology, and presumed expansive characteristics from the minus 19-mm material. If, based on information from petrographic examination or rock cylinder tests (ASTM C 586) or both, the plus 19-mm material differs from the minus 19-mm material in those respects, then instructions are given for testing the coarser fraction. In CSA A23.2-14A, the coarse aggregate is separated on 20-, 14-, 10-, and 5-mm sieves, oversize and undersize material discarded, and equal masses of the three sizes recombined.

Expansion limits for ASTM C 1105 have not been adopted in ASTM C 33 but are suggested in the Appendix to ASTM C 1105 and are, therefore, nonmandatory. As such, they place greater responsibility on the specifier of the concrete to understand the factors promoting expansion due to alkali-carbonate reactivity versus the service environment of the concrete.

The expansion limit for the CSA prism test is specified and tabulated in Table 2 with the suggested ASTM C 1105 limits.

It may be noted with respect to, for example, the three-month C 1105 limit, that fifteen-thousandths of one percent is not a deleterious degree of expansion. But the logic of the test is such that, based on correlation with longer term tests and field performance, a rock which produces 0.015 % expansion at three months will produce deleterious expansion in field concrete at a later age.

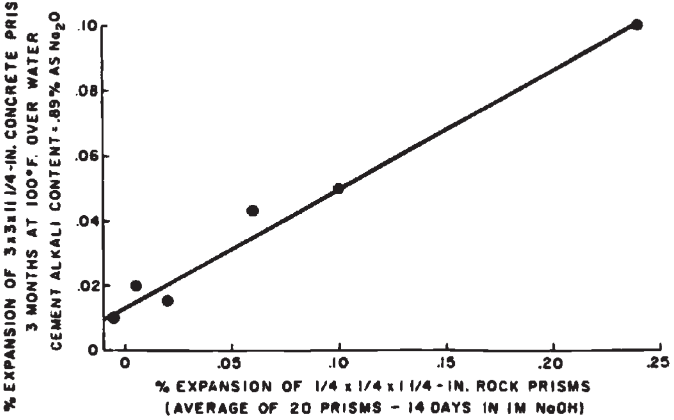

Fig. 9—Expansion of small rock prisms in alkaline solution correlates well with expansion of concretes using these rocks as aggregate [4].

The expansion limit of 0.040 % used in the CSA standard is the approximate level at which cracking and distress are first observed on the prisms, and it relates well to observed field performance [39].

Rock Cylinder Expansion Test

The expansion of small (approximately 1.4 in. in length by 0.4 in. in diameter, 35.5 by 10 mm) cylinders of carbonate rock that are continuously immersed in 1 N NaOH solution has been used as a direct indication of expansive alkali-carbonate reactivity of the rock since the method, originally using square cross section specimens, was first described and used by Hadley in 1961 [20]. And since its earliest use, the correlation between rock cylinder (or rock prism) expansion and concrete prism expansion has been substantiated (Figs. 9 and 10). However, note in Fig. 10 the spread of the six rock prism expansion values corresponding to the concrete prism expansion value (average of three) and, in Fig. 9, that the rock prism expansion values plotted are the average of 20 specimens.

Thus, despite good correlations between the average values of rock prism and concrete prism expansions, due to the variability inherent in the rock, the differences that can exist between restrained (in concrete) and unrestrained (in ASTM C 586) behavior, and the other factors governing expansion in concrete (for example, w/c, total water soluble alkalies, paste to aggregate ratio, etc.), the situation is such that ASTM C 586 is

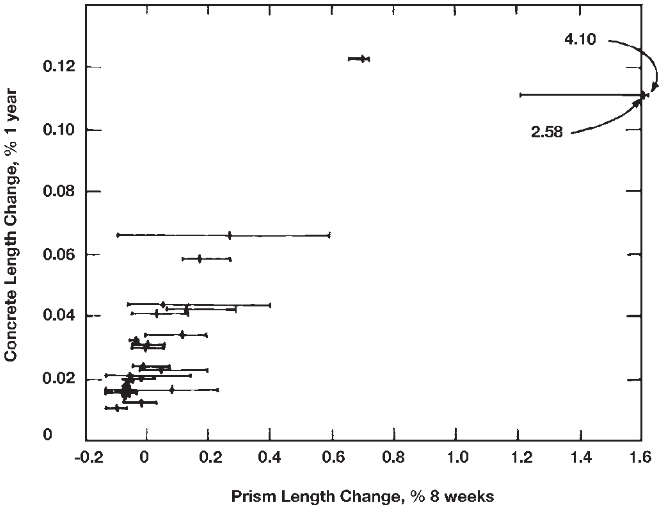

Fig. 10—Relationship between one-year length change of (ASTM C 157) concrete prisms (average of three) with high alkali cement (0.95 %) and length change of (ASTM C 586) rock prisms at eight weeks. Average and range of six rock prism length changes are shown for each sample [23].

a relatively rapid indicator of potential expansive reactivity, and an effective tool for screening aggregate sources, but it does not predict in-concrete expansion of the rock and is not used as an acceptance test.

One test specimen per sample of rock is the minimum required by the method. It should be taken normal to the bedding if bedding is discernible. If bedding is not discernible, then three mutually perpendicular specimens are made. In general, even if bedding is discernible, it is desirable to prepare three mutually perpendicular specimens; one perpendicular to the bedding and two at right angles parallel to the bedding. Of three such specimens, the one exhibiting the greatest expansion is the one on which to base the test result.

An expansion of 0.10 % at 28 days is considered to indicate the presence of material with a potential for deleterious expansion when used in concrete. For example, as reported by Rogers [8], most quarry faces sampled in Ontario, Canada, that gave average rock cylinder expansions of greater than 0.1 % at four weeks also caused excessive expansion and cracking when used in concrete.

Although 0.1 % expansion at 28 days is the implied limit given in ASTM C 586, an alternate limit of 0.20 % at 16 weeks is also considered applicable by several researchers in view of the fact that some rocks contract before they begin to expand [23,40]. For example, a delayed expander tested by Newlon, et al. [23] contracted 0.1 % at four weeks but expanded to 0.25 % at 16 weeks, eventually peaking at 0.5 % at 44 weeks. A typical alkali carbonate reactive rock producing on the order of 0.1 % concrete prism expansion at one year with high alkali cement might expand on the order of 0.5 % at four weeks in ASTM C 586.

In the suite of sample test results reported by Rogers [8] there were considerably fewer samples in the category "$<$ 0.1 % at four weeks but $>$ 0.2 % at 16 weeks" than there were in the category "$>$ 0.1 % at four weeks."

In view of the requirements of specimen selection and the occasional irregular, or late expanding, behavior of samples in the rock cylinder test, it is strongly recommended that all aspects of the testing program and interpretation of the results be guided by petrographic information.

Petrographic Examination

Petrographic examination of dolomitic carbonate rocks (ASTM C 295) proposed for use as coarse aggregate in concrete will detect the presence of the characteristic reactive lithologic texture. And, the characteristic reactive texture, as described in ASTM C 294 and discussed elsewhere in this chapter, when it occurs in a dolomitic limestone, will undergo reaction or dedolomitization in an alkaline environment. Whether, when tested, the reaction will produce expansion of a rock cylinder (ASTM C 586) or concrete beam (ASTM C 1105), insofar as the rock is concerned, depends on the perfection of the type lithology, as regards ASTM C 586, and as regards ASTM C 1105, on the amount and distribution of the reactive lithology within the stratigraphic section proposed for use as concrete aggregate.

Following the initial use of petrographic examination for detecting the reactive carbonate rock texture (see Quarry Sampling) in order to identify candidate samples for further testing by ASTM C 586 or C 1105, petrographic examination is valuable during successive steps of the evaluation to guide the understanding of the behavior of samples in, and the interpretation of results from, the rock cylinder test (ASTM C 586) and the concrete prism test (ASTM C 1105).

Petrographic examination speaks directly to the concerns of Paragraphs 10.1, 10.2, and 10.3, "Interpretation of Results" of ASTM C 586. And, although not specifically called for in ASTM C 1105, petrographic examination of the concrete prisms at the conclusion of the test can furnish information helpful for interpretation of results at, or near, the test limits. The Canadian Standard notes the usefulness of petrographic examination at this stage.

Determination of Potential Alkali Carbonate Reactivity of Carbonate Rocks by Chemical Composition

The Ontario, Canada, Ministry of Transportation and Communications has developed a quick chemical screening test for the determination of potential alkali-carbonate reactivity of carbonate rocks by chemical composition–Canadian standard CSA A 23.2-26A.

The test is based on work by Rogers [8] and uses relationships based on a correlation between expansion of concrete prisms and rock cylinders, and performance of aggregates in field structures, and the CaO:MgO ratio of the limestone versus the alumina (Al_2O_3) content or the insoluble residue content.

For example, in Fig. 5, using insoluble residue, regions are delineated in which the potentially expansive carbonate rocks cluster. Rocks with compositions in these regions should be considered to be potentially deleterious until their nondeleterious level of expansion is demonstrated by service record or by a concrete prism expansion test.

Other Methods and Approaches

Concrete Microbars

Xu et al. [41] developed an accelerated method for determining potential alkali-carbonate reactivity using concrete microbars 20×20×80 mm and 40×40×160 mm stored in 1 N NaOH solution at 80°C for four weeks. The aggregate particle size used is 5–10 mm, with cement (boosted by addition of KOH) of 1.5 % Na_2O eq. at a w/c of 0.30 and aggregate to cement ratio of 1:1. An expansion of 0.1 % at four weeks is judged to be appropriate for recognizing ACR limestones, based on comparing the behavior of aggregates in the test and in field concrete.

Grattan-Bellew et al. [42] propose a modification of the microbar test, using 40×40×160 or 286 mm bars, for alkali-carbonate reactive aggregates [41] which is also applicable to alkali-silica reactive aggregates. They state also that siliceous ASR limestones can be differentiated from dolomitic ACR limestones by running two tests, one with 100 % portland cement, the other replacing a portion of the cement with 30 % fly ash. If the limestone is alkali-silica reactive the expansion will be significantly reduced, whereas the fly ash will have little effect on the expansion of the ACR limestone aggregate.

Selection of Samples for Testing and for Concrete Aggregate

Quarry Sampling

Sampling of the stratigraphic section in limestone quarries for detecting the presence of alkali-carbonate reactive rock by petrographic examination should recognize that the characteristic reactive texture and composition are only subtly and indirectly related to the primary factors of sedimentation that produce bedding. The reactive texture is of secondary, or replacement, origin and may or may not conform faithfully to lithologic or stratigraphic units, or individual beds as observable in the field. The amount of reactive texture and the expansivity of that texture may vary laterally and vertically within beds or within a sequence of beds comprising a definable, or mappable, stratigraphic unit, to the same extent that the variations in rock prism (ASTM C 586) expansions within a given bed, or lithology, may be as large or larger than those between lithologies [23].

Sampling for the reactive lithology should therefore be guided by petrographic examination using as many thin or polished sections as necessary to delineate the reactive rock and to select samples for further tests. And, because of the variability that can exist, any thin or polished section that shows even a minor area of reactive texture should, at the first sampling stage (Stage I), require the field sampling unit that it represents to be considered suspect [23].

The objective of the first (Stage I) sampling is to establish the presence or absence of reactive rock within a quarry. It is understood that the establishment of 100 % absolute absence requires examination of every bed. Lithology and structure should be considered in a way that samples are located with due regard to their relationship to the quarry production horizons, and in sufficient detail to permit a calculated “weighting” for comparison with the maximum “allowable” reactive content of the total aggregate that can be established by subsequent testing using ASTM C 1105. In Stage I sampling, a minimum of five and an average of approximately ten samples should be taken in a quarry. Each lithology representing more than 10 % of the production should be sampled. Lithologies representing less than 10 % can be combined and sampled at random locations in the combined thickness. Where the face exhibits relatively thin beds of varying lithology, it should be sampled at a minimum of ten systematically located stations [25].

If Stage I sampling establishes the presence of reactive rock within a quarry, then the objective of the Stage II sampling will be to identify, as precisely as the limitations of time and budget will permit, the precise beds and their amounts of reactive material. Perhaps from 50 to 100 samples may be required. Previous experience suggests that the results of the more extensive and finer-scale sampling can end up delineating a lesser volume of reactive material in the quarry face than may have been indicated from the results of the Stage I sampling [25].

Using Coarse Aggregate from a Quarry with Potentially Expansive Rock

If potentially expansive rock is present in a quarry, the following courses of action should be considered.

If the stratigraphy of the producing section is favorable, avoid the reactive rock by selective quarrying. If the reactive rock cannot be totally avoided, utilize selective quarrying such that the aggregate from the produced section, when tested in concrete by ASTM C 1105, does not produce a deletrious expansion. If the “natural” dilution approach is infeasible, dilute the produced aggregate with nonreactive stone such that the resulting mixture does not produce a potentially deleterious level of concrete expansion by ASTM C 1105.

Research and experience with a group of Virginia quarries indicated that, for those sources, an acceptable level of dilution was reached when the reactive rock did not exceed 20 % of the coarse aggregate, 20 % of the fine aggregate, or 15 % of the total aggregate, if both fractions contained reactive material [25].

In the concrete, use the smallest aggregate size that is economically feasible and the lowest alkali cement, bearing in mind that cement with total Na_2O equivalent alkali at the 0.4 % level can produce alkali-carbonate reaction, and that alkali content per unit volume of the concrete is the important measure. Therefore, the proposed job concrete mixture should be tested by ASTM C 1105.

Unlike the alkali-silica reaction, the alkali carbonate reaction is not very well controlled by the use of fly ash or ground granulated blast furnace slag (GGBFS). Tang et al. [34] found that even with cement of 0.43 alkalies, expansion of concrete at one year, cured at 40°C and 100 % RH, was effectively reduced only at 70 % replacement of cement by fly ash, or 90 % by slag. Rogers and Hooton [43] in a five year comparison study of laboratory and field concrete with and without GGBFS concluded that up to 50 % replacement of high alkali cement (1.04 %) with GGBFS did not reduce expansion sufficiently so that the Kingston, ON, reactive aggregate could be safely used. Thomas and Innis [44], using the same aggregate in concrete prism tests (CSA A23.2-14A) and up to 65 % replacement of high (1.25 %) alkali and 50 % replacement of low (0.50 %) alkali cement, found that the slag did not effectively control expansion.

Also, unlike the ASR, the use of lithium compounds does not mitigate the expansion caused by ACR. Results of Wang and Gillott [45], using the Kingston, ON, reactive aggregate, showed that concrete prisms (CSA A23.2-14A) with either LiCL or LiOH, at nine months, had the same expansion as the control prism, and the prism with $LiNO_3$ had significantly more expansion than the control. Stark [46] recommends that the preferred compound for ASR mitigation is LiOH due to its greater solubility than either lithium fluoride or lithium carbonate. Tang et al. [47], using the Kingston, ON, aggregate in concrete bars treated at 150°C in water and in 10 % LiOH and KOH, obtained the greatest expansion for the KOH, intermediate for the LiOH, and lowest for the water (control) sample. The authors note that LiOH produced dedolomitization and brucite formation, similar to the reactions with Na and K hydroxides, and that the use of LiOH could be an effective means to differentiate alkali-carbonate from alkali-silica reaction.

In the use of the concrete in constructions that will contain diluted, or otherwise minimized, amounts of reactive aggregate and alkali, restrict availability of moisture to the concrete by barriers or by design of the construction if possible.

If no mitigating approach is feasible, prohibit the use of the reactive rock in portland-cement concrete.

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Thermal Properties of Aggregates

D. Stephen Lane[1]

Preface

THE CHAPTER ON THERMAL PROPERTIES OF concrete aggregates was authored by H. K. Cook in the first three editions of *Significance of Tests and Properties of Concrete and Concrete Aggregates*, and by the current author in the fourth. Because little has changed in this subject area since the publication of *ASTM STP 169C*, this chapter is substantially the same as in the previous edition, with only minor revisions and additions to the text and reference list.

Introduction

Aggregates typically compose 65–70 % or more of the volume of concrete. Because the thermal properties of composite materials such as concrete are dependent on the thermal properties of their constituents, the aggregates have a great influence on the thermal properties of concrete. The degree to which thermal properties of concrete aggregates are of concern to the user depends upon the nature of the concrete structure and the exposure to which it is subjected. Knowledge of thermal properties are important in the design of massive structures, such as dams, where thermal and volume stability of the composite are important; in concretes exposed to extreme temperatures or temperature cycles, where the relative properties of the individual constituents may be important; and in lightweight concrete structures where insulating value of the composite is a primary factor.

The thermal properties of aggregates that are of general concern are the coefficient of thermal expansion, conductivity, diffusivity, and specific heat. Of these, the thermal expansion of aggregates is by far the most important because of the importance of the thermal expansion of concrete in the design of all concrete structures. The conductivity, diffusivity, and specific heat of aggregates are usually of concern only in special instances. The most obvious of these is massive structures where the dissipation of heat generated by cement hydration is important to control thermal cracking. Other instances include insulating concrete and the durability of concrete exposed to cyclical temperature changes. The design engineer should carefully evaluate the circumstances of a particular concrete application to estimate the importance that thermal properties of aggregates will have on concrete performance.

The attention that has been given to the thermal properties of concrete aggregates is not as great as that given to other properties of aggregates. This is probably because in many instances the effects do not appear to be as important as the effects of other properties of aggregates and, consequently, the significance of thermal properties is not as apparent. The need for a better understanding of the effects of the thermal characteristics of aggregates, particularly the coefficient of thermal expansion on the durability of concrete, has been expressed by Allen [1], Woolf [2], Scholer [3], and others.

The discussion in this paper is primarily confined to the significance of tests and thermal properties of the aggregates. While this necessarily requires discussion of the thermal properties of the concrete as affected by the aggregates, the significance of tests and thermal properties of concrete is discussed in Chapter 24 of this publication.

General Concepts of Thermal Properties

Thermal properties of rocks are related directly to the thermal properties of the constituent minerals in proportion to their relative abundance. A reasonably good approximation of the value of thermal expansion, specific heat, conductivity, or diffusivity of a rock can be calculated if the mode (mineral content, percent by volume) of the rock is known using the following formula [4]

$$Pt(rock) = S(n_1^{*}Pt_1 + n_2^{*}Pt_2 + n_3^{*}Pt_3...) \qquad (1)$$

where

n_1 = percent by volume, Mineral 1;
Pt_1 = thermal property value of Mineral 1; and
S = solidity (1 − decimal porosity).

The mode of an aggregate can be determined petrographically using ASTM Guide for Petrographic Examination of Aggregates for Concrete (C 295). The value thus calculated for a given aggregate will not be as accurate as could be measured. However, given the considerable care and skill necessary to accurately measure these properties, a calculated value may prove acceptable for many general applications. Robertson [4] has compiled a large volume of data on the thermal properties of rocks and minerals.

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TABLE 1—Average Linear Coefficient of Thermal Expansion of Some Common Rocks and Minerals [5]

Mineral	$\times 10^{-6}/°C$
Quartz	11.5 to 12
Orthoclase, microcline	6.5 to 7.5
Pyroxenes, amphiboles	6.5 to 7.5
Olivine	6 to 9
Albite	5 to 6
Calcite	4.5 to 5
Oligoclase, andesine	3 to 4
Labradorite, Bytownite	3 to 4
Anorthite	2.5 to 3
Rock	$\times 10^{-6}/°C$
Quartzite, silica shale, chert	11.0 to 12.5
Sandstones	10.5 to 12.0
Quartz sands and pebbles	10.0 to 12.5
Argillaceous shales	9.5 to 11.0
Dolomite, magnesite	7.0 to 10.0
Granites and gneisses	6.5 to 8.5
Syenite, andesite, diorite, phonolite, gabbro, diabase, basalt	5.5 to 8.0
Marbles	4.0 to 7.0
Dense, crystalline, porous limestones	3.5 to 6.0

Coefficient of Thermal Expansion

Numerical Values for Thermal Coefficients of Expansion

The coefficient of thermal expansion of aggregates is directly dependent on the coefficients of the constituent minerals and thus on the mineral composition of the rock. Zoldners [5] has reported coefficients of thermal expansion for common rock-forming minerals and some common rocks that are presented in Table 1. Although values for the thermal coefficients of expansion of aggregates from specific locations are contained in many of the references listed and are available from many other sources, Refs 4–16 list more values than most of the others.

As will be noted from the table, quartz has the highest coefficient of thermal expansion of the common minerals, while calcite and high calcium feldspars exhibit the lowest. Thus, the coefficients of rocks vary to a large degree in direct proportion to their quartz content.

While most minerals will exhibit some degree of anisotropy by expanding more in a direction parallel to one crystallographic axis than another, this is generally not of significance except when the differences are great. The most notable example is calcite, which can have a linear thermal coefficient of expansion as great as $25.8 \times 10^{-6}/^{\circ}C$ parallel to its *C*-axis and as low as $-4.7 \times 10^{-6}/^{\circ}C$ perpendicular to this direction. Potassium feldspars are another group of minerals exhibiting rather extreme anisotropy

For this reason, the cubical expansion of rocks and minerals is not always related directly to the linear expansion, and this possibility should be kept in mind when investigating the thermal properties of aggregates.

The paper by Meyers [17] is concerned completely with thermal coefficient of expansion of portland cement rather than aggregates. It is included because a discussion of the thermal properties of concrete aggregates would not be complete without some information on the thermal properties of the cements with which they are used. The coefficient of thermal expansion of hydrated cement pastes may range from $10.8 \times 10^{-6}/^{\circ}C$ to $16.2 \times 10^{-6}/^{\circ}C$ and mortars from about $7.9 \times 10^{-6}/^{\circ}C$ to $12.6 \times 10^{-6}/^{\circ}C$. Mitchell [7] has found that at early ages or at certain critical saturations the linear thermal coefficient of expansion of cement pastes may be somewhat higher than those reported earlier. He reports values as high as $22.3 \times 10^{-6}/^{\circ}C$ for some samples of neat cement specimens.

An average value for the linear thermal coefficient of expansion of concrete may be taken as $9.9 \times 10^{-6}/^{\circ}C$, but the range may be from about $5.8 \times 10^{-6}/^{\circ}C$ to $14.0 \times 10^{-6}/^{\circ}C$, depending upon the type and quantities of the aggregates, the mixture proportions, and other factors.

Effect of Thermal Expansion

The significance of the coefficient of thermal expansion of aggregates with respect to its effect on concrete is two-fold. The primary, or at least better understood, effect is that on the volume change of concrete. Aggregates with high coefficients of thermal expansion will produce concretes of low thermal volume stability and vice versa, all other things equal. The thermal expansion of concrete should be accounted for in the design of the structure and is further discussed in Chapter 24 on Thermal Properties in this publication. The other effect is the development of internal stresses as a result of large differences between the coefficients of thermal expansion of the various components of concrete subjected to temperature extremes or thermal cycling and the impact this may have on concrete durability.

There seems to be fairly general agreement that the thermal expansion of the aggregate has an effect on the durability of concrete, particularly under severe exposure conditions or under rapid temperature changes. There is less agreement on whether more durable concrete will be produced with an aggregate of high thermal coefficient of expansion similar to that of the cement paste or with an aggregate of low thermal coefficient.

In one of the earliest papers on the subject, Pearson [18] attributes a case of rapid concrete deterioration to the use of an aggregate of low thermal coefficient. The aggregate, a dolomitic marble with a coefficient of thermal expansion of $3.6 \times 10^{-6}/^{\circ}C$ was reported to be very resistant to freezing and thawing deterioration. The concrete was severely cracked after the first winter, having been subjected to very low temperatures and to severe frost action. Given the aggregate's freezing and thawing durability and because the deterioration occurred much more rapidly than would have been expected from freezing and thawing action alone, other causes were suspected. Pearson speculated that tensile stresses developed when the matrix contracted three to four times that of the coarse aggregate at extremely low winter temperatures. These stresses were sufficient to cause fine cracking of the concrete providing pathways for water into the concrete and subsequent freezing and thawing damage. Pearson conducted a laboratory investigation [19] with concretes containing aggregates of both low and high thermal coefficients. The concretes were subjected to 100 thermal cycles between -28.9 and $21.3^{\circ}C$ before being subjected to freezing-and-thawing tests. Concretes containing the aggregates of low thermal coefficient failed much more rapidly in the freezing-and-thawing test than the concretes containing aggregates of high thermal coefficient.

Callan [20,21] statistically analyzed 78 combinations of aggregate in concrete with respect to durability in freezing and thawing and differences in thermal expansion between the coarse aggregate and the mortar. He concludes that large differences between the coefficients of expansion of coarse aggregate and mortar may considerably reduce the durability of concrete from that predicted by the results of the usual acceptance tests. He suggests that differences between these coefficients exceeding $5.4 \times 10^{-6}/^{\circ}C$ should be considered large enough to warrant caution in the selection of materials for highly durable concrete exposed to temperature extremes. Swenson and Chaly [22] include some discussion on the effect of thermal coefficient of expansion of the aggregates on concrete durability and caution against the possible deleterious effects if large differences are observed. Smith [23] presents calculations to indicate the potential magnitude of physical incompatibility of the matrix and aggregate in concrete based on differences in thermal expansion. Kennedy and Mather [24], in attempting to correlate laboratory-accelerated freezing and thawing with natural weathering at Treat Island, Maine, state among other conclusions that, while there appears to be a correlation between the resistance of concrete to freezing and thawing and differences in thermal expansion between the coarse aggregate and the mortar, the correlation is probably usually of lesser importance than other characteristics of the concrete.

Walker et al. [25], on the other hand, report the results of heating and cooling concrete specimens over the temperature range of $4.4-60^{\circ}C$ at various rates. They found that "changes in temperature were destructive to the concrete with sudden changes in temperature being much more severe than slower ones; and concretes having higher coefficients of expansion were less resistant to temperature changes than concretes with lower coefficients." It also was determined that the thermal coefficients of expansion of concrete and mortar containing different aggregates varied approximately in proportion to the thermal coefficient and quantity of aggregate in the mixture.

The findings of Walker et al. [25] stand in contrast to those of Pearson [18,19], Callan [20,21], and the others [22,24] that aggregates with low coefficients of thermal expansion compared to the cement paste may reduce concrete durability. One explanation for the difference in finding is that Walker's study did not subject specimens to temperatures below the freezing point. Pearson, however, used temperatures as low as $-28.9^{\circ}C$. Koenitzeer [26] subjected several concretes and the aggregates used in them over temperature ranges of -12.8 to $26.7^{\circ}C$, 29.4 to $88^{\circ}C$, and -12.8 to $88^{\circ}C$, in both moist and dry conditions. One of Koenitzer's conclusions was that the elastic and thermal expansion properties for any one material vary with the conditions of test, the greatest variation being caused by freezing.

However, the U.S. Bureau of Reclamation produced concrete at Grand Coulee Dam using a predominately basalt aggregate with a thermal expansion of about $7.2 \times 10^{-6}/^{\circ}C$ that showed an extremely high resistance to freezing and thawing. The Bureau of Reclamation also found [8] in the Kansas-Nebraska area that replacement of part of the aggregate with limestone, which had a thermal expansion averaging $4.5 \times 10^{-6}/^{\circ}C$, greatly increased the durability of the concrete in which it was used. Although the addition of limestone was for the purpose of inhibiting alkali-aggregate reaction and the improvement in durability probably was primarily because of the inhibition of this reaction; the resulting lower thermal expansion apparently introduced no adverse effects.

More recently, Venecanin [27,28] has renewed interest in the durability of concrete produced with aggregates of low thermal coefficient of expansion. He has derived equations [27] to evaluate the potential stresses developed between binders and aggregates of different thermal coefficients and attributed abnormal cracking in several bridges to a limestone aggregate with low thermal coefficient and a high degree of anisotropy [28]. The bridges are in areas subject to extreme subfreezing winter temperatures. Other researchers [29,30] following Venecanin's lead have concluded that limestone aggregates that exhibit extreme anisotropic thermal characteristics can adversely affect concrete durability even in environments not subject to sub-freezing temperatures.

The preceding discussion has dealt with the effects of thermal expansion of aggregates over temperature ranges that can occur under natural exposure conditions. Zoldners [5] indicates that thermally stable aggregates such as fine-grained rocks of anorthositic composition are best suited for concretes that will be subjected to extreme elevated temperatures. For such applications, aggregates containing quartz should be avoided since at a temperature of $572.7^{\circ}C$ quartz changes state and suddenly expands 0.85 %, usually producing a disruptive effect at the surface of concrete in which it is used. Endell [31] has reported the results of experiments to determine the structural and expansion changes of concrete aggregates with temperatures up to $1200^{\circ}C$. These are considered to be highly specialized conditions and are not discussed further here, but are discussed further in Chapter 25, Resistance to Fire and High Temperature, in this publication.

Methods of Determining Thermal Expansion of Aggregates

Several ingenious methods have been developed for determining the thermal coefficient of expansion of coarse aggregate. Most of the test methods are based on the measurement of linear expansion over a temperature range. This range is usually $55^{\circ}C$ or more because the change in unit length per degree is extremely small, and the multiplication of the change over a substantial temperature range greatly increases the facility and precision of the determination. However, the size of a representative specimen that can be obtained from a coarse aggregate rapidly approaches a practical maximum. Except in the case of a crushed aggregate of sufficient uniformity to permit obtaining a larger specimen that will be representative of the sand sizes, this means of multiplying the change in unit length is not available for determining the linear expansion of fine aggregate.

The method described by Willis and DeReus [32] allows measurements to be made over a considerable temperature range with the use of an optical lever. The optical lever has also been used to obtain additional multiplication of the length change. The specimens used by Willis and DeReus were 25.4-mm-diameter cores, 50 mm long, drilled from the aggregate specimens to be tested and placed in a controlled-temperature oil bath with a range of $2.78 \pm 1.7^{\circ}C$ to $60 \pm 2.8^{\circ}C$. The vertical movement of the specimen as the temperature was varied was measured by reading the image reflected by the mirror of the optical lever, having a 25.4-mm lever arm, on a vertical scale placed 6.1 m from the mirror, by means of a precise level. It is reported that consideration of the possible errors involved in the measurements indicates that the calculated coefficients are probably accurate to $\pm 3.6 \times 10^{-7}/^{\circ}C$.

The use of an interferometer is described by Merritt [33] and modified by Saunders [34]. Detailed descriptions of the apparatus, the preparation of specimens, and the test procedure with specific application to concrete aggregates is given by Johnson and Parsons [10]. ASTM Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry (E 289) covers the use of Michelson interferometers for which the typical specimen will possess either blunt or rounded ends.

The Corps of Engineers [21,35] describes a method for measuring the thermal coefficient of expansion of coarse aggregate particles in which an SR-4 strain gage is bonded to a prepared piece of aggregate and readings are taken over a temperature range of 1.7 to 57.2°C. This method requires that the piece of aggregate be sliced in three mutually perpendicular directions, two of these directions to lie in the major structural plane of the rock, if such a plane can be located. The strain gages are then mounted so as to measure strain in each of the three directions. The purpose of this requirement is to determine if anisotropy or preferred crystal orientation exist. Recently, Venecanin [28] has reported on a similar, but more elaborate setup, where strain gages are mounted to obtain measurements parallel to the edges and in both diagonal directions on each of the six faces of a cube of rock.

Mitchell [7] describes a method that employs specimens from 25.4−76.2 mm in size coated with wax and held in fulcrum-type extensometer frames. Measurements are made with electromagnetic strain gages with electronic indicators while the specimen is immersed in a circulating ethylene glycol solution held at the desired temperature.

Because of the size and usually heterogeneous nature of fine aggregate, none of the preceding methods are readily adaptable to the determination of the coefficient of expansion of this type of material. The usual approach has been to determine the linear expansion of mortar bars containing the fine aggregate. Of course, the results obtained include the effects of the length change contributed by the cement. Verbeck and Hass [36] developed a dilatometer method for determining the thermal coefficient of expansion that is particularly adaptable for use with fine aggregate. The method determines the cubical thermal coefficient of expansion from which the linear expansion may be calculated. Description of modern dilatometer equipment can be found in ASTM Test Method for Linear Thermal Expansion of Solid Materials With a Vitreous Silica Dilatometer (E 228).

Thermal Conductivity, Thermal Diffusivity, and Specific Heat

Thermal conductivity, thermal diffusivity, and specific heat are largely interrelated, and all three normally are determined only for concrete as used in massive structures. The Bureau of Reclamation [37] indicates the application of these data in connection with computing concrete placement temperatures and designing cooling systems, and in other thermal calculations aimed at reducing thermal volume change and thus cracking in large dams. The same type of measurements and calculations would apply equally to other massive structures. Thermal conductivity is also of importance in lightweight concrete for insulating purposes. It has been indicated by some [38–40] that thermal diffusivity may have an important effect on concrete durability.

Thermal conductivity, measured as the rate of heat flow through a body of unit thickness and unit area with a unit temperature difference between two surfaces, normally is expressed in calculations as watts per metre kelvin (W/mK).

Thermal diffusivity is defined as the thermal conductivity divided by the specific heat and density and is a physical property of the material that determines the time rate of change of temperature of any point within a body. Its units are square metres per second (m^2/s).

Specific heat is the amount of heat required to raise the temperature of a unit mass of material one unit of temperature. Its units are joules per kilogram kelvin (J/kgK).

Thermal Conductivity

The thermal conductivity of aggregates varies in large measure with their mineral content. Quartz is one of the more important minerals to consider in evaluating the conductivity of an aggregate as it exhibits a relatively high conductivity. The feldspars on the other hand are also important because of their abundance and relatively low conductivity. The porosity of an aggregate also plays a significant role in determining its conductivity; which is impacted by the aggregate moisture content since air is a very good insulator and water a very good conductor of heat. Tyner [41] reports that in a 1:5 mixture of Florida limerock concrete, an increase of moisture from 0–5 % increases the conductivity by 23 %. Although one of the more useful properties of lightweight concrete is its low thermal conductivity, Davis and Kelly [42] state that “the presence of a small amount of moisture in the interior of a lightweight concrete greatly increases its thermal conductivity; hence, under conditions of continuous or intermittent exposure to moisture, if a high degree of insulation is desired, an aggregate (and concrete) of relatively low absorption should be used.” Kluge et al. [43] and Price and Condon [44] also have found pronounced reductions in the thermal conductivity of concrete containing lightweight aggregate, but they indicate that the reduction seems to be influenced more by the reduction in the density of the concrete than by the characteristics of the aggregate. Thermal conductivity values for some common rock types reported by Clark [45] are presented in Table 2.

For most heavy-weight aggregates used in radiation shielding, thermal conductivities will be high. The exception is barite, a sulfate mineral with a thermal conductivity somewhat lower than normal concrete aggregate [46].

TABLE 2—Thermal Conductivity Values for Some Common Rocks (Data From Clark [45])

	Thermal Conductivity (W/mK)	
Rock	**Mean**	**Range of Values**
Quartzite	6.7	5.9 to 7.4
Dolomite	4.6	4.0 to 5.0
Gneiss, parallel to foliation	3.5	2.6 to 4.4
Granite, quartz monzonite	3.3	2.8 to 3.6
Granite	3.2	2.6 to 3.8
Granodiorite (California)	3.2	2.9 to 3.5
Diabase	3.0	2.6 to 3.4
Amphibolite	2.9	2.6 to 3.8
Granodiorite (Nevada)	2.8	2.6 to 2.9
Gneiss, perpendicular to foliation	2.6	2.0 to 3.6
Limestone	2.6	2.0 to 3.0

Thermal Diffusivity

As mentioned earlier, thermal diffusivity is directly proportional to conductivity. The thermal diffusivity of a normal-weight aggregate is thus primarily dependent on its quartz content. Investigations have indicated that the normal diffusivity of the aggregate may have an influence on the durability of the concrete in which it is used. Thomson [38] states that for a given body with specified boundary conditions the thermal stresses depend on certain physical properties of the materials. In a homogenous body, such physical properties as thermal conductivity, specific heat, and the density of the material influence the temperature distribution and the thermal stresses during the transient period only in a certain combination known as the thermal diffusivity. If in a mixture such as concrete the thermal diffusivities and conductivities are the same for each material, the body can be thought of as being thermally homogenous. Since a difference in diffusivities would result in different rates of diffusion of heat through the aggregate and paste, it is believed that such a combination would result in higher thermal stresses than those existing in homogenous bodies. Nothstine [39] and Weiner [40] have reported the results of their approach to the problem. Weiner's work was instigated by the failure of a gravel concrete, exposed to natural freezing and thawing accompanied by thermal shock and characterized by bond failure and internal expansion. He attributes the failure to the relatively high thermal coefficient of expansion of the concrete, which is responsible for surface stress, and to the diffusivity of the gravel that, being higher than the mortar, responds more quickly to temperature changes, resulting in differential volume change. In an investigation of four limestones, Fox and Dolch [47] found a large change in the thermal diffusivity with a relatively small degree of saturation. The increases in diffusivity ranged from 20–59 % for saturations of less than 5 %. The authors of the references cited essentially agreed that the thermal diffusivity of the aggregates apparently has an effect on the durability of concrete, but that further work is needed to determine the significance of the effect and to find a practical means for using this knowledge to improve concrete durability. Calculated diffusivities for some rocks are presented in Table 3.

Specific Heat of Aggregates

Specific heat is of considerable importance in connection with the calculations involved in the control of placement temperatures and the limiting of thermal volume change of mass concrete. The specific heat of the aggregate contributes materially to the specific heat of the concrete [37]. The specific heat of rocks can be calculated from the mineral composition of the rock using Eq 1. The effect of temperature on specific heat is considerable and should be considered. Table 4 presents values of specific heat for some common minerals and rocks.

TABLE 3—Calculated Diffusivities of Some Common Rocks (Data From Robertson [4])

Rock	Diffusivity, 10^{-6}m^2/s
Basalt	0.9
Marble	1.0
Limestone	1.1
Gabbro	1.2
Sandstone	1.3
Rhyolite	1.6
Peridotite	1.7
Quartzite	2.6
Dolomite	2.6

TABLE 4—Specific Heat of Some Common Minerals and Rocks (Data From Robertson [4])

		10^3J/kgK
Mineral	**27°C**	**127°C**
Fayalite	0.64	0.72
Quartz	0.75	0.88
Pyroxene	0.75	0.92
Microcline, Sanidine	0.77	0.85
Anorthite	0.77	0.88
Phlogopite	0.77	0.92
Forsterite	0.77	0.95
Albite	0.80	0.92
Muscovite	0.81	0.95
Calcite	0.86	0.95
Dolomite	0.86	0.98

Rock	0°C	50 to 65°C	200°C
Diabase	0.70	…	0.87
Granite	0.65	…	0.95
Granite	0.80	0.77	0.95
Granodiorite	0.70		0.95
Quartzite	0.70	0.77	0.97
Diorite	0.71	0.81	0.99
Gabbro	0.72	…	0.99
Slate	0.71	…	1.00
Marble	0.79	0.85	1.00
Limestone	…	0.83	…
Limestone	…	1.00	…
Granitic gneiss	0.74	0.79	1.01
Basalt	0.85		1.04

Methods of Test for Conductivity, Diffusivity, and Specific Heat

Methods are available for the direct determination of thermal conductivity, thermal diffusivity, and specific heat. However, as a matter of practical convenience, it is customary to determine diffusivity and specific heat and to calculate conductivity or to determine conductivity and specific heat and to calculate diffusivity. This is possible because the formula includes all three values, and knowing the values and density for any two, permits solving the equation for the unknown property. The formula is

$$k = hcp \qquad (2)$$

where

k = thermal conductivity in W/mK,
h = thermal diffusivity in m^2/s,
c = specific heat in J/kgK, and
p = density in kg/m^3.

Whether conductivity is determined directly and diffusivity is calculated, or vice versa, is largely a matter of the most convenient equipment setup available and the preference of the laboratory doing the work. Some of the test methods included in the references to this paper are based on determinations made on concrete specimens, but in most cases, they may be used with aggregates if properly modified with respect to specimen size and shape.

The Corps of Engineers [35], Thomson [38], and Fox and Dolch [47] describe methods for the direct determination of diffusivity of stone and concrete. All of the methods depend basically on obtaining time-temperature differential curves between the temperatures at the center and the surface of a specimen by starting at essentially equilibrium temperature, then changing the surface temperature, and plotting the time-temperature curve until equilibrium conditions are obtained at the new surface temperature.

Depending upon the degree of accuracy desired, refinements can be made by grinding the specimen to a sphere, for example, and by refining the instrumentation. The determination is by no means routine in nature and requires careful experimentation, precise equipment, and a capable operator.

Recently, Sass et al. [48] have described the use of a conventional needle probe to measure the thermal conductivity of machined, drilled, or unconsolidated rock material. This method offers a simplified and versatile means for measuring the conductivity of aggregates. Thermal conductivity can also be measured directly by ASTM Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus (C 177) or similar methods [41,49]. Tan et al. [50] present a laboratory procedure using this methodology to measure the thermal properties of unbound aggregates. The Bureau of Reclamation [51] has developed a method for the determination of the thermal conductivity of concrete by the use of a 203 × 406-mm (8 × 16-in.) hollow cylindrical concrete specimen. This procedure probably could be used for coarse aggregate, provided a specimen of the required size and shape could be fabricated from a large rock specimen.

The specific heat of aggregate usually is determined by a procedure known as the method of mixtures [35]. It is a calorimetric procedure where in the net heat required to raise the temperature of a specimen of known weight, a given amount is measured.

References 44–60 provide additional background information on the theory and mathematics of thermal tests. Other references may be found appended to many of the references cited here.

Conclusions

Test methods are available that are entirely adequate for the determination of the thermal properties of aggregates when used with proper attention to procedure, specimen size and shape, instrumentation, and technique.

There appears to be no doubt that the normal properties of aggregates, particularly thermal expansion, have an effect on the durability and other qualities of concrete. Investigations reported to date do not present a clear-cut picture of the effects that might be expected, and some aspects of the problem are controversial. The ultimate solution must be based on the performance of aggregates of known thermal properties in concrete, and, as is normal in this field of investigation, the major difficulty is to separate the effects of the thermal properties of the aggregates from the numerous other variables existing in the concrete. There is a real need for additional research work on the subject, both to resolve existing controversy and to improve concrete further as a construction material.

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PART V
Other Concrete Making Materials

37

Hydraulic Cements—Physical Properties

Leslie Struble[1]

Preface

PHYSICAL PROPERTIES OF HYDRAULIC CEMENTS were covered for the first time in the previous edition of *ASTM STP 169* in a chapter that covered the full range of physical properties–fineness, consistency, setting properties, strength, and durability–of hydraulic cements used in making concrete, significance of the standards, and how they might be improved. In preparation of the current chapter, the contents of that earlier chapter (excluding expansive cement) were extensively drawn upon. The current edition introduces relevant new technology and attempts to clarify certain issues.

Introduction

Hydraulic cements are manufactured products that find their principal uses in concrete and related construction materials. When cement and water are mixed, they undergo various chemical reactions that gradually change the mixture from a plastic (or fluid), which can be molded or cast, into a rigid solid, capable of bearing substantial compressive loads. Thus, cement and its reactions with water are largely responsible for most of the key aspects of concrete–its workability, set, strength, creep, shrinkage, and durability.

Hydraulic cements and their components have a wide range of physical properties. The objective of this chapter is to review those properties that are included in the standard specifications for hydraulic cements; these are found in Tables 3 and 4 of the ASTM Specification for Portland Cement (C 150), Tables 2 and 3 of the ASTM Specification for Blended Hydraulic Cements (C 595), and Table 1 of the ASTM Performance Specification for Hydraulic Cements (C 1157). The previous edition of this chapter included properties of expansive cements, but this edition does not. The types of cements in each specification are summarized in Table 1. The properties reviewed in this chapter are summarized in Table 2 and their test methods are listed in Table 3. The focus of this chapter is on these physical properties–their significance, limits in their use, and recommendations about ways the tests might be improved.

The two principal types of cement used in concrete are portland cement and hydraulic cement. Portland cement is based on crystalline calcium silicate phases whose hydration leads to hardening and strength gain in concrete. Hydraulic cement is a more general term for any cement that hardens and gains strength by chemical interaction with water. Thus portland cement is a hydraulic cement, but hydraulic cement may not be a portland cement. Blended cement, formed by blending portland cement with other reactive materials such as pozzolan or slag, is an example of a hydraulic cement. Slag by itself may be a hydraulic cement. These terms are also discussed in a separate chapter of this book (Hydraulic Cements–Chemical Properties).

There is a general trend in ASTM C01 (and elsewhere) to shift from prescriptive specifications, in which a certain composition is required, to performance specifications, in which performance in certain standard tests is required. Because portland cement is defined by its chemical composition, the portland cement specification, C 150, is prescriptive, although it includes several performance requirements as listed in Table 2 of this chapter. Most performance requirements in C 150 are optional, intended to replace certain prescriptive requirements at the discretion of the user and reflecting the general trend towards performance specifications. The core of C 150 remains prescriptive, and the limits on chemical composition of the various types of portland cements are discussed in the chapter of this book on chemical properties. The specification for blended cements, C 595, is similarly a mix of prescriptive and performance requirements, with greater emphasis on performance than prescriptive. The specification for hydraulic cements, C 1157, first published in 1992, is entirely based on performance tests, and a Task Group of the National Ready Mixed Concrete Association and the Portland Cement Association recently suggested revisions to increase its acceptance [1].

The physical tests covered in this chapter vary in their significance and use. Some are used to control quality during cement production (for example, fineness, set, and optimum sulfate content). Others are needed for determination of some other cement property (for example, density to calculate fineness). Most of the properties attempt to predict how cement influences the performance of concrete, and the significance of these properties lies in how well they predict the concrete behavior. Unfortunately, predicting this behavior is often very difficult because the relationships between cement properties and concrete behavior are often not understood, at least not quantitatively or in detail.

There are numerous references on cement and concrete, many of which were used in preparing this chapter. Particularly useful were two textbooks on concrete [2,3], two textbooks on cement chemistry [4,5], and two publications on cement, one from the American Concrete Institute [6] and the other from the Portland Cement Association [7].

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TABLE 1—Summary of Cement Classifications (Adopted with Permission from Ref 2)

Specification	General purpose	Moderate heat of hydration	High early strength	Low heat of hydration	Moderate sulfate resistance	High sulfate resistance	Resistance to alkali-silica reactivity
C 150	I	II	III	IV	II	V	Low alkali option
C 595	I IP I(PM) I(SM) S,P	IS(MH) IP(MH) I(PM)(MH) I(SM)(MH)		P(LH)	IS(MS) IP(MS) P(MS) I(PM)(MS) I(SM)(MS)		Low reactivity option
C 1157	GU	MH	HE	LH	MS	HS	Option R

Density

The density of hydraulic cement, though not in itself a particularly important property and not included in the cement specifications, is used to calculate fineness in C 204 and C 115, and is used in calculating concrete mix proportions by the absolute volume method [8]. Density is measured using the ASTM Test Method for Density of Hydraulic Cement (C 188), which utilizes a Le Chatelier flask filled with kerosene.

The standard test method using the Le Chatelier flask is accurate and inexpensive. However, it tends to be time-consuming and uses a large quantity of kerosene, which requires special disposal. The standard test allows use of alternate methods, and pycnometers are now available that provide rapid and reproducible density measurements and entail no special disposal.

Though the density of portland cement is usually assumed to be 3.15 Mg/m^3, it in fact ranges rather widely, from as low as 3.05 Mg/m^3 to as high as 3.25 Mg/m^3. Blended cements may vary even more substantially in density. This variation is enough to affect the cement volume used in calculating concrete mix proportions. It would be better to measure density of portland cement more frequently and to report the measured value. Unfortunately, the difficulties with the present standard method make it unlikely that routine measurement of density would be specified.

TABLE 2—Summary of Physical Properties Specified for Hydraulic Cements

Physical Property	Portland Cement (C 150)	Blended Hydraulic Cement (C 595)	Hydraulic Cement (C 1157)
Fineness	s	r,m	r
Density	...	...	...
Activity index	...	m	...
Water requirement	...	s	...
Set			
Time of set	s	s	s
False set	o	...	o
Heat of hydration	o	s	s
Volume change			
Drying shrinkage	...	s	...
Expansion	c	...	s
Autoclave expansion	s	s	s
Strength			
Minimum	s	s	s
Maximum	o	...	s
Durability			
Air Content	s	s	r
Alkali reactivity	...	o,m	o
Sulfate expansion	o	s	s

KEY:

s = specified for one or more types.
m = specified for constituent materials.
c = specified under certain conditions.
o = specified optionally.
r = report required but no limit specified.

TABLE 3—Summary of Test Methods for Physical Properties of Hydraulic Cements

Physical Property	ASTM Test Method
Fineness	
Air permeability	C 204
Turbidimeter	C 115
Sieving	C 430 (No. 325, wet)
Density	C 188
Activity index	C 595 Annex
Consistency	
Water requirement	included in C 109
Flow	C 1437
Normal consistency	C 187
Set	
Time of set	C 266 (Gillmore), C 191 (Vicat)
False set	C 451
Heat of hydration	C 186
Volume Change	
Drying shrinkage	C 157
Expansion	C 1038
Autoclave expansion	C 151
Strength	C 109
Optimum SO_3	C 563
Durability	
Air content	C 185
Alkali reactivity	C 227 (using Pyrex glass)
Sulfate expansion	C 452 (for portland cement) C 1012 (for blended cement)

Activity Index

Significance

The activity index is a useful property of pozzolan or slag when used in a blended cement or in concrete, so it is discussed here and also in the two chapters of this book dealing with pozzolan and slag in concrete (Supplemental Cementitious Materials, and Ground Slag). The activity index is simply the compressive strength of mortar using a blend relative to the compressive strength of mortar using plain portland cement.

Standards

Several versions of the activity test exist. For slag and pozzolan used in cement, the test is described in an annex to C 595. For mineral components in concrete, it is described in ASTM Method for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete (C 311) and in ASTM Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete or Mortars (C 989). All three tests compare the compressive strength of mortar containing the mineral component with the strength of mortar not containing the mineral component, and all three tests have the same objective, to test the acceptance of mineral admixtures, either for use in a blended cement or for use directly in concrete. These tests are related to ASTM Test Method for Compressive Strength of Hydraulic Cement Mortars (C 109).

There are several important differences between these four test procedures (C 595, C 109, C 311, and C 989). The activity index test in C 595 originally used the procedure in C 311, but a few years ago the procedure in C 311 was changed such that the specifications in C 595 were no longer appropriate, so the older C 311 procedure was added to C 595. This situation is an example of how a lack of communication between C01 and C09 can lead to illogical differences between standards. The test in C 595 uses a flow of 100–115, and C 109 and C 989 use a flow of 105–115, with no obvious rationale for the difference. The test in C 595 allows the use of any portland cement in the activity test (but encourages using the same brand that will be used in the blended cement), C 311 for pozzolan specifies that the portland cement have total alkalies in the range 0.5 to 0.8 %, and C 989 for slag specifies that the portland cement have total alkalies in the range 0.6 to 0.9 %. The effect of cement alkali on reaction of pozzolan or slag is well documented and use of a high-alkali portland cement is sensible in a general acceptance test for pozzolan or slag (if the portland cement used in a particular blend does not contain sufficient alkali to initiate reaction, the blended cement will not meet the strength specification), but the alkali levels in C 311 and C 989 should be brought into conformance. The test in C 595 uses a pozzolan replacement of about 35 %, whereas C 311 uses a pozzolan replacement of 20 % (by mass); and C 595 uses a slag replacement of about 70 %, whereas C 989 uses a slag replacement of 50 % (by mass). These replacement levels could surely be brought into conformance. The tests in C 595 and C 311 both use sand contents of 75 %, whereas C 109 and C 989 use sand contents of 73 %, again with no obvious rational for this minor difference. The test in C 595 uses a replacement level by volume, so the mass depends on the specific gravities, whereas C 989 and C 311 use replacement levels by mass. Replacement by volume may be sensible in certain applications, but is not necessary in an acceptance test. The test in C 595 stores cubes in a close-fitting sealed container, whereas C 989, C 311, and C 109 store cubes in saturated lime solution. The C 595 uses a storage temperature of 38°C, whereas C 989, C 311, and C 109 all use a storage temperature of 23°C, probably the most important difference, as the reaction rate of mineral admixtures may be particularly sensitive to temperature.

The specification for blended cement (C 595) requires that the activity index of slag or pozzolan for use in blended cement be a minimum of 75 % at 28 days. There is no specified minimum activity index of slag as a mineral component in concrete (C 989), but the activity index controls the grade of slag. The specification for fly ash and natural pozzolan in concrete (C 618) also requires that the activity index at 7 and 28 days be a minimum of 75 %. Thus the requirement for fly ash in concrete is somewhat more demanding (measured at a shorter time and lower temperature) than the requirement for fly ash in blended cement.

As shown in Table 2, C 595 specifies an activity index for pozzolan used in blended cement, but C 1157 does not. Activity index is a physical property but a prescriptive specification when applied to the pozzolan or slag. As a performance specification, C 1157 sets limits on strength of the hydraulic cement but no prescriptive limits (physical or chemical) on the materials used in the cement.

Recommendations

Some of the differences noted above may be important, but most are probably unnecessary, given that all three tests have the same general objective, so it is recommended that the tests be brought into conformance wherever possible. If the cement alkali level, the sealed storage conditions, and the higher storage temperature are important in C 595, then they are equally important in C 989 and C 311. A careful examination of all test

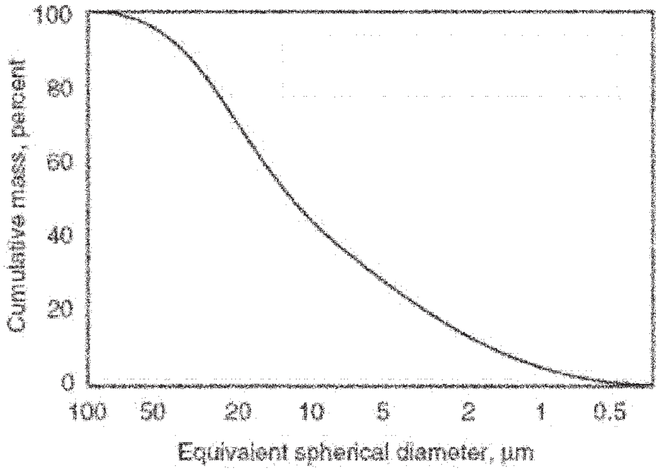

Fig. 1—Particle size distribution of a typical portland cement (reprinted with permission from Ref 7).

parameters concerning the acceptability of pozzolan and slag for use in concrete is warranted.

Fineness

Of the various physical properties that relate to cement quality, probably the most important and widely used is fineness. Because cement is a ground material, it has an inherently broad particle size distribution (Fig. 1). The median particle diameter of Type I or II portland cement is typically about 10 to 20 µm, but particles range in size from a few tenths of a micrometre to 50 µm or more. Type III portland cement is typically finer than Type I, and Types IV and V portland cements are often coarser. Blended cements are also broad in their particle size distribution, may cover a somewhat different size range, and are often bimodal.

Significance

Fineness is a very important physical property for cement. Hydration rate is a function of fineness, so setting time, strength, shrinkage, heat of hydration, and permeability are all influenced by fineness. Increasing the fineness substantially increases the rate of hydration, thereby shortening the setting time, speeding up the strength gain, and speeding up the permeability reduction that accompanies hydration. Increasing fineness is a common strategy for meeting the faster strength gain specified for a Type III portland cement. Finer cements adsorb chemical admixtures more rapidly, often requiring higher admixture dosages. Finer cements also generate higher temperatures during hydration. Effects of fineness on performance properties were discussed by Bentz et al. [9].

Fineness also affects consistency of fresh cementitious mixtures (paste, mortar, and concrete). The particle size distribution controls the density with which cement particles pack, therefore influencing the fluidity (viscosity) and, indirectly, the strength.[2] With increased fineness generally comes a higher proportion of submicron-sized particles, which are prone to flocculate. Unless prevented through the use of dispersing admixtures (water-reducers or superplasticizers), flocculation substantially increases the water demand of the cement, either reducing slump or, if a higher water content is used, reducing strength.

Until recently, there have been few parameters in the grinding process that could be used to control the particle size distribution. Recent grinding technology utilizing high-efficiency classifiers allows better control of the distribution [10]. Helmuth and colleagues [11–13] have shown that controlling the particle size distribution of cement, particularly by limiting the coarse particles (>20 µm) and the fine particles (<2 µm), can substantially improve both performance and energy efficiency during grinding. This particle size control requires specialized mechanical particle separation equipment, which is gradually becoming available. It is anticipated that this technology will find increasing application in the future.

Standards

There are three standard tests to measure cement fineness–air permeability, turbidimeter, and sieving. The first, ASTM Test Method for Fineness of Portland Cement by Air Permeability Apparatus (C 204), allows measurement of the specific surface area, called the Blaine surface area after the inventor of the apparatus. The second test, ASTM Test Method for Fineness of Portland Cement by the Turbidimeter (C 115), uses the Wagner apparatus to measure the particle size distribution by sedimentation. In the third, ASTM Test Method for Fineness of Hydraulic Cement by the 45-µm (No. 325) Sieve (C 430), powder is allowed to pass through a standard-sized sieve assisted by a water spray.[3]

Fineness is included in most specifications for hydraulic cements. In C 150, a minimum specific surface value is specified for all types of portland cement except III and IIIA: either 280 m^2/kg by the air permeability test or 160 m^2/kg by the turbidimeter test. In C 595 for blended cements, fineness (both the amount retained on the 45-µm sieve and the specific surface by the air permeability method) is listed as a physical requirement and must be included if the purchaser requests certification, but no fineness limits are specified. In addition, in blended cements containing pozzolan, no more than 20.0 % of the pozzolan may be retained on a 45-µm sieve. Similarly, in C 1157 for hydraulic cements, fineness must be reported but no limits are specified.

Recommendations

The Wagner test (C 115) appears to be seldom used, and it would be sensible to replace it with the Blaine test (C 204) in all specifications.

Using particle size distribution rather than specific surface area would improve the specifications. The influence of fineness on hydration rate is well established, and there is a large body of literature relating Blaine surface area and such properties as rate of strength gain and heat of hydration. Likewise, the Blaine surface area has been a very reliable parameter for use in controlling particle size distribution during cement production. However, the surface area does not fully describe fineness. The full particle size distribution curve should be considered when assessing the effects of fineness on cement and concrete behavior. Two cements may have the same Blaine but quite different particle size distributions. The Blaine surface area is easy to

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[2] An example of the benefits when the particle size distribution is optimized to achieve very dense packing is Ductal™, a high-performance cementitious product that has high flowability, high strength, and high ductility.

[3] Two other tests are available using coarser sieves: (1) ASTM Test Method for Fineness of Hydraulic Cement by the 300-µm (No. 50), 150-µm (No. 100), and 75-µm (No. 200) Sieves by Wet Methods (C 786); and (2) ASTM Test Method for Fineness of Hydraulic Cement by the 150-µm (No. 100) and 75-µm (No. 200) Sieves (C 184).

measure, but new instruments for measuring particle size distribution based on sedimentation or light scattering [14] are also easy to use. Efforts have been underway for a while within ASTM Committee C01 to develop a standard test using such methods. The Blaine surface area is convenient to use because it is a single parameter, whereas the full particle size distribution is difficult to apply in correlations with other parameters. One solution is to use the Rosin Ramler distribution [4,10], which describes the particle size distribution in terms of two parameters, the average particle size and the breadth of the distribution.

Given the major influence of fineness on cement and concrete behavior, one might wonder why the minimum fineness is not specified in C 1157 as it is in C 150 and C 595. The answer is that fineness is a physical property but a prescriptive specification. The corresponding performance specifications are properties such as setting time, heat of hydration, strength, and water requirement (discussed below), and these are included in C 1157.

The prescriptive specifications should include maximum fineness values as well as minimum values. Low fineness slows down setting and strength development. High fineness increases stiffness and speeds up setting and strength development.

Consistency

Consistency refers to the flow behavior of a fresh mixture. It falls under rheology, the study of flow. Standard measures of consistency (for example, slump of concrete, flow of mortar, and penetration resistance of paste) are all empirical. Fundamental rheological measures (for example, yield stress and plastic viscosity) exist but are not used for cement and concrete except perhaps in research studies. Tattersall and Banfill [15] argued that fundamental measures of consistency have clear advantages over empirical measures, and that it is particularly important to be able to measure both yield stress and viscosity; whereas slump and flow relate largely to yield stress, the standard tests provide no information about viscosity. De Larrard and Ferraris [16] proposed a modification in the concrete slump to provide a measure of viscosity, but this modification has not been adopted as a standard. Cement paste, mortar, and concrete are fluid at high stress, but highly viscous or even solid at low stress. Therefore, behavior during processes that involve high stress, such as consolidation using vibration, is expected to correlate with viscosity; whereas behavior during processes that involve low stress, such as settling of aggregate due to gravity, is expected to correlate with yield stress.

The consistency of cement paste depends on the water-to-cement ratio and on various aspects of the cement: fineness, flocculation, and rate of hydration reactions. Flocculation (i.e., aggregation of particles), is substantially reduced by use of water-reducing chemical admixtures, which thereby cause reduction in yield stress and viscosity. The consistency of mortar and concrete depends on the consistency of the cement paste plus various aspects of the aggregate: quantity, grading, and shape.

Significance

The significance of cement consistency is twofold. Strength (for some cements), autoclave expansion, setting time, premature stiffening, and other properties are measured at a specified consistency rather than a specified water-to-cement ratio. A standard consistency is presumably used in these tests to avoid errors due to incomplete consolidation in samples with a very stiff consistency and errors due to bleeding in samples with a very fluid consistency. More importantly, cement consistency is generally assumed to affect concrete workability. Concrete workability (slump) is assumed to correlate with paste consistency at the same water-to-cement ratio and including the same mineral and chemical admixtures. Unfortunately, this correlation has eluded researchers, probably due (at least in part) to the highly empirical nature of both the cement and concrete consistency test methods. On the other hand, encouraging preliminary work on models of mortar and concrete flow behavior was reported by Ferraris et al. [17]. Nonetheless, changes in cement that in some way alter its consistency are assumed to have a similar affect on the workability of concrete made using that cement.

Standards

Consistency tests are normally used to measure the amount of water required to produce some standard consistency. There are two procedures: flow and penetration resistance. The flow test, ASTM Test Method for Flow of Hydraulic Cement Mortar (C 1437), provides for measuring the consistency of mortar using a standard flow table. This test is most commonly used, as in C 109, to establish the amount of water required to obtain a specified flow (105–115 in the case of C 109). Mortar with this flow is quite fluid and might be described as a batter (fluid enough to pour or drop from a spoon).[4]

The penetration test, ASTM Test Method for Normal Consistency of Hydraulic Cement (C 187), involves measuring the consistency of paste using penetration resistance. The normal consistency is measured using the Vicat apparatus equipped with a plunger and is achieved when the plunger penetrates to a point 10 ± 1 mm below the original surface. This consistency might be described as a dough (stiff enough to knead or roll).[4] This test is used when preparing pastes to measure setting time (discussed later) and, at a slightly different consistency, to measure premature stiffening (also discussed later).

Consistency is specified for blended cement, but not for portland or hydraulic cements. However, it must be measured and adjusted to a standard value when testing any hydraulic cement for compressive strength (other than portland and air-entraining portland, which are tested at fixed water contents), autoclave expansion, setting time, and premature stiffening (discussed later).[5]

Recommendations

The consistency tests allow selection of water-to-cement ratio for use in various other tests and are satisfactory for this purpose. However, the tests and specifications concerning consis-

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[4] As defined in the Webster's New International Dictionary, 3rd Edition, Unabridged.

[5] The same approach is used for mortar-bar expansion tests: ASTM Test Method for Effectiveness of Mineral Admixtures or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction (C 441) uses a flow of 100 to 115; and ASTM Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-bar Method) (C 227) uses a flow of 105 to 120 and a slightly modified procedure. A similar approach is used for various other tests, sometimes with modifications in the flow level: ASTM Test Method for Early Volume Change of Cementitious Mixtures (C 827), ASTM Test Method for Air Content of Hydraulic Cement Mortar (C 185), ASTM Test Method for Drying Shrinkage of Mortar Containing Portland Cement (C 596), ASTM Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution (C 1012), and ASTM Test Method for Length Change of Hardened Hydraulic-Cement Mortar and Concrete (C 157).

tency are particularly confusing and would benefit from clarification. In the previous edition of this chapter, it was recommended that the flow test be a separate standard, not part of C 109 as was then the case, and this change was subsequently made. There is considerable variability in flow values among the various standards, although there is no obvious reason for this variability, and a further improvement would be to adopt a single standard flow value. The relationship, assuming one exists, between cement consistency and concrete workability would be more clear if tests were structured to measure consistency at some standard water-to-cement ratio, rather than measuring the amount of water to provide some standard (and arbitrary) consistency. Such an approach would facilitate the use of cement consistency tests to study the influence of other factors on concrete workability (for example, chemical admixtures, mineral components, temperature, time). Finally, because cement consistency is an important contributor to concrete workability, consistency specifications should be developed for all hydraulic cements and for the paste system in concrete.

Because water requirement is such an important property of hydraulic cement, adding a specification on water requirement for standard consistency or requiring that the water requirement be reported would improve the specification for hydraulic cement, C 1157.

Test methods are needed that provide fundamental measures of consistency based on rheology, and efforts are underway to develop such methods. Consistency should be described using more than one parameter (for example, yield stress and plastic viscosity). Unfortunately, there are major technical difficulties in measuring rheological properties of mortar and concrete, and these difficulties will continue to limit development of improved standards in this area.

Set

Set refers to the transformation of cement paste, mortar, or concrete from a fluid material to a rigid solid. Set is a gradual and progressive change controlled by hydration of the cement and is closely linked to consistency. It is logical to expect that setting times of concrete would correlate with setting times of cement paste or mortar prepared using the same cement and the same water-to-cement ratio, although it is recognized that such correlation is not generally observed. Two setting times are recognized, initial set and final set. These times are arbitrary, in that they do not correspond exactly to any specific change in properties or to any specific levels of hydration reaction.

It is particularly important to show that the cement is not prone to premature stiffening. Normal set is generally attributed to the formation of calcium silicate hydrate (C-S-H). Cements occasionally show premature stiffening, either false set or flash set. In false set, the cement stiffens rapidly soon after mixing but regains its fluidity if remixed. False set may result from hydration of calcium sulfate hemihydrate ($C\overline{S}H_{1/2}$) to form gypsum ($C\overline{S}H_2$). It is also thought that false set can result from the formation of excess ettringite ($C_3A{\cdot}3C\overline{S}{\cdot}H_{32}$) soon after mixing, but this phenomenon has not been satisfactorily demonstrated. Finally, false set may result from flocculation of cement particles, rendering the mixture highly thixotropic in its rheological behavior. Flash set, on the other hand, occurs when the level of calcium sulfate is not sufficient to retard hydration of tricalcium aluminate (C_3A), allowing the formation of hexagonal calcium aluminate hydrates (C_4AH_{13} or $C_3A{\cdot}3C\overline{S}{\cdot}H_{12}$). In the case of flash set, fluidity cannot be regained on remixing. As noted in Wang et al. [18], one should not assume a particular hydration reaction based solely on measurements of setting behavior.

Significance

It is very important to predict and control setting time during concrete processing so concrete remains workable for a sufficient time that it can be placed and consolidated, but not for such a long time that finishing or form removal is excessively delayed. It is particularly important to prevent premature stiffening of the concrete due to false set or flash set. Because concrete set is controlled by reactions of the cement and water, perhaps modified by admixtures, it is expected that setting times of concrete may be measured using cement paste or mortar. The tests are used to ensure that the cement does not produce abnormal setting times or to test the response of a particular combination of cement and chemical admixture. Limits in setting time of paste and mortar also help assure overall concrete performance.

It has been noted (e.g., Ref 2) that set generally corresponds to the end of the induction period measured using isothermal calorimetry, the period when hydration begins to occur at a rapid rate. However, it is important to recognize that calorimetry is a measure of the extent of chemical reactions in the hydrating cement, while set is a microstructural phenomenon. It is reasonable to expect the chemistry to correspond to the microstructure only in a narrow range of initial conditions. The hydration reactions are necessary for set but not sufficient.

Standards

There are two laboratory tests for setting time of cement paste. In the ASTM Test Method for Time of Setting of Hydraulic Cement by Vicat Needle (C 191), normal consistency paste is used to measure resistance to penetration by a 1-mm needle at specified time intervals. Initial set occurs when the penetration is 25 mm, and final set occurs when there is no visible penetration. This apparatus is shown in Fig. 2. In the ASTM Test Method for Time of Setting of Hydraulic Cement Paste by Gillmore Needles (C 266), set is the time elapsed from mixing until the paste supports either the initial or the final needle without appreciable indentation. The test is run at a normal consistency (C 187, as described previously). There is no obvious reason to prefer one or the other of these tests, but it appears that the Gillmore test is being phased out and the Vicat test being retained as the standard.

There is also a test for premature stiffening (false set or flash set) of cement paste, ASTM Test Method for Early Stiffening of Portland Cement (Paste Method) (C 451), which utilizes the Vicat needle. Stiffening is measured shortly after mixing (5 min for paste and 11 min for mortar) to test for early stiffening and may be measured after remixing to help differentiate between false set and flash set. Similarly, the ASTM Standard Test Method for Early Stiffening of Portland Cement (C 359) utilizes the Vicat test to differentiate between false set and flash set of mortar.

Setting time is specified for all hydraulic cements in both prescriptive and performance specifications. For portland cement (C 150), initial set must be not less than 60 min and final set not more than 600 min using the Gillmore test (C 266); or initial set must be not less than 45 min and not more than 375 min using the Vicat test (C 191). There is also an optional false set specification, in which final penetration must be a minimum of 50 % of the initial penetration (C 451). For blended cement (C 595), initial set must be not less than 45 min and not more

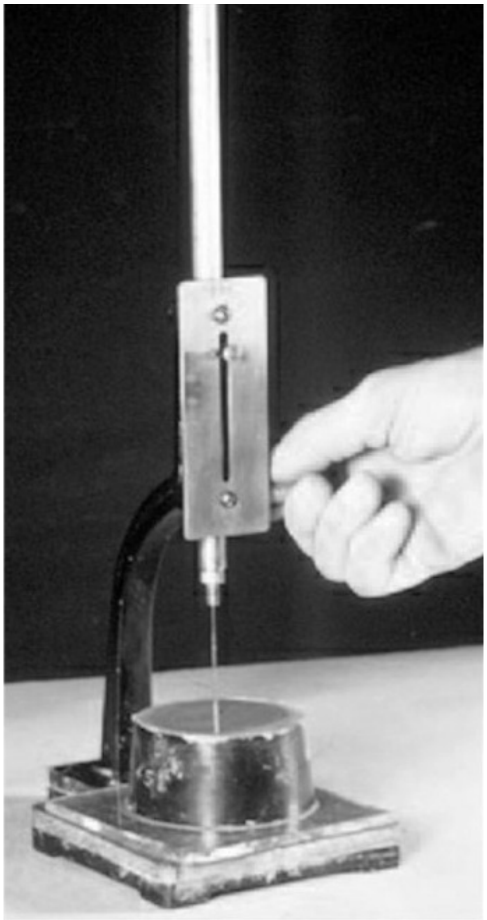

Fig. 2—Vicat apparatus (reprinted with permission from Ref 7).

than 630 min using the Vicat test (C 191), and there is no false set specification. For hydraulic cement (C 1157), initial set must be not less than 45 min and not more than 420 min using the Vicat test (C 191), and there is an optional false set specification that is the same as that just described for portland cement.

There is a separate test for set of concrete, the ASTM Test Method for Time of Setting of Concrete Mixtures by Penetration Resistance (C 403). This test is applied to mortar obtained from concrete by sieving coarse aggregate and uses a set of needles with varying diameters to measure the force to penetrate 25 mm. Penetration resistance is then computed as applied force divided by the needle bearing area. Interestingly, there is no specification for setting time in the ASTM Specification for Ready-Mixed Concrete (C 94), although there are specifications in the ASTM Specification for Chemical Admixtures for Concrete (C 494).

Recommendations

Although there is presumed to be a correlation between paste (or mortar) and concrete setting time, it can only be verified under standard laboratory conditions, because concrete setting time depends not only on cement content, water content, and admixture addition, but also on shear history and environmental conditions. It is hoped that the different setting tests (C 191, C 266, and C 403) approximately correspond with one another. While it appears that cement meeting the arbitrary limits in C 191 and C 266 produce satisfactory concrete, more research could lead to more specific and more meaningful definitions of initial and final set based on reactions occurring during early hydration (especially in the presence of admixtures, which can substantially affect the reaction kinetics). For premature stiffening, the limitations are even more arbitrary, since there are no guidelines to judge how and when an occurrence of premature stiffening in a cement may be manifested in concrete. Premature stiffening can also be drastically affected by cement and water contents, admixture presence, type of mixing, and environmental conditions in the concrete.

There are several improvements that could be made regarding set. As noted, efforts are underway to phase out the Gillmore test (C 266) in favor of the Vicat test (C 191). Even better would be to use the test for concrete (C 403), which is more robust, uses a more typical consistency, and produces more reasonable results [19]. In the future, rheological parameters might provide a way to define set that is not so arbitrary and allow separating the effects of hydration reactions from the effects of flocculation. Improvements could also be made in the specifications concerning set. It is not logical that initial and final set should be specified in C 150 for the Gillmore test, but only initial set should be specified in all three specifications (C 150, C 595, C 1157) for the Vicat test.[6] Furthermore, the range of times in all three specifications for the Vicat test is wide and should probably be narrowed, and the same values of setting time should be used in the three specifications. Finally, false set is specified in C 150 and C 1157 but not in C 595, where it seems equally important.

Heat of Hydration

During its first few days of hydration, concrete may produce a substantial amount of heat; and if this heat is not dissipated, it produces a substantial increase in temperature. Concrete that is subject to large temperature changes will crack, especially if the paste and aggregate have substantially different coefficients of thermal expansion. Concrete that reaches a temperature greater than about 70°C may deteriorate later due to delayed ettringite formation. The heat generated in concrete is a direct result of the cement hydration reactions, which are exothermic and occur most rapidly during the first hours and days after mixing. Thus, it is important to use cement with a moderate or low heat of hydration for structures from which heat is not rapidly dissipated or during hot weather, and preventing excessive temperature rise is the basis for specification of a Type II or IV portland cement.

The ASTM Test Method for Heat of Hydration of Hydraulic Cement (C 186) measures the difference in heat of solution of dry cement and cement that has been hydrated for either 7 or 28 days. The portland cement specification (C 150) includes optional limits for maximum heat of hydration using this test for Type II or Type IV portland cement (290 kJ/kg for Type II, 250 kJ/kg for Type IV at 7 days, and 290 kJ/kg for Type IV at 28 days). The blended cement specification (C 595) includes optional limits for maximum heat of hydration using this test for most types of cement if the moderate heat or low

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[6] In 1917 the final Vicat set in C 150 was limited to less than 10 h, but this limit was removed by 1958. In 1971 there was again a limit on final Vicat set, this time less than 8 h, but this limit was removed in 1983. Similar changes were made to C 595.

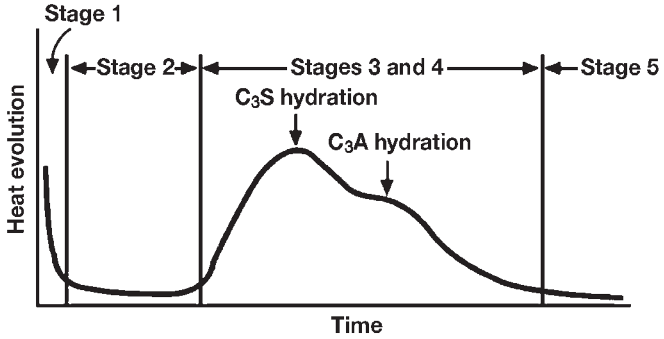

Fig. 3—Isothermal calorimetry curve of a typical portland cement (reprinted with permission from Ref 2).

heat option is specified (290 kJ/kg at 7 days and 330 kJ/kg at 28 days for Types IS and IP and 250 kJ/kg at 7 days and 290 kJ/kg at 28 days for Type P). The hydraulic cement specification (C 1157) includes limits for maximum heat of hydration using this test for moderate and low heat cements (290 kJ/kg at 7 days for MH and 250 kJ/kg at 7 days and 290 kJ/kg at 28 days for LH).

In addition, the heat of hydration makes it convenient to monitor degree of hydration using either isothermal or adiabatic calorimetry. A calorimetry curve of the type shown in Fig. 3 is often used in research studies to test hydration kinetics. Such a curve could be used to estimate heat of hydration at any specific age as an alternate method to the heat of solution measurement (C 186) if a suitable test method were developed. The advantage of using a calorimeter is that information would also be obtained concerning other aspects of hydration (for example, the time at which the induction period ends, which correlates approximately with initial set, and the balance between calcium sulfate and C_3A).

Volume Change

Concrete can undergo changes in volume (shrinkage or expansion) for a number of reasons: load (creep), drying and wetting cycles, expansive reactions that may occur in the cement, and expansive reactions that may occur between cement and aggregate. Creep and drying shrinkage are expected; these result from the porous nature of the cement hydration products. They are to some extent restrained in concrete by the aggregate, and must be taken into account during the design of concrete structures. Excessive expansion (unsoundness) may occur due to the hydration of free lime (CaO) to form calcium hydroxide ($Ca(OH)_2$) or the hydration of periclase (MgO) to form magnesium hydroxide ($Mg(OH)_2$). Excessive expansion may also occur due to the formation of ettringite ($C_3A \cdot 3C\overline{S} \cdot H_{32}$) through reaction of calcium aluminate (C_3A) or of calcium monosulfoaluminate ($C_3A \cdot 3C\overline{S} \cdot H_{12}$) with gypsum ($C\overline{S}H_2$) or some other source of calcium and sulfate. Finally, expansion may occur due to reactions between cement and aggregate. These fall under the category of durability problems and are discussed later in this chapter as well as in other chapters of this book.

Significance

The significance of the tests for volume change is to prevent deleterious volume changes, both expansion and shrinkage, in concrete.

Standards

Drying shrinkage is measured using ASTM Test Method for Length Change of Hardened Hydraulic Cement Mortar and Concrete (C 157). Although the test measures expansion or contraction due to any cause other than applied force or temperature change, it is used in this case to measure drying shrinkage. As noted above, all hydraulic cements are prone to drying shrinkage. It is known that composition and fineness of the cement can influence drying shrinkage, though the effects may not be large. Yet drying shrinkage is specified only for certain blended cements (P and PA in C 595), for which it is limited to a maximum of 0.15 %.

Expansion tends to be slow, so it must be accelerated in the laboratory by testing at an elevated pressure and temperature. The tendency to expand due to hydration of CaO or MgO is measured using the ASTM Test Method for Autoclave Expansion of Portland Cement (C 151). The apparatus and specimens are shown in Fig. 4. The specification for portland cement (C 150) sets a maximum autoclave expansion of 0.80 %, the specification for blended cement (C 595) sets a maximum of 0.50 %, and the specification for hydraulic cement (C 1157) sets a maximum of 0.80 %. Similarly, the ASTM Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete (C 618) sets a maximum autoclave expansion of 0.8 %. It is not obvious why the allowed expansion is lower for blended cement than for portland cement, or why the expansion is lower for blended cement than it is for mineral admixtures in concrete.

Although the autoclave expansion test is used in several specifications for blended cements and mineral admixtures, the test is explicitly applicable only to portland cement. One reason is that blends of portland cement and fly ash may produce excessive autoclave expansion due to alkali-silica reaction [20]. Autoclave expansion test results do not correlate well with concrete performance in cases of unsoundness due to MgO and C_3A hydration [21].

The blended cement specification (C 595) also sets a maximum limit for autoclave contraction, but the significance of this specification is not obvious. The limit was apparently imposed to prevent excessive contraction that had been reported in blended cements containing slag. The contraction may have merely been autogenous shrinkage (that is, shrinkage due to hydration because the hydration products occupy less volume than the reactants).

As previously noted, expansion during hydration of portland cement may also occur due to excessive formation of ettringite ($C_3A \cdot 3C\overline{S} \cdot H_{32}$). The limit on SO_3 level in C 150 (and discussed in the chapter on chemical composition) is intended to prevent such expansion. However, for some portland cements the SO_3 required to optimize strength or drying shrinkage is greater than allowed by this limit. In that case, the higher level of SO_3 may be used as long as the cement does not develop expansion greater than 0.020 % at 14 days when tested using the ASTM Test Method for Expansion of Portland Cement Mortar Stored in Water (C 1038). The same requirement is included in the specification for hydraulic cements (C 1157), but there is no expansion requirement in the specification for blended cements (C 595).

Expansion similar to that produced by alkali-silica reaction, which may occur in concrete and is discussed in this chapter in the section on durability, is possible in blended cements due to reaction between alkalies from the cement and silica in the pozzolan. To ensure that such a reaction does not cause

Fig. 4—Autoclave expansion apparatus and specimens (reprinted with permission from Ref 7).

deleterious expansion in concrete, a mortar expansion test for pozzolan used in blended cement is included as part of C 595 and C 1157. This method tests combinations of pozzolan and cement or clinker using the ASTM Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method) (C 227). This test is usually used to detect the alkali-silica reaction between cement and aggregate, but in this case it is used with a nonreactive aggregate to measure expansive reactions between pozzolan and cement. For C 595, the test is run with various proportions of pozzolan between 2.5 % and 15 % in order to detect excessive expansion due to an adverse proportion of pozzolan in combination with portland cement, and the maximum expansion for all mixtures is 0.05 % at 91 days. For C 1157, the test is run with the proportion of pozzolan used in the cement and 5 % more and less than that proportion; and, although the specification describes the test, it does not list a maximum expansion level.

Recommendations

Several of the standards relating to volume change need improvement. Drying shrinkage is an important issue in concrete; it initiates in the hydrated cement and is affected by properties of the hydraulic cement, so it seems important to have a shrinkage requirement for hydraulic cements. The current restriction of the autoclave expansion test, C 151, to portland cement is a major problem, and the subcommittee responsible for this test is currently exploring tests for cements containing pozzolan to measure expansion due to CaO and MgO hydration associated with unsoundness in concrete. There should be a limit on expansion due to ettringite in C 595. The test for cement-pozzolan expansion would be easier to utilize if it were described more fully in a separate method, rather than included as part of the blended cement specification. Finally, although there is a limit on cement-pozzolan expansion in C 595, there is no similar limit for fly ash used as a mineral component in concrete (C 618) nor for hydraulic cements that contain fly ash, a puzzling inconsistency.

Strength

Strength is the property that is probably most important to engineers, both as a general indicator of concrete quality and to assure that the concrete will perform as intended during design of the structure. Although concrete strength may be measured in tension, shear, or compression, compressive strength is generally most important and most often specified.

Significance

It is widely recognized that concrete strength depends on the strength of cement paste, on the paste-aggregate bond, and on the aggregate strength. For ordinary concrete, the strengths of paste and the paste-aggregate bond control concrete strength. It is only for high-strength concrete or unusually low-strength aggregate that aggregate characteristics become important. The strengths of both paste and the paste-aggregate bond depend largely on paste porosity. Porosity is reduced (and strength is increased) by initially packing particles very densely and by filling remaining voids with hydration products. So strength is increased by reducing the water-to-cement ratio and by increasing the degree of hydration. The latter parameter is influenced considerably by the composition and microstructure of the cement, and both parameters are influenced by fineness of the cement.

Based on these considerations, one would expect a good correlation between concrete strength and strength of mortar at the same water-to-cement ratio and degree of hydration. Weaver et al. [*22*] showed no such correlation. However, Neville [23] reported a reasonably good linear correlation. Gaynor [24] similarly showed a good correlation between mortar and concrete strength, concluding that concrete strength may be predicted from mortar strength, water-to-cement ratio, and air content of the concrete. The lack of correlation in some studies may reflect variability in parameters such as air content, aggregate moisture, and curing conditions.

The nature of the fracture process has important implications to mortar and concrete strength. When concrete is loaded, small cracks develop at the interface between cement paste and aggregate. The cracks may develop because this interface is weaker than the bulk paste or the aggregate, or they may exist in concrete before it is loaded due to drying shrinkage or thermal stresses. The development and growth of these cracks reduce concrete strength. Therefore, it is expected that fracture mechanics, the area of research concerned with development and growth of cracks, will provide a fuller understanding of strength and fracture.

The significance of mortar strength is broader, however, than its relationship to concrete strength. Many of the potential problems in cement hydration affect its strength. Furthermore, permeability of hydrated cement is a function of water-to-cement ratio and degree of hydration just as is strength, so permeability decreases as strength increases, and many aspects of durability are improved by increasing strength. Thus, strength provides an indication of the overall quality of hydrated cement, mortar, or concrete.

Standards

There are several tests for mortar strength (compressive, tensile, and flexural strength), though the specifications utilize only C 109 (compressive strength).[7] This laboratory test allows measurement of compressive strength of mortar prepared using a graded standard sand[8] and water adjusted to provide either a specified water-to-cement ratio or a specified flow. Specimens are shown in Fig. 5. A specified water-to-cement ratio is used for portland cement, either 0.485 for portland cement or 0.460 for air-entraining portland cement. A specified flow of 105 to 115 is used for all other cements. Cubes are moist-cured 20–72 h in their molds, then stripped and immersed in lime-saturated water until they are tested.

Minimal compressive strength requirements are specified for all hydraulic cements. These strength levels are easily met by cements with a wide variety of chemical composition and fineness levels.

In C 150 and C 595, minimum average compressive strength levels measured using C 109 are specified. Most types of portland cement (C 150) have compressive strength requirements at three and seven days, though some cement

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[7] Other mortar strength tests include ASTM Test Method for Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure) (C 349); ASTM Test Method for Tensile Strength of Hydraulic Cement Mortars (C 190), now discontinued; and ASTM Test Method for Flexural Strength of Hydraulic Cement Mortars (C 348).

[8] A natural silica sand from Ottawa, IL, that conforms to the ASTM Specification for Standard Sand (C 778).

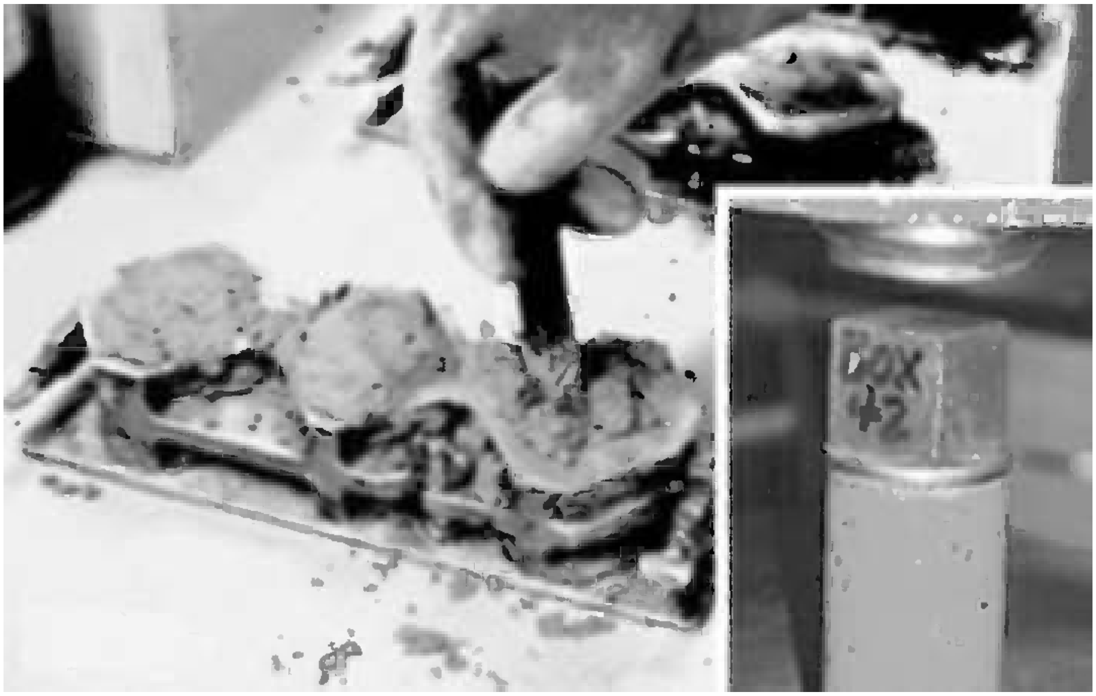

Fig. 5—Casting and crushing mortar cubes for strength (reprinted with permission from Ref 7).

types have 1- and 28-day requirements (some 28-day requirements are optional). For Type I, the 7-day specified strength is 19.0 MPa. The values are slightly lower for Type II cement, even lower for Type V cement, much lower for Type IV cement, and higher for Type III cement. Values are lower for each corresponding air-entraining cement. Likewise, strengths are specified at 3, 7, and 28 days for most blended cements (C 595), though some types do not have three-day specifications. For Types IS and IP blended cement, the 7-day strength level is 20.0 MPa, similar to the level specified for Type I portland cement, while specified levels are slightly lower for Type MS cement, and much lower for Types P and S cement.

In C 1157, the performance specification for hydraulic cement, compressive strength may be specified as a range (both a minimum and a maximum strength value). Most types of hydraulic cement have compressive strength requirements at three and seven days, though some have 1- and 28-day requirements. For Type GU hydraulic cement, the 7-day specified strength range is 17–30 MPa, somewhat lower than the minimum for Type I portland cement and Types IS and IP blended cement. The specified range is the same for Type MS cement, lower for Type MH cement, much lower for Type LH cement, and higher for Type HE cement.

Recommendations

As noted previously, the existing strength specifications are well below strength levels obtained in current practice. In fact, these strength specifications do not represent acceptable minimum strength levels, and they are so low as to be of limited use. It would be an improvement if the strength specifications were raised. By raising the specifications to levels more consistent with standard practice, cement users would get a product that provides acceptable minimum strength levels.

Even more important is the issue of strength uniformity. Strength specifications for all hydraulic cements would be enhanced if they were modified to include some restriction on variability. In this way, the cement user would be guaranteed a uniform performance as well as a specified minimum strength level. There is a method for uniformity, ASTM Test Method for Evaluation of Cement Strength Uniformity from a Single Source (C 917), but no specification.

Although we use strength in compression to describe the performance of cement and concrete, failure (that is, development and growth of cracks) typically occurs in tension. Concrete is a brittle material, with a tensile strength only about one tenth of its compressive strength. Other types of strength, tensile and shear, are generally influenced by the same parameters that control compressive strength–in particular, the water-to-cement ratio and the degree of cement hydration. Therefore it is assumed that specifications and tests for compressive strength are applicable to other types of strength. This is a simplifying assumption, in that compressive strength is much easier to measure. However, this assumption is not without risk. It is known that the ratio of tensile strength to compressive strength is not constant, but rather is affected by a number of parameters, from air drying during aging (which reduces tensile strength more than it does compressive strength) to air entrainment (which reduces compressive strength more than it does tensile strength). While it is probably not necessary to have additional test methods and specifications for tensile strength of cement, it is certainly important to appreciate the differences between tensile and compressive strength.

As discussed earlier, fracture mechanics is concerned with failure due to cracking. This is an active area of research and should provide a better understanding of the processes of cracking in concrete, mortar, and paste. Although there is currently no ASTM test for fracture mechanics parameters of concrete, fracture mechanics tests have been developed that are suitable for use as standard test methods for these materials [25]. The ACI Committee on Fracture Mechanics of Concrete has concluded that fracture mechanics now (after intense research during the last decade) appears ripe for applications in design practice [26]. However, considerable work remains to be done before concrete strength (or strength-related performance of concrete structures) can be predicted from measurements of fracture mechanics parameters, and it is not yet clear whether fracture mechanics parameters of concrete may be estimated from parameters measured on its constituents.

Optimum Sulfate Content

The amount of calcium sulfate in cement is not a physical property (and therefore not included in Table 2). However, it is discussed in this chapter because it is generally determined using physical tests.

Significance

Calcium sulfate is added to cement to control hydration of C_3A. Without sufficient sulfate available in solution, rapid C_3A hydration producing C_2AH_8 and C_4AH_{13} (so-called hexagonal hydrates) causes premature stiffening known as flash set, as discussed previously. With sufficient sulfate, calcium sulfate reacts with C_3A and water to produce ettringite, and the initial ettringite slows down subsequent C_3A reaction and prevents false set. If too much calcium sulfate is used, on the other hand, the cement may produce excessive expansion at later ages due to formation of ettringite after set, also discussed previously. Thus one would expect to select the amount of calcium sulfate that produces normal setting without excessive expansion.

Interestingly, the amount of calcium sulfate in hydraulic cement is instead determined based on early strength. The effect of sulfate content of cement on both strength and drying shrinkage was established as early as 1946 [27], when Lerch showed that strength is maximized and shrinkage is minimized as a function of sulfate content. The mechanism of this phenomenon is not well understood, but it seems likely that the proportion of sulfate affects the degree of porosity during early aluminate hydration reactions, though porosity is modified by subsequent silicate hydration. Optimum strength presumably corresponds to minimum porosity at any age. Some cements, however, show a change in their sulfate requirements at later ages, an observation that is difficult to explain.

Standards

The standard test for this determination is the ASTM Test Method for Optimum SO_3 in Portland Cement (C 563). This method only evaluates optimum sulfate for one-day strength and does not address drying shrinkage.

If the amount of sulfate shown to be optimum by this test exceeds the amount allowed for the particular cement, use of the higher amount must be shown to cause no harm. Details vary according to the specification. In C 150, the maximum sulfate allowed is 2.3 % to 4.5 %, depending on type and on the level of C_3A; and if the amount determined according to C 563 exceeds this maximum, the higher amount may be used only if it is shown that the cement does not produce expansion exceeding 0.020 % at 14 days according to C 1038. In C 595, the maximum sulfate allowed is 3.0 % or 4.0 %, depending on type; and if the amount determined according to C 563 exceeds 0.5 % less than this maximum, the higher amount may be used only if it is shown that the soluble sulfate in mortar does not exceed 0.50 g/L when tested according to ASTM Standard Test Method for Water-Extractable Sulfate in Hydrated Hydraulic Cement Mortar (C 265). No additional requirement is needed in C 1157 because that specification does not have a prescriptive sulfate level and already has a limit on expansion using C 1038.

Recommendations

There is a caution in C 563 that the sulfate content shown to produce optimum one-day strength may not provide maximum strength at later ages or at other temperatures and may not provide minimum drying shrinkage. Flow behavior with water-reducing admixtures is sometimes affected by the sulfate level [28], yet flow is not even measured when setting the sulfate level in cement. Further research is needed to understand how sulfate affects flow and set in complex mixtures of cements and admixtures, so that tests and specifications can more clearly be tied to the critical concrete properties (strength, shrinkage, flow, and set).

It would be more rational if C 150 and C 595 had the same additional requirements when using excessive sulfate.

Durability

There are several concrete deterioration processes that relate in part to properties of the cement–freeze-thaw deterioration, alkali-silica expansion, and sulfate reaction. These deterioration processes are discussed in greater detail in other chapters of this book. Because they relate in part to cement properties, tests to predict the influence of the cement on each deterioration process are discussed here.

Significance

Air Content

The air content of concrete controls its ability to withstand cycles of freezing and thawing. Without entrained air, most concrete will deteriorate after only a few freeze-thaw cycles. Air is obtained in concrete using an air-entraining admixture, a surfactant that lowers the surface tension of water and thereby stabilizes air bubbles. Whether the added air is sufficient to protect against freeze-thaw damage depends not only on the volume of air, but also on the size and distribution of the bubbles throughout the paste portion of the concrete.

An air-entraining admixture may be added to the concrete or as part of the cement (called an air-entraining cement). As noted in Table 1, ASTM recognizes several air-entraining cements (designated A) in both portland and blended cements.

The volume of entrained air in concrete depends not only on the dosage of air-entraining agent (whether added directly to the concrete or as part of an air-entraining cement), but also on various other parameters of the concrete and how it is mixed. It should be noted that even a non-air-entraining cement may influence the air content in concrete. Finely ground cements entrain less air than do coarsely ground cements. Use of finely ground slag or pozzolan in blended cements also reduces the air content and necessitates a larger dosage of air-entraining admixture. Most importantly, fly ash in a blended cement may reduce the air content due to adsorption of air-entraining agent, in particular by carbon impurities in fly ash.

Gaynor [24] addressed the correlation of air content in mortar and concrete. Correlations were good (standard errors of estimated air content in concrete were 0.3 % to 0.8 %), especially considering the inherent variability of air content tests. But he noted that air contents in mortar are higher than air contents in concrete and that many non-air-entraining cements produce mortar air contents greater than 10 %.

Concrete air content is also important because air (whether entrained or entrapped) reduces strength. The rule of thumb is that every 1 % increase in air content reduces strength of concrete by about 5 % [3]. Thus, it is important that no more air than necessary be entrained.

Alkali-Silica Reaction

Another common cause of concrete deterioration is the alkali-silica reaction, in which certain forms of silica (amorphous or reactive crystalline forms), generally part of the aggregate, react with the highly alkaline pore fluid in concrete to produce an alkali-silica gel. This gel may sorb water and swell, causing expansion or cracking of the concrete.

The alkalies (sodium and potassium)[9] in concrete are typically derived from the cement. Deleterious expansion is usually prevented through use of a low-alkali cement, an optional specification for portland cement. Hydraulic cement is tested for its expansion when combined with a reactive aggregate. Expansion is generally reduced by using a blended cement (pozzolan and slag reduce the alkalinity of the pore fluid), but not every blended cement is effective in reducing expansion, so specific pozzolans and slags must be tested.

Sulfate Reaction

Attack of concrete by sulfate ions is a particular problem where the sulfate concentration surrounding the concrete is high, such as in soils in the western part of the United States. The attack involves reactions between cement hydration products and sulfate ions, reactions that are expansive and cause cracking and deterioration. The primary reaction is between $C_3A{\cdot}\overline{C}S{\cdot}H_{12}$ and calcium sulfate to produce $C_3A{\cdot}3\overline{C}S{\cdot}H_{32}$. To prevent this deterioration, it is necessary to limit the amount of $C_3A{\cdot}\overline{C}S{\cdot}H_{12}$ in the hydrated cement, usually by reducing the amount of C_3A in the cement, as in a Type II or V portland cement or a blended cement. If the environmental sulfate salt contains a cation other than calcium, then further reaction may occur in which calcium hydroxide reacts to form gypsum ($C\overline{S}H_2$); and when calcium hydroxide is depleted the calcium silicate hydrate is progressively decalcified to produce additional gypsum. Because the calcium silicate hydrate is the principal strength-producing phase in hydrated cement, this reaction leads to major loss of strength as well as expansion. To prevent this deterioration it is also important to reduce the diffusivity of the cement paste, generally by reducing its water-to-cement ratio.

The resistance of a particular cement to sulfate attack is important because it relates directly to the performance of concrete when exposed to sulfate ions.

Standards

Air Content

The potential of cement to entrain air is determined using the ASTM Test Method for Air Content of Hydraulic Cement Mortar (C 185). In this test, the air content of mortar is measured by the difference between measured and calculated volume, the calculated volume depending on the proportion and density of each mortar constituent.

Both minimum and maximum air contents are specified for each air-entraining hydraulic cement. For non air-entraining cements (portland and blended), only a maximum air content is specified. The minimum air content assures that the air-entraining cement provides entrained air in concrete for resistance to freeze-thaw deterioration. The maximum air content assures that no unacceptable loss of strength will occur due to entrained air.

The specified air content is the same for portland cement (C 150) and blended cement (C 595). The maximum air content for non-air-entraining cement is 12 %. For air-entraining cements, the minimum air content is 16 % and the maximum air content is 22 %. For hydraulic cements (C 1157), there is no air-entrained option, so there is no specified air content, though there is a requirement that it be measured and reported.

Alkali-Silica Reaction

The tests to determine whether a specific pozzolan or ground slag used in a blended cement or a specific hydraulic cement is reactive when combined with reactive aggregate are based on one of the mortar tests used to determine whether a specific aggregate is reactive, C 227. Two tests are used, C 227 with crushed Pyrex[10] glass as the reactive aggregate, and C 441, there being no substantive difference between the two procedures. These tests are only reliable, however, insofar as the alkali-silica expansion of mortar bars corresponds to the occurrence of deterioration in concrete, and there is concern within ASTM Committee C09 about the validity of mortar-bar tests.

Portland cement (C 150) relies on the optional prescriptive low-alkali requirement to prevent alkali-silica reaction. The other specifications rely on performance limits. Both the specification for blended cement (C 595) and the specification for hydraulic cement (C 1157) include optional maximum limits for mortar expansion due to alkali-silica reaction: 0.020 % expansion at 14 days and 0.060 % expansion at eight weeks. These expansion levels are different than those in the ASTM Specification for Concrete Aggregates (C 33), which are 0.05 % at three months and 0.10 % at six months. They are also different from the levels specified in related standards for mineral components in concrete, which are 100 % of the value using a low alkali cement control for fly ash and natural pozzolans in C 618, and an expansion of 0.020 % at 14 days for slag in C 989.

Sulfate Reaction

The resistance of portland cement to external sulfate attack is measured using the ASTM Test Method for Potential Expansion of Portland Cement Mortars Exposed to Sulfate (C 452). Excess sulfate is added to the portland cement in the form of gypsum, and length changes of mortar bars prepared from this cement-gypsum mixture are measured. This was found to be a satisfactory test for predicting the performance of portland cement in concrete exposed to sulfate ions, but not for blended cement [31]. Therefore an alternative test was developed for blended cements, the ASTM Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution (C 1012). In this test, bars are immersed in a sodium sulfate solution and their length changes measured. Because the sulfate salt is sodium, not calcium, the test method produces deterioration due to precipitation of gypsum as well as precipitation of ettringite, making C 1012 appear more aggressive than

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[9] These chemical constituents and their determination are discussed in a separate chapter of this book, Hydraulic Cements–Chemical Properties.

[10] Pyrex Glass No. 7740 from Corning Glass Works, Corning, NY.

C 452, although the expansion level is higher and produced more rapidly in C 452, perhaps because the excess sulfate is part of the cement.

Sulfate expansion as an alternative to the prescriptive limit on C_3A is an optional requirement for Type V portland cement; the maximum expansion at 14 days using C 452 is 0.040 %. The limit of sulfate expansion for moderate sulfate-resisting blended cement (Type MS) using C 1012 is 0.10 % at 180 days. Similarly, the limit on sulfate expansion for moderate sulfate-resisting hydraulic cement using C 1012 is 0.10 % at six months and for highly sulfate-resisting hydraulic cement using the same test it is 0.05 % at six months and 0.10 % at 12 months.

Recommendations

Alkali-Silica Reaction

The level of expansion specified for blended cement and for hydraulic cement is more stringent than the level specified for aggregate (C 33) and for pozzolan in concrete. It seems reasonable that these levels should be the same, in that they apply the same test method to the same deterioration process, and it has been recommended that the cement limits be relaxed [29].

A more basic concern regarding these tests is their use of Pyrex glass as a standard reactive aggregate. Studies have shown that Pyrex is not suitable as a standard aggregate [30]. For many years now, the subcommittee responsible for the alkali-silica tests in concrete has been exploring alternatives for the standard reactive aggregate.

Sulfate Reaction

The specifications for moderate sulfate-resisting cements should be brought into conformance; C 595 limits expansion at three months and C 1157 limits expansion at six months.

Discussion

More knowledge is needed about the fundamental mechanisms responsible for the various properties of cement paste, mortar, and concrete. Many of the tests described here are empirical and need a better fundamental basis. In some cases, the fundamental knowledge is available but just needs to be applied in standards (as in the case of consistency); but, in most cases, additional research is needed.

There are many benefits of testing paste or mortar rather than concrete. Tests using paste or mortar are generally preferred because they are simpler than tests using concrete. There is no need to develop a standard coarse aggregate. The effects of mineral and chemical admixtures on concrete performance may be studied using paste or mortar. But the results from paste or mortar do not necessarily correlate with concrete, and further research is needed to make such correlations. Furthermore, it is important that tests of paste or mortar are able to evaluate the mixtures and proportions of materials used in concrete (typical water-to-cement levels, chemical admixtures, and mineral admixtures). Finally, it is important to be testing the paste system, not just combinations of cement and water.

Many tests developed for portland cement have been applied to blended cement and expansive cement without modification. Some tests are satisfactory for all hydraulic cements, but many tests (for example, fineness and sulfate deterioration) are not. Efforts are generally underway to modify existing tests or develop new tests for blended cement and expansive cements.

One aspect of concrete performance that we do not currently measure, and which depends largely on properties of the cement, is permeability. Permeability, or the related parameter of diffusivity, refers to the rate at which fluid, or ionic species, moves through the material. In hardened paste, mortar, or concrete, such movement takes place through the small pores, either capillary pores or the much smaller gel pores. Thus, permeability is in many respects the inverse of strength, in that it is reduced either by lowering the water-to-cement ratio or by increasing the degree of hydration. As with strength, the permeability of concrete depends, at least broadly, on the permeability of the paste portion of the concrete. Permeability of paste depends on the volume and interconnectedness of pores. Concrete permeability depends also on permeability of the aggregate and of the paste-aggregate interface, and may be increased by microcracking in the paste or at the interface. Despite its importance, there is currently no standard test method by which to measure paste or concrete permeability. Several laboratories are carrying out concrete permeability measurements, so perhaps a standard test method will be developed in the near future. Because permeability is so important to concrete durability, it is an important area for further research and for the development of new standard test methods for both cement paste and concrete.

Creep is another important property of hydrated cement that is not currently measured. Concrete deforms under load, and these creep deformations must be measured or predicted and incorporated into design of concrete structures. Creep, like drying shrinkage, occurs in the hydrated paste and is restrained by aggregate. Creep depends on the level and duration of load. Like shrinkage, permeability, and strength, creep also depends on the pore structure of the hydrated paste, the water-to-cement ratio, the degree of hydration, and the moisture content. Much recent research is concerned with early-age creep, which was the topic of a recent RILEM report [32]. It would seem useful to measure creep of cement paste or mortar in order to determine the influence of the specific cement on creep in concrete.

Finally, there is as yet no standard or specification concerning deterioration in concrete due to excessive formation of ettringite after set, so-called delayed ettringite formation (DEF). As discussed in the section on Optimum Sulfate, C 1038 and C 265 are used in C 150, C 1157, and C 595 to prevent the use of excess sulfate, which may be one cause of DEF. However, these limits are only invoked in C 150 and C 595 in the event that the optimum sulfate exceeds a prescriptive level. Furthermore, C 1038 appears to not be a suitable performance test for DEF [33]. Clearly, a better strategy is needed to protect against this deterioration process.

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38

Hydraulic Cement-Chemical Properties

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Preface

A CHAPTER ON THE CHEMICAL PROPERTIES OF hydraulic cements was included in *ASTM STP 169C* and this chapter is largely similar to that in the previous edition. It provides an overview and introduction to the chemistry of hydraulic cements, which has been, and remains, a fascinating and challenging topic for researchers, engineers and specifiers. Many aspects of hydraulic cement chemistry directly influence cement properties and performance in concrete, and an understanding of the chemistry can provide insight into specification provisions and test methods.

Introduction

This chapter describes the ASTM specifications and test methods covering the composition and analysis of hydraulic cements. More importantly, the effects of chemical properties on concrete performance are described because this area is of more concern to the user than a detailed discussion of testing procedures.

The audience is assumed to be those who use or specify cement to make ready-mixed concrete and other types of concrete products. The information may also be of interest to laboratory personnel, students, and researchers new to the field of cement chemistry and concrete technology. The objective of this chapter is to provide sufficient information about the chemical properties of hydraulic cements to enable the user to interpret ASTM specifications and understand the technical documentation provided by cement manufacturers. An understanding of the chemical properties of cement will also aid in evaluating and predicting the performance of cement in concrete. For the reader desiring more detailed information on cement chemistry, there are a number of textbooks and articles that provide more complete coverage of the subject [1–3].

Hydraulic cement is defined in ASTM Terminology Relating to Hydraulic Cement (C 219) as “a cement that sets and hardens by chemical interaction with water and that is capable of doing so under water.” This chapter will cover two classes of hydraulic cements—portland cements and blended hydraulic cements—although emphasis will be given to portland cement because there are more constraints on chemical composition in ASTM specifications for these cements. Masonry, expansive, and other portland-cement-based materials will not be discussed, although many of the principles related to chemical composition also apply to special cements.

Portland Cement

According to the definition in ASTM C 219, portland cement is “a hydraulic cement produced by pulverizing portland-cement clinker, and usually containing calcium sulfate.” Portland-cement clinker is made by heating a finely-ground raw mix in a large kiln, to temperatures of about 1500°C. A raw mix is made by blending a carefully-proportioned mixture of calcium, silica, alumina, and iron-bearing materials to achieve the correct chemical composition for the kiln product—nodules of portland-cement clinker. As the raw mix is heated, moisture and carbon dioxide are driven off, and compounds of silicates, aluminates, and ferrites are formed during the clinkering reactions. These chemical compounds, referred to as phases, are responsible for the setting and strength development characteristics of portland cement. The final step in the manufacturing process requires crushing and grinding the clinker nodules to make the powder that is sold commercially as portland cement. Gypsum, or another form of calcium sulfate, is added during this final grinding step to reduce the rate of the aluminate reactions, which would otherwise make the cement difficult to use.

The properties of portland cement can be varied to some extent by using raw materials of different proportions or chemical composition and thereby changing the composition of the resulting clinker. The performance of portland cement will also be influenced by the manufacturing process; the heating and cooling conditions can affect clinker reactivity, and finish grinding determines the particle size distribution (effects of which are discussed in Chapter 39). This discussion will focus on the chemical composition, with attention to the requirements in the ASTM Specification for Portland Cement (C 150).

Chemical Composition of Portland Cement

The chemical elements found in portland cement are generally expressed as oxides; SiO_2, Al_2O_3, Fe_2O_3, and CaO are the four main oxides found in clinker and cement. The compounds (or phases) responsible for hydraulic activity are expressed as combinations of oxides. One of the major components in cement is tricalcium silicate, a phase consisting of a combina-

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TABLE 1—Cement Chemist's Shorthand for Oxides

Oxides	Abbreviations
SiO_2	S
Al_2O_3	A
Fe_2O_3	F
CaO	C
MgO	M
SO_3	$\bar{S}$
Na_2O	N
K_2O	K
CO_2	$\bar{C}$
H_2O	H

tion of calcium oxide (CaO), and silicon dioxide (SiO_2). Using standard chemical nomenclature, the formula for tricalcium silicate is written Ca_3SiO_5, but a cement chemist often expresses it as $3CaO{\cdot}SiO_2$, indicating that the compound is composed of three units calcium oxide and one unit silicon dioxide. To further simplify the notation, individual oxides are abbreviated as a single letter. The abbreviations used to describe the major and minor oxides in portland-cement clinker are listed Table 1.

Using these abbreviations, $3CaO{\cdot}SiO_2$ becomes C_3S. The other primary phases are C_2S ($2CaO{\cdot}SiO_2$), C_3A ($3CaO{\cdot}Al_2O_3$), and C_4AF ($4CaO{\cdot}Al_2O_3{\cdot}Fe_2O_3$). These chemical formulas can be threatening to the nonchemist, but it is quite possible to comprehend and discuss the chemical properties of cement referenced in ASTM standards using only the abbreviations.

Note the use of the term *phase* is used instead of *compound*; a phase is a chemically impure compound, and cement phases like C_3S contain several percent by mass of other elements. These "impurities" are part of the materials that make up the raw feed and actually help make the cement more reactive.

Phase Composition

Although the chemical composition of a cement is expressed in terms of its oxides, it is important to note that the phase composition of a cement is not adequately described by a list of oxide weight percentages. Table 2 shows the chemical analysis results for an ASTM Type I/II cement. In addition to the oxide results (Table 2*a*), the phase composition is given (Table 2*b*). In cement chemistry, the term "phase composition" is often used to indicate the composition of clinker and cement. In this paper, the phrase "chemical composition" refers to the oxide analysis, and the phrase "phase composition" will be used when discussing the compounds (phases) found in clinker and cement. The single-letter abbreviations will only be used in the name of the phases. The oxides are listed in reports in the approximate order in which they are determined by classical methods of chemical analysis, as recommended in ASTM C 114, Standard Test Methods for Chemical Analysis of Hydraulic Cement.

TABLE 2a—An Example of a Typical Portland-Cement Composition, Expressed as Oxides

Oxide	Percentage
SiO_2	22.0
Al_2O_3	4.3
Fe_2O_3	3.1
CaO	64.5
MgO	2.2
SO_3	2.6
Na_2O equivalent	0.65

TABLE 2b—An Example of a Typical Portland-Cement Composition, Expressed as Potential Phases

Phase	Percentage
C_3S	54
C_2S	22
C_3A	6
C_4AF	9

The oxide analysis in Table 2 shows 22.0 % total SiO_2, but the SiO_2 is distributed between the two major calcium silicate phases, C_3S and C_2S. Similarly, the cement contains 64.5 % CaO, but it is distributed among each of the major phases; C_3S, C_2S, C_3A, and C_4AF. There may also be about 1.0 % free lime (f-CaO), that is CaO that was not combined during the clinkering process. In addition, about 2 % of the CaO in the cement will be from calcium sulfate added during the grinding process.

Bogue Calculations

Determination of the oxide composition of a cement is fairly straightforward, but the determination of the actual phase composition is more difficult. In order to correlate chemical composition and cement performance, it is necessary to know how much of each phase is present. Historically, the Bogue calculation has been used to calculate the "potential" phase composition. Developed by Bogue in 1929, this procedure is based on a number of assumptions [4]. There are many variations of the Bogue calculations [for example 1,5], but the following equations are used in ASTM C 150:

$$C_3S = 4.071CaO - 7.600SiO_2 - 6.718Al_2O_3 - 1.430Fe_2O_3 - 2.852SO_3 - 5.188CO_2 \quad (1)$$

$$C_2S = 2.867SiO_2 - 0.7544C_3S \quad (2)$$

$$C_3A = 2.650Al_2O_3 - 1.692Fe_2O_3 \quad (3)$$

$$C_4AF = 3.043Fe_2O_3 \quad (4)$$

The use of these equations assumes that the clinker reactions go essentially to completion, and also that the four major phases are pure C_3S, C_2S, C_3A, and C_4AF. Equations 3 and 4 are modified if the ratio of Al_2O_3 to Fe_2O_3 is more than 0.64, because a solid solution–expressed as $ss(C_4AF + C_2F)$–will be formed instead of C_3A and C_4AF.

$$ss\,(C_4AF + C_2F) = 2.100Al_2O_3 + 1.702Fe_2O_3 \quad (5)$$

$$C_3S=4.071CaO-7.600SiO_2-4.479Al_2O_3-2.859Fe_2O_3-2.852SO_3-5.181CO_2 \quad (6)$$

The sum of the four major compounds will be less than 100 %, because the Bogue calculation does not account for minor phases and the incorporation of impurities into the major phases. The version of the Bogue calculations used in ASTM C 150 also does not correct for the amount of f-CaO present in the cement and so may overestimate the value for C_3S. Historically, another source of error arose in the past because ASTM C 150 included TiO_2 and P_2O_5 in the weight percentage of Al_2O_3. The value used for Al_2O_3 now does not include these trace elements; however differences could arise due to these elements when comparing past data.

Although based on many assumptions, the Bogue calculations have the advantage of being easy to calculate. When used appropriately, the results can be effectively used to compare cement properties. This is especially true when evaluating cement produced at a single plant where the raw materials and kiln conditions do not change radically over time. However, it should be borne in mind that the Bogue calculations give only the *potential* phase composition, and that the results are not always close to the true phase composition.

Incorporation of Substituent Elements in Clinker Phases

As has been shown, there are a number of ways of expressing the composition of a cement, and that it is necessary to differentiate between oxide and phase compositions. As mentioned previously, the Bogue equations do not take into account the incorporation small quantities of other elements in the major phases. In commercial clinker, the phases contain these impurities, and they will have some affect on cement performance. In general, impure phases are more reactive than the pure compounds. C_3S may contain up to 2 % of MgO, and will also incorporate Al_2O_3 and Fe_2O_3. Sulfur, sodium, and potassium, and many other elements have been identified within the C_3S phase in portland cement [6]. Alite is the mineral name given to impure C_3S, which includes C_3S in all commercial clinker. Impurities in the C_3S crystals, as well as the heating and cooling conditions in the kiln, lead to a variety of polymorphic forms of C_3S (polymorphs have essentially the same chemical composition, but a different crystal structure). Monoclinic and triclinic are two of the polymorphs of C_3S discussed in the literature. The mineral names of the clinker phases are shown in Table 3.

Belite, the mineral form of C_2S found in clinker, can incorporate aluminum, iron, and other elements. Like alite, belite exists in a number of polymorphic forms, and the β-polymorph of belite is the predominant form in commercial clinker. C_3A in clinker can contain iron and silicon. The alkalies, sodium and potassium, also influence the aluminate crystal structure, stabilizing the orthorhombic form (also called alkali-aluminate). The principal type of aluminate in clinker is referred to as cubic. Ferrite contains significant amounts of silicon and magnesium.

TABLE 3—Nomenclature for Major Clinker Phases

Name of Pure Compound	Mineral Name	Shorthand
Tricalcium silicate	alite	C_3S
Dicalcium silicate	belite	C_2S
Tricalcium aluminate	aluminate	C_3A
Tetracalcium aluminoferrite	ferrite	C_4AF

Clinker contains other impurities that originate from the raw materials or from the kiln fuel–TiO_2, P_2O_5, SrO, Mn_2O_3, and various sulfur compounds are examples. These do not usually have a significant impact on performance, but Jeknavorian and Hayden [7] have reported one example where excessive amounts of zinc caused severe retardation of setting and hardening. Chlorine, fluorine, and trace metals can also affect cement performance. Studies on impurities in clinker phases are numerous, and their effects on cement performance are generally secondary and too complex to be covered in this introduction.

Hydration Reactions

Hydration refers to the chemical reactions that take place when cement is mixed with water. Mathematical modeling of hydration and characterization of hydration products are active fields of research, and the reader is referred to a number of references for more detailed information [1,8–10]. There are a few reactions that should be reviewed in order to better understand the effects of composition on cement performance.

The calcium silicates react with water to form C-S-H, an amorphous calcium silicate hydrate gel that is primarily responsible for the strength development of hydraulic cement paste. For example, C_3S undergoes the following reaction

$$\begin{gathered} C_3S + (y+z)\,H_2O \rightarrow C_xSH_y + {}_zCH \\ \text{(alite + water} \rightarrow \text{C-S-H + calcium hydroxide)} \end{gathered} \tag{7}$$

where the composition of C-S-H is expressed as C_xSH_y and $x + z = 3$, but x, y, and z are not necessarily integers [3]. The actual composition of C-S-H may vary, and hyphens are used between the oxide abbreviations to show that the composition is indefinite. A byproduct of the hydration reaction is CH, calcium hydroxide. The C_2S also hydrates to form C-S-H and CH. The quantity of CH produced by C_2S hydration is about one third of the quantity from C_3S hydration.

The C_3A participates in the reactions affecting setting (early stiffening with no substantial development of compressive strength) and early strength gains. In the presence of gypsum, the following reaction occurs:

$$\begin{gathered} C_3S + 3C\overline{S}H_2 + 26H \rightarrow C_6A\overline{S}H_{32} \\ \text{(aluminate + gypsum + water} \rightarrow \text{ettringite)} \end{gathered} \tag{8}$$

The C_3A can also participate in many other reactions, with and without sulfate. All of the hydration reactions involving the clinker phases are exothermic, and the amount of heat generated is an important property of the cement. These hydration reactions and compounds are not specifically part of ASTM cement standards, but their chemistry underlies many test methods designed to predict the performance of cement: performance with respect to flow properties, strength, and durability.

The pozzolanic reaction, that is, the reaction of siliceous phases present in supplementary cementitious materials (such as fly ash, ground granulated blast furnace slag, silica fume, and calcined clays) with calcium hydroxide, is an approximation of the reactions of these materials in cementitious systems;

other compounds are present in these materials. The idealized reaction is:

$$\text{CH} + \text{S} \rightarrow \text{C-S-H} \qquad (9)$$

$$\text{(calcium hydroxide + silica} \rightarrow \text{calcium silicate hydrate)}$$

A benefit of using supplementary cementitious materials is that additional C-S-H is produced (if the hydration reactions continue), which can increase the strength and decrease the permeability of the mortar or concrete, both of which can improve concrete durability.

Contributions of the Major Cement Phases to Cement Performance

Because the properties of the major phases are generally similar in different portland cements, the approximate effect of changing the cement composition can be predicted and can provide a basis for control of performance of cement. Following is a summary of the properties of the four major phases.

C_3S

Alite hydrates rapidly and is responsible for early strength and early setting characteristics. Taylor [1] reports that approximately 70 % of the C_3S in portland cement will have reacted by 28 days, and virtually 100 % by one year. Portland cements with higher C_3S contents will generally show higher strengths through about seven days. C_3S hydration also generates more heat than C_2S hydration, and the heat of hydration of a cement is related to its C_3S content. ACI Committee Report 225 [11] includes a discussion on heat of hydration. In the USA, C_3S contents typically range from about 45 % to 65 %, by mass, with an average of about 55 % [12].

C_2S

Belite hydrates more slowly than alite, and contributes mostly to later age strength. Taylor [1] reports 30 % complete hydration at 28 days for C_2S, and 90 % completion at one year. C_2S, hydration generates less heat than C_3S, and also produces less calcium hydroxide. C_2S contents of U.S. portland cements range from about 8 to 28 %, by mass, with an average value of about 19 % [12].

C_3A

The aluminate phase influences setting and early strength development. Gypsum, or other forms of calcium sulfate, is needed to control the otherwise very rapid hydration of C3A and to avoid flash setting. The hydration reactions involving aluminate contribute significantly to heat of hydration of a cement. C_3A hydration products are the main participants in the reactions leading to sulfate attack [11]. C_3A contents of U.S. portland cements are controlled by the cement type; for most Type I cements they range between 5 % and 13 % and average about 9 %. For Type II cements they range from about 4 % to 8 %, and average about 6 %. For Type V cements they range from trace amounts of C_3A to about 5 %, with an average of about 4 % [12].

C_4AF

The contributions of C_4AF are not well-understood, but it may function similarly to C_3A. Cement color is influenced by the composition and amount of the iron-containing phase. A report by Chiesi, Myers, and Gartner suggests that the strength-producing capabilities of C_4AF can be enhanced with the use of chemical additives [14]. C_4AF contents tend to be higher when C_3A contents are lower because these phases are liquid at kiln temperatures and control of the amount of liquid phase is important to clinker burning and kiln operation. Thus, C_4AF contents range from 3 % to 13 %, averaging about 8 % to 10 % [12].

Chemical Requirements IN ASTM C 150

The chemical requirements for portland cement are summarized in Table 4. This table shows the compositional limits for ASTM Types I, II, III, IV, and V. ASTM C 150 also includes Types I-A, II-A, and III-A; the A indicates that an air-entraining admixture has been interground during the manufacturing process. The chemical requirements are the same for regular and air-entrained cements. In addition to air-entraining additives, ASTM C 150 permits the addition of water, calcium sulfate, limestone, and processing additions (usually grinding aids and pack-set inhibitors) conforming to ASTM Specification for Processing Additions for Use in the Manufacture of

TABLE 4—Standard Chemical Requirements for Portland Cements (From ASTM C 150, Table 1)

	Cement Type				
	I	II	III	IV	V
Al_2O_3, max, %	...	6.0	...	...	...
Fe_2O_3, max, %	...	6.0	...	6.5	...
MgO, max, %	6.0	6.0	6.0	6.0	6.0
SO_3, max, %					
when $C_3A \leq 8$ %	3.0	3.0	3.5	2.3	2.3
when $C_3A > 8$ %	3.5	...	4.5	...	...
LOI, max, %	3.0	3.0	3.0	2.5	3.0
Insol. Res., max, %	0.75	0.75	0.75	0.75	0.75
C_3S, max, %	...	...	...	35	...
C_2S, min, %	...	...	...	40	...
C_3A, max, %	...	8	15	7	5
$[C_4AF + 2(C_3A)]$ or $(C_4AF + C_2F)$, max, %	...	...	...	...	25

Hydraulic Cements (C 465). If requested, the manufacturer must supply in writing all relevant information about the air-entraining and processing additions used in processing.

The limitations in Table 4 are based on the oxides, as well as the potential phase composition. The different types of cement have different oxide limitations because the composition of the cement affects the resulting properties of concrete. Type I, a general purpose cement, has the fewest restrictions, whereas Types II, III, IV, and V have more limits. The chemical properties limited for all types of cement will be discussed first.

Limitations Common to All ASTM Types of Portland Cement

MgO

All ASTM types show a limit on MgO of 6 % by mass. The MgO is limited because of concern about disruptive expansion of concrete that may occur if free MgO hydrates. MgO content was one of two chemical properties limited in the original ASTM cement specification proposed in 1902. The maximum percentage was first set at 4 %, but was raised to 5 % in 1926 and to 6 % in 1976 [15].

Insoluble Residue

All ASTM types of cement are subject to a maximum limit on insoluble residue, the portion of the cement that cannot be dissolved in strong acid or alkaline solutions. Insoluble residue was first limited to 0.85 % in 1916 and was lowered to 0.75 % in 1941 [15]. The purpose of this limit is to preclude deleterious amounts of siliceous and argillaceous components from being present in portland cements. Insoluble residue may result from raw materials that did not combine completely in the burning process, or possibly from contamination during clinker reclaiming. It is usually a silicate or alumino-silicate material. All portland cements contain a small amount of insoluble residue, and the most common source is silicate impurities in the calcium sulfate and limestone added during the finish grinding. The 0.75 % limit on insoluble residue must be considered when determining the level of acceptable purity of calcium sulfate and limestone sources for portland-cement manufacture.

Loss on Ignition

Commonly abbreviated LOI, loss on ignition is the weight percentage lost when portland cement is heated at 950°C. Set at a maximum of 3.0% today (2. 5% for Type IV cement), LOI was added to the specification in 1917 to preclude the presence in the cement of deleterious amounts of carbonate minerals such as limestone and dolomite [15]. Water and carbon dioxide are the main contributors to LOI. The primary source of LOI in modern cements is from water combined in calcium sulfate dehydrate or gypsum ($CaSO_4{\cdot}2H_2O$) and carbon dioxide in limestone ($CaCO_3$). For example, a portland cement containing 5 % of gypsum has a theoretical LOI of about 1.5%, if only the combined water from gypsum is contributing to LOI. An additional 1.3 % LOI would result from 3 % addition of limestone ($CaCO_3$). In practice, some of the combined water from gypsum is lost through dehydration during the mill-grinding step. Additional moisture may be picked up from the atmosphere and from water sprays used to cool grinding mills.

Another source of LOI is moisture picked up from the clinker components during storage as well as during grinding. For example, free lime (f-CaO) not combined during burning is hygroscopic and readily absorbs water. During storage, particularly if stored outside and exposed to rain, f-CaO hydrates to form $Ca(OH)_2$ and it may absorb CO_2 from the atmosphere to form $CaCO_3$. These reactions contribute to a higher LOI. Other clinker components, particularly C_3A and C_3S are subject to prehydration during storage. These reactions can be quite complex and may affect the cement performance [16].

TABLE 5—Relationship Between C_3A and SO_3 (From ASTM C 150, Table 1)

Cement	C_3A, %	SO_3, %
Type III	> 8	4.5
Type I	> 8	3.5
Type II	≤ 8	3.0
Type IV	≤ 7	2.3
Type V	≤ 5	2.3

SO_3

All types of portland cement have a limit on the percentage of allowable SO_3, although the limits differ for Types II, III, and V. The SO_3 limit, one of the original two chemical requirements, was fixed at 1.75 % in 1904 [15]. The SO_3 content expresses the amount of sulfate in the cement. The SO_3 may be present in the one of the forms of calcium sulfate (gypsum, hemihydrate, anhydrite), but it may also be present in other forms. SO_3 may be present in clinker in the form of alkali sulfates will be included in the measured SO_3 content; it will reduce the amount of SO_3 that needs to be added. Sulfate is added to regulate the initial setting and hardening reactions that take place during hydration. The amount required is related to the fineness and composition of the cement, particularly the C_3A content as shown in Table 5. Type V portland cements have the lowest allowable SO_3 content, because of the low C_3A level in Type V cements. This same rationale holds for the SO_3 limits on Types I, II, and III, with Type III having the highest limit because it usually has a high C_3A content and high fineness. The SO_3 limits are all maxima; there are no restrictions on the minimum SO_3 contents. However, it will almost always be necessary to add some form of sulfate in order to meet the physical requirements in ASTM C 150.

Optimum SO_3

Careful examination of Table 1 in ASTM C 150 shows a note concerning the SO_3 limits. Note D allows the SO_3 content of cement to exceed the maximum in the table if it can be shown that the compressive strength will be improved at the higher SO_3 levels. The present method for determining optimum SO_3 is ASTM Test Method for Optimum SO_3 in Portland Cement (C 563), which defines the "optimum SO_3" based on compressive strength at 24-h. As a precaution against oversulfating, cements that exceed ASTM C 150 Table 1 limits must also be tested to show that the added SO_3 does not cause expansion greater than 0.020 % as demonstrated by ASTM Test Method for Expansion of Portland Cement Mortar Stored in Water (C 1038). The phrase "optimum SO_3" may be misleading, as different SO_3 contents may maximize early strength than maximize strength at later ages. SO_3 contents may also vary when optimizing other properties such as setting time and shrinkage. Furthermore, the optimum SO_3 content of a cement may be influenced by the use in concrete of chemical admixtures, supplementary cementitious materials, or heat treatment. Optimum SO_3 testing for a particular cement should be repeated if there is a change in manufacturing conditions or chemical composition of the clinker.

ASTM C 150 Chemical Requirements for Different Types of Cements

Changing the chemical composition causes a change in the properties of a cement. Examples of the phase composition, calculated by the Bogue method, for Types I through V are given in Table 6. Reference to Table 4 shows that there are only a few limitations on phase content. Even though the chemical requirements are not listed for each oxide, a manufacturer's certification will normally show the complete chemical analysis, along with the calculated phase composition. The results reported in the test certificate (also called mill report or mill certificate) are a good starting point for correlating cement properties with concrete performance characteristics.

Type I Portland Cement

Type I portland cement is to be used when the special properties specified for any other type are not required. According to ASTM C 150, Type I has no restrictions on the major oxides or the phase composition. A Type I cement could have a C_3S content of 20 or 70 %, but typically it will be 55 to 60 % C_3S by mass. Even though ASTM C 150 does not limit C_3S, there are theoretical and practical limitations. In addition, the chemical composition cannot be so extreme as to preclude meeting physical requirements such as strength and setting time, discussed in Chapter 39. Cement composition is influenced by the composition of available raw materials. For example, a plant with readily available high-purity limestone and a very expensive silica source may find it more economical to make a high C_3S cement (C_3S uses more calcium oxide, the major contribution of limestone to the clinker, than does C_2S). Economies of raw material sourcing must be balanced with manufacturing costs–it takes more energy to produce C_3S than C_2S. Therefore, cement compositions tend to be more alike throughout the country than might be expected. Chemical composition will also be affected by market demands. As the construction industry strives for higher early strengths, there may be greater demand for cements with higher C_3S and C_3A contents. When composition is changed to maximize one property, such as, for example, strength, changes to other properties may also occur.

TABLE 6—Average Phase Composition for ASTM C 150 Portland Cements [after 12]

	Cement Type				
	I	II	III	IV	V
C_3S, %	54	55	55	42	52
C_2S, %	18	19	17	32	22
C_3A, %	10	6	9	4	4
C_4AF, %	8	11	8	15	13

Type II Portland Cement

Type II portland cement has several restrictions on composition. Contents of Al_2O_3, and Fe_2O_3 are limited, in addition to limits on insoluble residue, LOI, and SO_3. Type II cement is designed for general use when moderate sulfate resistance or moderate heat of hydration is desired. The upper limits on Al_2O_3 and Fe_2O_3 restrict the amounts of C_3A and C_4AF that can be made, leading to lower heat of hydration. The upper limit of 8% on C_3A also has the purpose of reducing susceptibility to sulfate attack. As long as the physical requirements are met, Type II cements also meet the chemical requirements for Type I. Many cements meet both Type I and Type II requirements and are often referred to as Type I//II cements. In many areas of the country, Type II cements have replaced Type I as the most common cement.

Type III Portland Cement

Type III portland cement, for use when high early strength is desired, has no additional restrictions other than a 15 % C_3A maximum. While some Type III cements may have a C_3A content close to the maximum, it is also acceptable to have a very low C_3A content because there is no minimum specified in ASTM C 150. Type III is allowed higher SO_3 contents than Types I and II, even if they have the same C_3A level. This is because Type III cements generally have greater surface areas (to meet the higher strength requirements), and more SO_3 may be required to control the C_3A reactions in a finer-ground cement. Also, as mentioned in the discussion of optimum SO_3, the SO_3 limits in ASTM C 150, Table 1, can be exceeded if certain other conditions are met.

Type IV Portland Cement

The compositions of Type IV portland cements are restricted to less than 35 % C_3S, a minimum of 40 % C_2S, and the C_3A content can be no more than 7 %. Designed for low heat of hydration, Type IV is not readily available in the United States. The use of pozzolans and granulated blast-furnace slags, either as mineral admixtures or as components of blended cements, has reduced the demand for Type IV cement. It is possible that, in the future, there may be renewed interest in low-heat cements for applications such as roller-compacted concrete.

Type V Portland Cement

Type V portland cement, for use when high sulfate resistance is desired, has a maximum limit of 5 % C_3A and 25 % for the quantity $[C_4AF + 2(C_3A)]$. The hydration products of C_3A can react with sulfate to lead to deleterious expansion, but research has shown that limiting C_3A may not always provide sufficient protection against sulfate attack [17]. The C_3A limit for Type V in ASTM C 150 does not apply when the sulfate expansion limit, an optional physical requirement, is specified.

Optional Chemical Requirements

C_3A and C_3S

The optional chemical requirements in Table 2 of ASTM C 150 apply only when specifically requested. Optional limits on C_3A for Type III are 8 % for moderate sulfate resistance and 5 % for high sulfate resistance. For Type II, the sum of C_3S and C_3A may be limited to 58 % to help ensure moderate heat of hydration.

Low Alkali Limit

The optional requirement for low-alkali cement limits the equivalent alkali content, calculated as ($Na_2O + 0.658K_2O$), to 0.60 % maximum. A review of the history of the alkali limit is given by Frohnsdorff et al. [18]. Briefly, the alkali limit is intended to eliminate deleterious expansive action arising from alkali silica reaction (ASR). Discussed in more detail in other chapters, deleterious ASR occurs when cement alkalies and reactive silica components in aggregate form a gel that leads to concrete expansion and cracking. It should be noted that an alkali content of below 0.60 % is not always sufficient to prevent deleterious expansion of concrete containing reactive aggregates.

Blended Hydraulic Cements

Blended hydraulic cements consist of two or more inorganic constituents (one of which is normally portland cement or finely-ground portland-cement clinker) that, separately or in combination, contribute to the strength-gaining properties of the cement. The most common non-portland cement (or portland-cement clinker) materials used in blended cements are the supplementary cementitious materials blast-furnace slag and fly ash. Natural pozzolans and silica fume (also called microsilica) are examples of other materials that may be used in blended cements. Blended cements may be made by intergrinding the constituents with portland cement clinker and calcium sulfate, or by blending the constituents with portland cement.

A pozzolan is a material that has little or no hydraulic activity of its own, but it acts as a hydraulic cement when mixed with water and a source of CaO or $Ca(OH)_2$. In blended cements, portland cement is the source of CaO for the pozzolanic reactions ($Ca(OH)_2$ is produced when portland cement hydrates). Pozzolans are high in SiO_2 and may also contain significant amounts of Al_2O_3. Fly ash, microsilica, and natural pozzolans are examples of pozzolanic materials. Blast-furnace slag may have latent hydraulic activity as well as pozzolanic properties.

ASTM C 595 Specification for Blended Hydraulic Cements

There are five major classifications of blended cements, and some of these classes are further subdivided, based on the percentage of portland cement in the mixture. The five main types are:

- portland blast-furnace slag cement (Type IS),
- portland-pozzolan cement (Type IP and Type P),
- slag cement (Type S),
- pozzolan-modified portland cement [Type I (PM)], and
- slag-modified portland cement [Type I(SM)].

There are only a few chemical requirements for blended cements, and the rationales for most of these requirements are similar to those requirements for portland cements in ASTM C 150. Table 7 summarizes the chemical requirements. The MgO content is limited to 5.0 % for all portland-pozzolan and pozzolan-modified portland cements, but there is no limit on MgO content for slag cements. The SO_3 limits for all types of blended cements may be exceeded if it can be demonstrated that the optimum SO_3 content is higher than the limit. Insoluble residue is limited to 1.0 % for slag cements, but there is no limit for pozzolan-containing cements. Loss on ignition varies from 3.0 to 5.0 %. The source of LOI for blended cements is the same as for portland cements, although pozzolans such as fly ash may contain unburned carbon that is measured in the LOI determination (the carbon can affect the efficiency of air-entraining admixtures). There is no total or equivalent alkali limit for blended cements.

TABLE 7—Chemical Requirements for Blended Hydraulic Cements (From ASTM C 595, Table 1)

	Cement Type		
	I(SM) I(SM)-A IS, IS-A	S, SA	I(PM) I(PM)-A P, PA, IP, IP-A
MgO, max, %	...	...	5.0
SO_3, max, %	3.0	4.0	4.0
Sulfide sulfur, max, %	2.0	2.0	...
Insoluble residue, max, %	1.0	1.0	...
Loss on ignition, max, %	3.0	4.0	5.0

The chemical composition of a pozzolanic material cannot always be directly related to the performance of concrete, but examination and tracking of chemical properties can provide a means to check product uniformity. In addition to the chemical requirements in ASTM Specification for Blended Hydraulic Cements (C 595), the standards dealing with the individual types of finely-divided mineral admixtures (C 618, C 989, C 1240) provide useful guidelines about the composition of these materials. These are summarized in a subsequent section, "Chemical Composition of Pozzolanic Materials Used as Mineral Admixtures in Concrete."

Performance-Based Specification for Hydraulic Cements

ASTM C 1157, Standard Performance Specification for Hydraulic Cements, is a relatively new specification for cements for general construction. It provides for portland or blended cements that meet performance requirements, rather than prescriptive (usually chemical) requirements. The first edition of ASTM C 1157, approved in 1992, covered only blended cements. However, at the time, a parallel effort was underway in ASTM Committee C01 to develop a performance specification for portland cements with the requirements being similar to those for blended cements in C 1157. It was generally agreed that the redundancy in having two, almost identical performance specifications was unwarranted and, in 1998, C 1157 was modified to include portland cements. The development of a performance-based specification is a logical step in the evolution of specifications: why indirectly specify a property of a material through prescriptive requirements when you can directly specify the performance required? However, at present not all performance requirements for cement can be easily measured by a test, and performance testing is usually more time-consuming. For these reasons, and because of the relatively conservative nature of the construction industry, ASTM C 150 and C 595 continue to be used much more extensively than C 1157.

Six basic cement types are considered in ASTM C 1157:

- general-purpose hydraulic cement (Type GU),
- high-early strength cement (Type HE),

- moderate- and high-sulfate resistance cements (Type MS and Type HS), and
- moderate- and low-heat of hydration cements (Type MH and Type LH).

The physical requirements for these cements are largely similar to their counterparts in C 150 and C 595. A few requirements will be discussed here. Although requirements for chemical composition, fineness and air content are not included, these characteristics are required to be determined and reported for informational purposes.

Default minimum compressive strength requirements are listed in Table 1 of C 1157; however other options are available for specifying a different (higher) minimum strength and for specifying a strength range (that is, a maximum and a minimum strength) at a particular age. C 109/C109M (Test Method for Compressive Strength of Hydraulic Cement Mortars) is used to measure the compressive strength.

For Types LH and MH, heat of hydration is measured by C 186 (Test Method for Heat of Hydration of Hydraulic Cement) while, for Types MS and HS, sulfate resistance is determined by C 1012 (Test Method for Length Change of Hydraulic Cement Mortars Exposed to a Sulfate Solution).

An Option R, low reactivity with alkali-reactive aggregates, is also included, and determined by a modified version of C 227 [Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar Bar Method)]. The modification consists of using crushed borosilicate glass as a model aggregate.

Chemical Composition of Pozzolanic Materials Used as Mineral Admixtures in Concrete

Fly Ash and Natural Pozzolans

ASTM Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete (C 618) gives the requirements for fly ash and natural pozzolans for use in concrete. Class N, raw or calcined natural pozzolans, include siliceous, or siliceous and aluminous, materials such as diatomaceous earths, opaline cherts and shales, tuffs and volcanic ashes or pumicites, and some calcined shales and clays. The sum of SiO_2 and Al_2O_3 must equal at least 70 %, and there are also limits on SO_3, moisture, and LOI. Class F fly ash, typically produced from the burning of anthracite or bituminous coal, must also meet a 70 % minimum sum of SiO_2, Al_2O_3, and Fe_2O_3. For Class C fly ash, typically produced from burning lignite or sub-bituminous coals, the sum of these same three oxides must equal 50 % or more. Class C fly ashes normally contain CaO and have some cementitious properties in addition to being pozzolanic. Helmuth has summarized the chemical composition and properties of a variety of fly ashes [19].

Ground Granulated Blast-Furnace Slag

Blast-furnace slag is the nonmetallic product formed during iron production, consisting mostly of silicates and aluminosilicates of calcium. The major oxides, SiO_2, Al_2O_3, CaO, and MgO, constitute 95 % or more of the total oxides [20]. If water-quenched, blast-furnace slag can have a glass content of greater than 95 %; pelletized blast-furnace slag, which is cooled more slowly, has a lower glass content. Air-cooled slag has little hydraulic activity, but may be used as aggregate. Attempts to relate the reactivity of granulated blast-furnace slag to chemical composition, such as the ratio $(CaO + MgO + Al_2O_3)/SiO_2$, have not been fully successful, but these constraints are included in some specifications. ASTM Specification for Ground Iron Blast-Furnace Slag for Use in Concrete and Mortars (C 989) uses the slag-activity index based on actual compressive strength test results to characterize slags.

Silica Fume

Condensed silica fume, also called microsilica, is covered by ASTM C 1240, Standard Specification for Use of Silica Fume for Use as a Mineral Admixture in Hydraulic-Cement Concrete, Mortar, and Grout. Silica fume is a by-product from the reduction of quartz (or quartz and iron) with coal to produce silicon and ferro-silicon alloys. Silica fume condenses from the gaseous phase, and the particles are spherical, amorphous, and have a very high specific surface area [21]. Silica fume from silicon alloy production usually contains more than 90 % SiO_2, and ferrosilicon alloy production produces silica fume with varying amounts of Fe_2O_3. Small amounts of other oxides may also be present. Silica fume is a very reactive pozzolan, because the very fine SiO_2 particles quickly react with $Ca(OH)_2$ and water to form C-S-H.

Methods for the Chemical Analysis of Hydraulic Cements

ASTM Test Methods for Chemical Analysis of Hydraulic Cement (C 114) describes specific chemical analysis procedures for both portland cements and blended hydraulic cements. The scope of ASTM C 114 states that any method may be used for analysis of hydraulic cement, as long as it can be demonstrated that the method achieves the required levels of precision and bias. ASTM C 114 procedures for analyzing cement are separated into Reference Test Methods and Alternative Test Methods. Reference test methods are "long accepted chemical test methods which provide a reasonably well-integrated basic scheme of analysis for hydraulic cements." Reference test methods can be performed in any reasonably-equipped chemical laboratory and do not require expensive analytical instrumentation. Generally, satisfactory results can only be obtained by experienced analysts, and ASTM C 114 requires that individual analysts demonstrate their ability to achieve acceptable precision and bias when using the reference test methods.

TABLE 8—Maximum Permissible Variations in Results (From ASTM C 114, Table 1)

Component	Maximum Difference Between Duplicates	Max Difference of the Average of Duplicates from SRM Values (+/−)
SiO_2	0.16	0.2
Al_2O_3	0.20	0.2
Fe_2O_3	0.10	0.10
CaO	0.20	0.3
MgO	0.16	0.2
SO_3	0.10	0.1
LOI	0.10	0.10
Na_2O	0.03	0.05
K_2O	0.03	0.05

Demonstration of Precision and Bias

The competency of an individual analyst is determined by comparing the analyst's duplicate results for an SRM (Standard Reference Material) cement to the maximum permissible variation in results shown in Table 8. Table 8 has been adapted from Table 1 in ASTM C 114, which provides information on oxides in addition to the ones included in Table 8 of this report. An SRM cement is prepared and certified by the National Institute of Standards Technology (NIST, formerly NBS) or other acceptable reference material as defined in ASTM C 114. Cement SRMs cover a range of compositions, and certified values are provided. In addition to being used to demonstrate competency of analysts and test methods, SRMs are used as calibration standards for instrumental methods.

Table 8 also provides a convenient means assessing the acceptability of optional test methods (also called rapid methods). A scheme for qualifying these test methods was developed and became part of ASTM C 114 in 1977. Briefly, the qualification procedure involves analysis of at least seven SRM samples; two rounds of tests on nonconsecutive days are required. Six of the seven differences between duplicates obtained for any single component shall not exceed the limits in Column 2 of Table 8. Also, the average of the duplicates must not exceed the limits in Column 3, again for six of the seven SRMs. Test methods must be requalified at least every two years, or when there is substantial evidence that the test method is not performing in accordance with Table 1 in ASTM C 114. Such evidence may come from a comparison of results with the average in the Cement and Concrete Reference Laboratory (CCRL) Proficiency Sample program [22]. Many companies also conduct their own interlaboratory sample exchange programs to monitor chemical analysis test procedures. Additional details and restrictions on the qualification procedure are given in ASTM C 114 in the section entitled Performance Requirements for Rapid Test Methods. A recent analysis of CCRL data [23] may provide the basis for updating the precision and bias statements in Table 1 of ASTM C 114.

Instrumental Methods for Cement Analysis

Table 1 in ASTM C 114 has facilitated the application of modern instrumental methods to cement analysis, and a manufacturer can use any qualified method, as long as the procedure used is noted on the manufacturer's certification. Examples of test methods used successfully to analyze hydraulic cements are discussed in *ASTM STP 985* [24]. Methods included are X-ray fluorescence, atomic absorption spectrophotometry, and a spectrophotometric scheme. The most common of these methods will be discussed further.

X-Ray Fluorescence (XRF)

Both wavelength and energy-dispersive X-ray methods have been used successfully to analyze portland cements, but wavelength is the more popular technique in cement plant quality control laboratories. XRF is well-suited to analysis in a cement plant because it can be used to obtain chemical analysis of quarry and imported raw materials, clinker, and portland cement. Analysis time is greatly reduced compared to classical "wet" methods, and varying degrees of automation are now in use. Three methods are used for sample preparation: pressed powder, fused pellets, and fusion followed by grinding. ASTM C 114 allows the use of any method, as long as acceptable precision and bias can be demonstrated.

Other Rapid Methods

Atomic absorption involves dissolution of the cement prior to elemental analysis. Used successfully in many laboratories, it is subject to error for the major elements because of the number of dilution steps that are required. Inductively-coupled plasma (ICP) spectrometry can also be used for complete analysis. Specialized techniques, such as ion chromatography, can also be used for selected elements. For more information on analysis of anhydrous cement, refer to the American Ceramic Society's series, *Cements Research Progress*, most recently printed in 1997 [25].

Selective Dissolution

Selective dissolution covers a variety of procedures that attempt to separate a component or phase from the composite. Methods for free CaO fall into this classification, as do procedures such as maleic acid/methanol (M/M) extraction. M/M extraction, or a variation using salicylic acid, can be used to remove the calcium silicates [26–30]. This then concentrates the aluminate, ferrite, periclase, and calcium sulfate phases. These techniques are sometimes used as precursors to other methods of analysis such as quantitative X-ray diffraction.

Significance of Test Results

Table 8 provides a means for the user to evaluate the significance of test results. When comparing two cements, Table 8 can be used as a guideline. For example, if the SO_3 content for Cement A is 2.70 % and that for Cement B is 2.75 %, examination of Table 8 shows that a difference of 0.10 % is allowed for duplicate analyses of the same sample. This provides a good indication that the SO_3 contents of Cements A and B are not significantly different. This type of examination may be helpful when determining if a cement that is close to a specification limit should be rejected. The values in Table 8 apply only to within-laboratory variation, but between-laboratory variation can be inferred because all laboratories are comparing their results to reference standards.

Looking at another example, it is possible to get an approximation of the variation that can occur between two laboratories, both of which have demonstrated acceptable analyses of SRM cements. If Laboratory A shows a consistent bias of +0.05 % in the analysis of Na_2O, and Laboratory B shows a consistent bias of −0.05 %, they may differ by as much as 0.10 %. Unlike the previous SO_3 example, a difference of 0.10 % in Na_2O content is not trivial, especially if the cement is near the optional 0.60 % alkali limit. Fortunately, it is unlikely that laboratories will show a consistent bias at the maximum allowable range. However, even a bias of 0.02 or 0.03 % can be important for plants operating near the limit for the minor oxides.

The preceding examples show that bias in test results can have a significant impact on the minor oxides, but testing variations for the major oxides can also influence the potential phase composition calculated by the Bogue equations. For example, errors or bias of a few tenths of a percent in measuring SiO_2, Al_2O_3, and CaO can cause differences of more than 5 % in the calculated C_3S content. This example illustrates the need for cement manufacturers, and others who test cement, to establish a quality assurance program for analytical procedures. One should use caution when comparing the composition of two or more cements, especially when the chemical analyses were not performed in the same laboratory.

Quantitative Phase Analysis

Currently, there are several task groups working within ASTM Subcommittee C01.23 on Compositional Analysis of Hydraulic Cements to develop standard test methods for measuring the phase compositions of cements. The impetus for this effort comes from the growing shift away from prescription to performance-based specifications. In addition, there is a desire to correlate cement properties with performance in concrete, requiring a more definitive characterization of cement in terms of the phases present and their distribution among the particles of different shapes and sizes.

Microscopical Techniques

Light microscopy has long been used to quantitatively determine the mineralogical composition of geological samples, and the technique can be applied to the measurement of clinker phase content. ASTM C 1356, Test Method for Quantitative Determination of Phases in Portland Cement Clinker by Microscopical Point Count Procedure, was approved in 1996. Methods for quantitative clinker microscopy with the light microscope have been reviewed by Campbell and Galehouse [31]. Determination of phase content by light microscopy is considered a direct method since phases are identified and counted to determine the volume percentage. The disadvantages of light microscopy include (1) it is difficult to obtain a representative sample, (2) it is time-consuming to count at least 2000 to 3000 points to get a statistical sampling, (3) the method is subject to errors of misidentification of phases, (4) the precision of the method can vary with the clinker microstructure (that is, size of crystals), and (5) the method may not be easily applied to ground cement.

The use of light microscopy to evaluate clinker reactivity and predict strength performance of portland cement has been the subject of numerous papers reported in the proceedings of the International Cement Microscopy Association (ICMA). One procedure is the Ono Method [32], named after Dr. Yoshio Ono from Japan, where a small amount of ground clinker or cement powder is evaluated to determine kiln burning and cooling conditions. The crystal observations can then be correlated to potential strength gain. This type of compositional analysis is not presently covered by ASTM standards, but it is an example of a method that might be investigated in the future.

Scanning electron microscopy (SEM) and other electronic imaging techniques have been successfully applied to characterizing portland-cement clinker, portland cements, and blended cements [33]. The SEM, if equipped with an X-ray analyzer, can be used to perform chemical analyses of individual crystals. X-ray mapping produces an image of the elemental composition, and also shows the distribution of the elements throughout the crystal structure.

Quantitative X-Ray Diffraction (QXRD) Analysis

X-ray diffraction has been used for many years to characterize cements and is useful for identifying phases in cement. It can also be used for quantitative phase analysis, but the difficulties are numerous. The status of QXRD analysis applied to cement and clinker is discussed by Struble [34] in a report prepared for a 1991 ASTM symposium on the characterization of hydraulic cements. Struble describes the problems associated with QXRD, in general, and the specific difficulties associated with analysis of clinker and cement. ASTM C 1365, Determination of the Proportion of Phases in Portland Cement and Portland Cement Clinker Using X-ray Diffraction Analysis, was originally approved in 1998 and covers the determination of C_3A, C_4AF, and MgO contents. Work on other phases is progressing.

Even though standard ASTM test methods for QXRD have not yet been finalized for all cement and clinker phases, the future is promising. Advances in X-ray diffraction automation and computer software have made it possible to collect and analyze large amounts of data, and these improvements will help to overcome some of the challenges inherent in the technique. Experienced diffractionists have successfully applied QXRD analysis to cement and clinker, reporting accuracy levels of better than 5 % for C_3S and 2 % for C_3A [1]. Some laboratories may find that good results can be obtained by combining microscopic and XRD techniques. It is very difficult to differentiate aluminate from ferrite in some clinkers, and current procedures for QXRD may already be capable of achieving greater accuracy than light microscopy. Conversely, the calcium silicates are usually easily identified by microscopy and an experienced microscopist may be able to achieve improved accuracy levels compared to present QXRD methods. Combined techniques of XRD and microscopy have also been used to successfully characterize periclase (free MgO).

Other Compositional Considerations

Carbonate (Limestone)

Limestone added to portland cements during finish grinding has been a common practice in other countries for many years, but it is relatively new in the United States. A recent PCA report discusses the effects of limestone in small amounts, up to 5 %, on the properties of cement and concrete [34]. An appendix to ASTM C 114 lists six methods for determining carbonate content of hydraulic cements. Included are split loss on ignition, thermogravimetric analysis (TGA), decomposition with HCl (refers to ASTM C 25, Chemical Analysis of Limestone, Quicklime, and Hydrated Lime), X-ray fluorescence spectroscopy (XRF), combustion by induction furnace, and combustion gravimetric method (refers to ASTM E 350, Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron). A brief synopsis of each method is given, along with cautions and limitations for the methods. (The methods generally determine the CO_2 content of cement, and back calculate to determine the carbonate content of the cement. Since the CO_2 contents of different carbonate (limestone) sources used in cements vary, when carbonate is used as a component of portland cement, ASTM C 150 requires the CO_2 content of the limestone to be reported on mill test reports.)

Calcium Sulfate

The SO_3 content was discussed in the section on ASTM C 150, but it is also important to determine the form of the sulfate. For example, when natural gypsum is added to clinker and ground in the finish mill, enough heat may be generated to dehydrate the gypsum to some degree. Natural gypsum may also contain some anhydrite, and there is an increasing use of calcium sulfate byproducts as sources of SO_3 in cement manufacture. The form and amount of the sulfate phases can affect the hydration reactions. As previously mentioned, gypsum and other sulfates can also react during storage, sometimes leading to cement flowability problems. There is interest in sulfate form and amount because reports during the last several years have shown that these parameters can affect a cement's interaction with chemical admixtures, particularly water reducers [36].

Calcium sulfates can be identified by X-ray diffraction, light and electron microscopy, and by differential thermal analysis/thermogravimetric analysis (DTA/TGA) methods. There is a need for reexamination of these techniques and the development of more accurate procedures for the quantitative measurement of the calcium sulfate phases.

Alkali Sulfates

In addition to forms of calcium sulfate, cements may contain alkali sulfates. The most commonly occurring alkali sulfates are arcanite, apthitalite, and calcium langbeinite. During the course of normal cement hydration reactions with water, these alkali sulfates can contribute to set control. However, hydration of alkali sulfates during cement storage can lead to lumping and flowability problems. Alkali sulfates can affect the strength characteristics of cement, reportedly improving early strengths but giving lowered 28-day and later strengths. Changes in cement alkali levels have also been reported to affect the effectiveness of certain air-entraining admixtures [36].

Trace Elements

A review of the literature on cement composition shows hundreds of references dealing with trace elements (primarily trace metals, but chlorine, fluorine, and rare earth metals are also included). Covered are effects on burnability of cement raw materials, effects on kiln refractory materials, effects on clinker reactivity and cement performance, and methods for measuring trace elements. Interest in recent years has increased because of the environmental concerns. A survey on effects of trace elements in portland cements and cement kiln dust has been published by the Portland Cement Association [37]. This reference also provides information about the sources and effects of trace elements. Of particular interest is the increased attention being given to trace elements as a result of the growing practice of using waste materials as both raw materials and fuels in the clinker manufacturing process. Barger [38] presented a thorough discussion on the utilization of waste solvent fuels, including a description of trace metal, hydrocarbon, and chloride balances. Analysis of cements produced with waste solvent fuels, using XRD and light microscopy techniques, showed no detrimental effects.

Testing for trace elements has brought a number of techniques to the cement quality control laboratory. Discussion of these methods is beyond the scope of this chapter, but they include atomic absorption (AA), inductively-coupled plasma (ICP), gas chromatography (GC), mass spectroscopy, and many other instrumental techniques for analysis of metals, halides, hydrocarbons, and other organics. Improved procedures for rapid determination of heating values for fuels are also being developed.

Concerns of the User

Cement manufacturers may be interested in pursuing alternative raw materials and fuels for economic reasons, but maintaining product uniformity and quality should be the prime concern of the knowledgeable user. Significant changes in raw materials or fuels may affect properties like alkali levels, sulfate form, and even reactivity of the major phases [39]. Also, physical properties such as setting time, bleeding rate, strength gain, and cement-admixture interactions may be affected. Data is just now beginning to be published about the effects of alternative materials on cement quality, so users should maintain close communication with their suppliers. Consideration should also be given, when developing ASTM standards, to ensure that specifications address all relevant properties of cement, both chemical and physical.

Performance Standards Versus Prescriptive Standards

Improved methods are needed to predict the performance of hydraulic cement in concrete, and many subcommittee and task group activities in ASTM are working on this issue. Ideally, there would be no need to specify limits on chemical composition because there would be rapid tests to correctly predict performance. However, all of the properties that contribute to the performance of hydraulic cement have yet to be identified, and adequate performance tests have not been developed for the properties we know to be significant. Further, even accelerated tests take up to two weeks or more to produce results, especially with respect to durability. It is likely that the standards process will continue to evolve, and prescriptive limits on chemical composition will be modified or eliminated as better performance test methods are developed. Is it conceivable that the matter will go full circle, and tomorrow's performance tests will be replaced with better prescriptions? If a hydraulic cement could be adequately characterized, chemically and physically, would it be necessary to run compressive strength or setting time tests? Or, as another example, if we fully understood the mechanisms of sulfate attack, would it be necessary to subject mortar bars to accelerated curing regimes? This is the approach being explored by a NIST-led research consortium, the Virtual Cement and Concrete Technical Laboratory (VCCTL), which hopes to use careful and complete characterization of cements to predict performance of concretes using computer modeling [40].

The composition of hydraulic cements may become more complex as environmental issues such as the concern for global warming dictate increased use of materials other than portland cement and the pozzolanic materials we are familiar with today. A necessary part of the evolution toward improved ASTM standards is the development of better methods to characterize the properties of hydraulic cements, leading to more useful predictive models.

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Mixing and Curing Water for Concrete

James S. Pierce[1]

Preface

WALTER J. MCCOY, THEN DIRECTOR OF RESEARCH for Lehigh Portland Cement Company, wrote the first version of this chapter for *ASTM STP 169.* For *ASTM STP 169A,* Mr. McCoy revised his chapter and added information on typical municipal water analyses, tolerable concentrations of impurities, the effects of sugar in mixing water, and the effects of water hardness on concrete air content. He also added several new references. For *ASTM STP 169B,* Mr. McCoy, then Director of Cement Technology for Master Builders, made only minor changes to the *ASTM STP 169A* version of the chapter. This current version is essentially Mr. McCoy's (now deceased) chapter with limited updating. There has been very little new technology published regarding mixing and curing water for concrete. The *ASTM STP 169D* edition as prepared does, however, reflect recent standards developments promulgated by ASTM Subcommittee C09.40 and Committee C09.

Introduction

This chapter is concerned primarily with the significance of quality tests of various types of waters for mixing and curing concrete and makes no attempt to include the effect of the quantity of mixing water. The quality of water is important because poor-quality water may adversely affect the time of setting, the strength development, or cause staining. Almost all natural waters, fresh waters, and waters treated for municipal use are satisfactory as mixing water for concrete if they have no pronounced odor or taste. Because of this, very little attention is usually given to the water used in concrete, a practice that is in contrast to the frequent checking of the admixture, cement, and aggregate components of the concrete mixture. In fact, most of the references appear to be outdated, but they still represent the bases for modern concrete technology with regard to water for mixing and curing.

Recent environmental regulations place restrictions on discharging wash water from mixer drums and provided the impetus to develop ASTM C 1602/C 1602M, Specification for Mixing Water Used in the Production of Hydraulic Cement Concrete, and this standard is referenced in discussions on mixing water.

Mixing Water

A popular criterion as to the suitability of water for mixing concrete is the classical expression, "If water is fit to drink it is all right for making concrete." This does not appear to be the best basis for evaluation, since some waters containing small amounts of sugars or citrate flavoring would be suitable for drinking but not mixing concrete [1], and, conversely, water suitable for making concrete may not necessarily be fit for drinking [2]. In an attempt to be more realistic, some concrete specifications writers attempt to ensure that water used in making concrete is suitable by requiring that it be clean and free from deleterious materials. Some specifications require that if the water is not obtained from a source proven to be satisfactory, the strength of concrete or mortar made with the questionable water should be compared with similar concrete or mortar made with water known to be suitable. The U.S. Army Corps of Engineers, in addition to a general description of acceptable water requirements [3], also states that if the pH of water is between 6.0 and 8.0 and the water is free from organic matter, it may be regarded as safe for use in mixing concrete. (An exception to this is the case where potassium or sodium salts or other natural salts are present in excessive amounts.) This standard also states that if water is of questionable quality before the water is judged to be acceptable, it should be tested in mortar cubes for which the average 7- and 28-day compressive strengths must equal at least 90 % of those of companion test specimens made with distilled water [4]. Other than comparative tests of this type, no special test has been developed to determine the quality of mixing water, and hence it is difficult to judge the fitness of water for use in concrete. However, due to environmental concerns, Specification C 1602/C 1602M was developed and is available for mixing water. This specification provides compositional and performance requirements for mixing water and may be used for nonpotable sources and for water from concrete production operations.

Guidance on mixing water quality is available using American Association of State Highway and Transportation Officials Designation T26. In ASTM C 94, Specification C 1602/C 1602M is cited for water requirements. When water quality is questionable, service records of concrete made with the questionable water should be examined. If service records are not available or not conclusive, then the water quality should be clarified by comparing compressive strengths and

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times of setting of specimens made with the water in question and with distilled or 100 % potable water. ASTM C 1602/C 1602M requires compressive strengths to be a minimum of 90 % of the strength of specimens made with distilled or 100 % potable water, and the time of setting in the test mortar should not be more than 1 h quicker nor more than 1 1/2 h slower than the time of setting when distilled or 100 % potable water is used.

This specification also permits the use of wash water from mixer washout operations for mixing water, as long as it meets the compressive strength and time of setting requirements. Specification C 1602/C 1602M also cites optional chemical limits that may be used when appropriate for the construction. These limits are being re-evaluated in light of the need to use more wash water.

Caution must be exercised when groundwater supplies are tapped for mixing water supplies. The rule of thumb about being potable is still only a guide, and before critical concrete operations are undertaken, comparative tests for strength and time of setting should be completed to ensure specification compliance. Specification C 1602/C 1602M prescribes testing frequency. The density of the water is used as an indicator and is determined using C 1603, Test Method for Measurement of Solids in Water.

The two principal questions regarding mixing water quality appear to be: "How do impurities in the water affect the concrete?" and "What degree of impurity is permissible?" The following discussion is a summary of available information on these two items.

Effects of Impurities in Mixing Water

The most extensive series of tests on this subject was conducted by Abrams [5]. Approximately 6000 mortar and concrete specimens representing 68 different water samples were tested in this investigation. Among the waters tested were sea and alkali waters, bog waters, mine and mineral waters, and waters containing sewage and solutions of salt. Tests with fresh waters and distilled water were included for comparative purposes. Time of setting tests and cement and concrete strength tests from 3 days to 2.33 years were conducted for each of the various water samples. Some of the more significant conclusions based on these data are as follows:

1. The times of setting of portland cement mixtures containing impure mixing waters were about the same as those observed with the use of clean fresh waters with only a few exceptions. In most instances, the waters giving low relative compressive strength of concrete caused slow setting, but, generally speaking, the tests showed that time of setting is not a satisfactory test for suitability of a water for mixing concrete.
2. None of the waters caused unsoundness of the neat portland cement pat when tested over boiling water.
3. In spite of the wide variation in the origin and type of waters used, most of the samples gave good results in concrete due to the fact that the quantities of injurious impurities present were quite small.
4. The quality of mixing water is best measured by the ratio of the 28-day concrete or mortar strength to that of similar mixtures made with pure water. Waters giving strength ratios that are below 85 %, in general, should be considered unsatisfactory.
5. Neither odor nor color is an indication of quality of water for mixing concrete. Waters that were most unpromising in appearance gave good results. Distilled waters gave concrete strengths essentially the same as other fresh waters.
6. Based on a minimum strength-ratio of 85 % as compared to that observed with pure water, the following samples were found to be unsuitable for mixing concrete: acid water, lime soak water from tannery waste, carbonated mineral water discharged from galvanizing plants, water containing over 3 % of sodium chloride or 3.5 % of sulfates, and water containing sugar or similar compounds. The concentration of total dissolved solids in these waters was over 6000 ppm except for the highly carbonated water, which contained 2140 ppm total solids. These data support one of the principal reasons for the general suitability of drinking water, for few municipal waters contain as much as 2000 ppm of dissolved solids and most contain far less than 1000 ppm. Very few natural waters other than seawater contain more than 5000 ppm of dissolved solids [6].
7. Based on the minimum strength-ratio of 85 %, the following waters were found to be suitable for mixing concrete: bog and marsh water, waters with a maximum concentration of 1 % SO_4, seawater (but not for reinforced concrete), alkali water with a maximum of 0.15 % Na_2SO_4 or NaCl, water from coal and gypsum mines, and wastewater from slaughterhouses, breweries, gas plants, and paint and soap factories.

Many of the specifications for water for mixing concrete, especially those requiring that it be potable, would have excluded nearly all of the aforementioned waters, but contrary to this rather general opinion, the test data show that the use of many of the polluted types of water did not result in any appreciable detrimental effect to the concrete. The important question is not whether impurities are present, but do impurities occur in injurious quantities? It should be noted that the conclusions on suitable waters cited earlier were based entirely on tests of specific samples from the indicated sources, and it should not be assumed that all waters of the type described would be innocuous when used as mixing water.

Typical analyses of municipal water supplies as reported by the U.S. Geological Survey are given in Table 1. Although dated, there is no reason to believe that typical analyses have changed much from those in Table 1. Collins [7] reported that these analyses represent public water supplies used by about 45 % of the cities of the United States that have a population of more than 20 000. These analyses indicate they would be acceptable sources for mixing water. However, when investigating a municipal source, analyses for several periodic tests should be examined to determine if comparisons with distilled water are needed. Such an examination will also provide a perspective on the variability of inorganic and organic compounds present. The presence of certain compounds may not create a serious deleterious effect, but the variability may cause varying effects on the efficiency of air-entraining admixtures, the time of setting, and the strength development.

A concrete manual [8] published in Denmark in 1944 points out that humic acid and other organic acids should be avoided because their presence means a danger to the stability of concrete. Most organic compounds will have an effect on time of setting and comparative tests should be conducted to evaluate the effect.

An article appearing in a 1947 British publication [9] discusses the harmful effects of using acid waters in concrete and claims that the harmful effects of organic acid are not evident

TABLE 1—Typical Analyses of City Water Supplies (PPM)[a]

Analysis Number	1	3	5	6	7
Silica (SiO_2)	2.4	12.0	10.0	9.4	22.0
Iron (Fe)	0.14	0.02	0.09	0.2	0.08
Calcium (Ca)	5.8	36.0	92.0	96.0	3.0
Magnesium (Mg)	1.4	8.1	34.0	27.0	2.4
Sodium (Na)	1.7	6.5	8.2	183.0	215.0
Potassium (K)	0.7	1.2	1.4	18.0	9.8
Bicarbonate (HCO_3)	14.0	119.0	339.0	334.0	549.0
Sulfate (SO_4)	9.7	22.0	84.0	121.0	11.0
Chloride (Cl)	2.0	13.0	9.6	280.0	22.0
Nitrate (NO_3)	0.54	0.1	13.0	0.2	0.52
Total dissolved solids	31.0	165.0	434.0	983.0	564.0

[a] Taken from Collins [7].

as soon as those of mineral acids, while deleterious salts have a greater effect on early-age strengths than at later ages.

Kleinlogel [10] stated that mixing water should not contain humus, peat fiber, coal particles, sulfur, or industrial wastes containing fat or acid.

An article in Ref 11 contains a tabulation of maximum limits for impurities in mixing water that is summarized in Table 2.

The limit for suspended particles in Table 2 agrees with the requirements of the U.S. Bureau of Reclamation [12], which has a turbidity limit of 2000 ppm for mixing water.

Sugar is probably the organic contaminant that causes the most concern. Steinour [13] explained that, although sugar as a contaminant in the field has gained a bad reputation as a retarder and strength reducer, these judgments need qualification. Laboratory tests have shown that, although smaller amounts retard the setting, they increase the strength development. With larger amounts, the setting is further retarded and early strengths such as those for two and three days (or even seven days) are severely reduced. The later strengths, however, are increased or at least not affected adversely, provided proper curing is maintained as required. With still larger amounts, the cement becomes quick-setting and strengths are reduced markedly for 28 days and probably permanently. The amount of sugar that can cause these different effects varies with the other factors involved, such as the composition of the cement, cement content, and ambient conditions.

Use of Seawater in Mixing Concrete

In addition to the supporting reference previously mentioned in Abrams's paper [5], the English article [9] also states that seawater with a maximum concentration of salts on the order of 3.5 % does not appreciably reduce the strength of concrete, although it may lead to corrosion of reinforcement.

A paper by Liebs [14] contains the results of comparative 7-, 28-, and 90-day compressive strength tests of concrete mixed with fresh water and with seawater. The data show that the seawater concrete had about 6 to 8 % lower strengths than the fresh water concrete. No efflorescence was observed. The article in Ref 11 pointed out that concrete made with seawater may have higher early strength than normal concrete, but strengths at later ages (after 28 days) may be lower. Steinour [13] stated that the use of seawater may cause a moderate reduction in ultimate strength, an effect that can be avoided by the use of a higher cement content. He also noted that concrete in which seawater is used as mixing water is sound but its use may cause efflorescence or dampness, and in reinforced concrete the risk of corrosion of the steel is increased. Seawater definitely should not be used for making prestressed concrete.

TABLE 2—Tolerable Concentrations of Impurities in Concrete Mixing Water

Impurity	Maximum Tolerable Concentration
1. Sodium and potassium carbonates and bicarbonates	1000 ppm
2. Sodium chloride	20 000 ppm
3. Sodium sulfate	10 000 ppm
4. Calcium and magnesium bicarbonates	400 ppm of bicarbonate ion
5. Calcium chloride	2 % by mass, of cement in plain concrete
6. Iron salts	40 000 ppm
7. Sodium iodate, phosphate, arsenate, and borate	500 ppm
8. Sodium sulfide	100 ppm warrants testing
9. Hydrochloric and sulfuric acids	10 000 ppm
10. Sodium hydroxide	0.5 %, by mass, of cement if set not affected
11. Salt and suspended particles	2000 ppm

Hadley [15] points out that seawater was used in the concrete for the foundation of the lighthouse at the extremity of the Los Angeles breakwater, which was built by the U.S. Army Corps of Engineers in 1910, and that 25 years later it was examined and found to be in good condition with sharp-edged corners and no disintegration. There are several references in the literature that indicate that salt water has been used in mixing plain concrete without incurring trouble at later periods. Much of the concrete for the Florida East Coast Railway was mixed with seawater with no detrimental effect due to its use [16]. Most engineers are of the opinion that seawater should not be used for mixing reinforced concrete; however, Dempsey [17] describes construction of military bases in Bermuda using coral aggregate and concludes that seawater seems to be satisfactory for making reinforced concrete and causes no problem beyond an acceleration in stiffening of the mixture. No harmful effect on the durability of reinforced concrete had occurred at the end of four years. Nonetheless, extreme caution is urged when mixing water for reinforced concrete is selected. If the water contains salts, the residual salts in the concrete when combined with air and moisture will result in some corrosion.

Effects of Algae in Mixing Water on Air Content and Strength of Concrete

A rather extensive series of laboratory tests reported by Doell [18] showed that the use of water containing algae had the unusual effect of entraining considerable quantities of air in concrete mixtures with an accompanying decrease in strength. The data in Table 3 were extracted from Doell's paper and are based on tests with 19.0-mm (3/4-in.) maximum-size aggregate concrete having a water/cement ratio of 0.5 and a slump of 40 to 75 mm (1.5 to 3 in.), with a constant ratio of coarse to fine aggregate.

In addition to the detrimental effect on strength, one of the important aspects of these data is that considerable quantities of air can be entrained in concrete by the use of mixing water containing algae.

Effect of Hardness of Mixing Water on Air Content of Concrete

Wuerpel [19] reported a series of air determination tests with waters of various degrees of hardness that shows that the air content was not affected by the hardness of the water.

Curing Water

There are two primary considerations with regard to the suitability of water for curing concrete. One is the possibility that it might contain impurities that would cause staining, and the other is that it might contain aggressive impurities that would be capable of attacking or causing deterioration of the concrete. The latter possibility is unlikely, especially if water satisfactory for use in mixing concrete is employed. In some instances the staining or discoloration of the surface of concrete from curing water would not be objectionable. The most common cause of staining is usually a relatively high concentration of iron or organic matter in the water; however, relatively low concentrations of these impurities may cause staining, especially if the concrete is subjected to prolonged wetting by runoff of curing water from other portions of the structure [3].

Test data from the Corps of Engineers [20] show that there is not a consistent relationship between dissolved iron content and degree of staining. In some cases, 0.08 ppm of iron resulted in only a slight discoloration, and, in other cases, waters with 0.06 ppm of iron gave a moderate rust-colored stain, while 0.04 ppm produced considerable brownish-black stain. Generally speaking, the conditions of these tests were such as to accentuate the staining properties of the water, since considerably more water was evaporated over a unit area than would be the case in most instances in the field.

With respect to organic impurities in water, it is virtually impossible to determine from a chemical analysis if the water would cause objectionable staining when used for curing concrete. It is advisable to use a performance-type test procedure, such as Designation CRD-C 401 of the Corps of Engineers [21]. This method outlines three procedures for evaluating the staining properties of water proposed for use in curing concrete. The Preliminary Method is intended for use in selecting sources that are worthy of more complete investigation and consists of evaporating 3000 mL of the test water in the concave area formed by the impression of a 100-mm (4-in.) watch glass in the surface of a neat white cement or plaster of Paris specimen. The Complete Method can be used to evaluate those sources that the Preliminary Method indicate to be promising. In the Complete Method, 11 dm^3 (3 gal) of test water drips on a mortar specimen exposed to heat lamps and forced air circulation. The Field Method is intended as a means of evaluating the water finally selected for use and involves the curing of a 1.9-m^2 (20-ft^2) slab of concrete with the test water for at least 28 days with maximum exposure to the sun with the test slab placed at a slight angle sufficient to keep it in a wet condition with minimum runoff. The test results by each of these three methods are evaluated by visual observation.

The Corps of Engineers' Standard Practice for Concrete [22] clearly states that there must be no permanent staining of surfaces where appearance is important. For these surfaces, the contractor has the option of using nonstaining water or of

TABLE 3—Effect of Algae in Mixing Water on Air Content and Strength of Concrete

Mixture Number	Algae in Mixing Water, %	Air in Concrete, %	Compressive Strength, 28 days, MPa (psi)
10	none control	2.2	33.3 (4830)
8	0.03	2.6	33.4 (4840)
7	0.09	6.0	27.9 (4040)
5	0.15	7.9	22.8 (3320)
9	0.23	10.6	17.8 (2470)

cleaning the surface after completion of moist curing. No cleaning is required for surfaces that will subsequently be stained when the structure is in service.

Summary

Mixing Water

The significance of the foregoing information presented is that any naturally occurring or municipal water supply suitable for drinking purposes can be used as mixing water for concrete and that most naturally occurring waters ordinarily used for industrial purposes are satisfactory. Many waters that upon casual examination would be judged to be unsuitable because of color, odor, or contamination with impurities, as in the case of marsh water, alkaline sulfate waters, and water containing industrial wastes, could be found to be satisfactory when tested in mortar or concrete since, in many instances, the strength would be greater than 90 % of the strength of comparative specimens made with pure waters. In the case of seawater, a strength reduction ranging from 8 to 15 % can be expected depending on job conditions; however, seawater ordinarily is not recommended for use as mixing water in reinforced concrete. The hardness of water usually does not affect air content of concrete; however, with certain anionic and nonionic admixtures, additional dosage may be required to obtain the desired air content. Algae in mixing water, however, can entrain air and significantly reduce strength.

Curing Water

It is improbable that a water used for curing would attack concrete if it were of the type suitable for use as mixing water. Organic matter or iron in the curing water can cause staining or discoloration of concrete, but this is rather uncommon especially where a relatively small volume of water is used; however, the suggested performance tests [21] will determine if a water possesses any potential staining qualities.

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Curing and Materials Applied to New Concrete Surfaces

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Preface

TO PREPARE THIS CHAPTER, THE CONTENTS OF ALL of the previous editions were drawn on. The author acknowledges the previous authors, Ephraim Senbetta [1], Rodger Carrier [2], C. E. Proudley [3], and John Swanberg [4]. The title of the chapter was changed from "Curing and Curing Materials" to reflect the change in scope of ASTM Subcommittee C09.22, which has initiated efforts to provide standards for materials that do not fulfill the curing function but are not addressed elsewhere, such as evaporation reducers, silicate treatments, bond breakers, and dry shake hardeners. The current edition will review and update the topics addressed by the previous authors, introduce new technology and perspectives, and include up-to-date references.

Introduction

Understanding of the processes that lead to the hardening of concrete continues to expand as more sophisticated techniques are used to observe and characterize the chemistry involved. However, the conversion of fresh concrete to a solid mass when cementitious materials hydrate is still the central fact, and the dependence of the ultimate properties of the material on the degree of hydration of the cement motivates interest in curing. ASTM Standard Terminology Relating to Concrete and Concrete Aggregates (C 125) and ACI Cement and Concrete Terminology (116R) [5], define curing as "action taken to maintain moisture and temperature conditions in a freshly placed cementitious mixture to allow hydraulic cement hydration and (if applicable) pozzolanic reactions to occur so that the potential properties of the mixture may develop." In addition, other properties, particularly of the surface region of a concrete mass, may be affected by materials applied and/or actions taken soon after placement.

No effort will be made in this chapter to review the history of the understanding of curing and related tests and specifications. The reader is referred to previous editions of this chapter, and to other documents referenced below.

A Task Group of The American Concrete Institute (ACI) Committee 308 on Curing, chaired by Ken Hover, has created an excellent Guide to Curing (ACI 308R) [6], which provides a thorough, up-to-date discussion of the current understanding of cement hydration with extensive references, as well as extended coverage of methods and materials, types of construction, and techniques of monitoring curing. The document is revised on a five-year cycle. Interest in high performance concrete has stimulated much research and discussion, and "Curing of High Performance Concrete: Report of State of the Art" published by the National Institute of Standards and Technology (NIST) [7] also contains a thorough discussion of the physical and chemical changes involved in cement hydration and a historical review of ACI curing requirements. It closes with suggestions on research needs to improve the assessment of curing and an extensive list of references to recent work. A more general discussion of curing theory and practice, also including extensive references, titled "Curing Portland-Cement Concrete Pavements," [8] has been prepared by Toy Poole of the U.S. Army Corps of Engineers (USACE) and is to be published by the Federal Highway Administration (FHWA).

Long established techniques and materials continue to be used effectively, and some refinements based on better understanding of the process are being adopted. The concept of initial curing is defined and discussed in ACI 308R as a means to minimize occurrence of shrinkage cracking from very early surface drying before traditional curing procedures may be applied. Fogging and evaporation reducers are effective for initial curing. Internal curing has been developed as a means of providing additional water to very low water/cement ratio (w/c) concretes where self-desiccation can create problems. This chapter will attempt to touch on these and other developments not addressed in previous versions.

Test methods to assess the effectiveness of curing continue to be a vexed issue. Whether it is preferable to measure some property of the final product concrete rather than the behavior of the materials or processes used in curing is still under debate. Some researchers persist in measuring concrete strengths as a function of curing procedures although curing procedures affect only the outermost few millimetres of any concrete. ASTM Test Method for Water Retention by Concrete Curing Materials (C 156) has been used since 1940 to measure loss of water from a "cured" specimen, despite widespread discontent with the reproducibility of the test. Recent changes to the procedure seem

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to have improved its precision. Attention has also turned to environmental measurements, which assay the worksite conditions to determine whether the concrete has received adequate curing action. The maturity method [9] of estimating concrete strength development using the measured temperature history of the concrete during curing is proving useful to better characterize overall results in testing programs. More exotic nondestructive test methods using X-rays, microwaves electrical resistance, and magnetic resonance are providing more detailed information on the progress of hydration in new concrete. Some of this technology will be addressed below.

In contrast to the situation reported by Senbetta [1] in 1994, the study and practice of curing concrete seems currently to be an increasingly active field. Indicators include the increasing number of presentations at the Transportation Research Board Annual Meetings and ACI sessions devoted to curing, as well as more publications in various journals.

Curing Methods and Materials for Water Retention

The methods and materials employed in the curing of concrete vary depending on the type of concrete, the type of structure including the orientation of the structural members, and the ambient conditions during the curing period. Control of loss of mix water may be accomplished with a barrier material, either a solid sheet, such as polyethylene film, or a liquid-applied film, which dries to form a membrane, or by maintaining a wet environment with fog, by ponding or with water soaked blankets. Flat work such as pavements and floors require much closer attention to control of water loss than formed mass concrete. On the other hand, care should be exercised to avoid large thermal gradients in mass concrete where heat of hydration may become a problem or in slabs exposed to direct sunlight.

Particularly for large flat surfaces (pavements or floors), initial curing may be required as soon as concrete is placed and consolidated and prior to the finishing operation. If, at this stage, conditions are such that rapid evaporation of water from the concrete is expected, and if the concrete is a type that has minimal bleeding, such as silica fume concrete, actions should be taken to either alter the ambient conditions near the surface of the concrete by fogging, or evaporation of water can be prevented or reduced by spraying an evaporation reducer on the fresh concrete surface. Fogging literally calls for the formation of a cloud of water droplets immediately above the concrete surface without appreciably adding to the water on the surface. Specialized fogging nozzles are commercially available for this purpose. Spray-applied evaporation reducers do not interfere with finishing the concrete nor do they have a lasting curing effect. They are different from liquid membrane-forming curing compounds. Although there are many evaporation reducers being marketed, the user is dependent on the manufacturer for details on use and performance expectations. No standardized performance tests are available. Under windy conditions, some kind of windscreen protection may be helpful or even required to complement other measures taken. On the basis of experience with minimizing plastic shrinkage cracking of bridge decks, the Kansas DOT has issued guidelines [10] providing specific sequences of procedures to be applied as dictated by weather conditions. Certain types of concrete, such as latex-modified concrete, may not require any action to prevent drying beyond the first 24 h.

After concrete is placed and all other subsequent operations such as finishing and texturing are completed, curing procedures may involve either keeping the concrete moist by ponding or covering with wet material and/or sealing the surface with sheet material or liquid membrane-forming curing compound to prevent evaporation. For formed concrete placements, forms will provide adequate protection against moisture loss as long as they remain in place. After removal, other curing procedures may be needed depending on the age of the concrete at the time of form removal.

Standard Test Methods

Although a wide range of test procedures have been used to evaluate whether a particular concrete specimen has been adequately cured, ASTM publishes only one test method to evaluate the water retaining properties of a curing material. Because the method has exhibited poor interlaboratory reproducibility, a multitude of variants have been developed by state agencies, and internationally, with the hope of improving local reproducibility, or better measuring particular characteristics of liquid membrane-forming curing compounds.

ASTM Test Method for Water Retention by Concrete Curing Materials (C 156) was first published in 1940. In a delayed response to the report of Leitch and Laycraft [11] and comments from many others, C 156 has been modified with a requirement that the test cabinet environment be characterized by a measured evaporation rate in addition to controlled temperature and relative humidity. Control of air velocity is necessary to achieve the desired evaporation rate, but air velocity is more difficult to control than evaporation rate over an extended time. A procedure for measuring the evaporation rate is specified in an Annex to the method. A further change to the method relaxes the specification for the mold used to cast mortar specimens for the test. In recognition of a trend established many years ago by state Department of Transportation (DOT) laboratories, and suggested by the work of Leitch and Laycraft [11] and of the Corps of Engineers [12], the 150 × 300 × 50 mm (6 in. × 12 in. × 2 in.) mold specification is replaced by a requirement of minimum surface area of 12000 mm^2 (18.6 $in.^2$) and minimum depth of 19 mm (3/4 in.). In the author's experience, smaller molds can be used by a single operator to run triplicate determinations with less time and effort than required for handling a single 150 × 300 mm (6 in. × 12 in.) mold, also consuming far less cement and sand. A recently completed round robin involving six laboratories and four different curing compounds, organized by the California DOT, is the basis of a new precision statement being balloted for addition to the method. It shows the single operator standard deviation among test results to be 0.038 kg/m^2 and the interlaboratory standard deviation among test results to be 0.070 kg/m^2. There appears to be no loss of precision with smaller molds used by some of the participants. Hopefully the state DOTs that have developed variants of the ASTM method will find the advantage of a uniform testing method across the country to be good reason to adopt the ASTM procedure.

It has long been recognized that the results of the C 156 test are sensitive to the timing of the application of curing compound. The procedure specifies that the mortar surface shall be free of surface water but not dry below the surface. Application of curing compounds to surfaces that are still bleeding or have standing water results in disruption of the film-forming characteristics of the curing material. In the field, if careful attention is not paid to time of application, a product that meets the laboratory test requirement may not provide the expected protection. Covarrubias [13] reported that three of eight compounds tested according to C 156 (and passing) also performed satisfactorily when applied 30 min after molding the specimens, i.e., while the mortar was bleeding. These products can presumably be applied to pavements immediately after finishing. No information is provided about the nature of these products.

ASTM Test Method for Evaluating the Effectiveness of Materials for Curing Concrete (C 1151) was published as a proposal in 1987 and as a standard in 1990 as a possible alternative to C 156, but withdrawn in 1998 due to lack of interest. The method measures a property of the affected specimen rather than the behavior of the applied material. By measuring the absorptivity of a near surface layer and comparing it to that of an internal layer, the relative degree of hydration, and therefore the efficacy of the applied curing procedure, is determined. The method has been applied to analyzing concrete of all ages in the field, but was not accepted as a routine test for liquid membrane-forming curing compounds because of the complexity of the procedure. The technique is still used with many variations in research situations.

Other tests that have been used in studies involving curing, but are not under the jurisdiction of ASTM C09 Concrete Subcommittee C09.22, include measurements of absorptivity, hardness, gas permeability, chloride intrusion, freeze-thaw stability, and strength. Most properties of new concrete are changing rapidly in the first few days of its life and the age and temperature history of the specimens must be carefully specified if results are to be compared.

Standard Specifications

ASTM C09 Concrete Subcommittee C09.22 has jurisdiction over three material standards related to curing, one for sheet material and two for liquid membrane-forming curing compounds. The American Association of State Highway and Transportation Officials (AASHTO) publishes related specifications as do the USACE and many state departments of transportation in the United States. Worldwide, specifications are written in many countries but most seem to resemble the ASTM requirements, so that products manufactured for the U.S. market are widely acceptable.

Sheet Materials

In addition to traditional burlap, polyethylene sheeting, poly-bonded burlap, and various types of water-soaked fabric, a number of new products have appeared on the market providing various reputed advantages. Several versions of polyethylene bonded to woven or nonwoven synthetic fabrics are available having superior strength, and are therefore more suitable for multiple uses. Some of these also act as a water reservoir if the sheet or the concrete is wetted before being covered, and possibly reduce or eliminate the blotchy surface marking often encountered with polyethylene alone. Another product employing an aluminized film bonded to the top surface offers exceptional protection from solar heating of flat surfaces by reflecting almost all-incident radiation. "Bubble-wrap" type materials are available to provide insulating properties in cold weather or to shield against heat gain from the environment in hot weather. None of these newer characteristics have been measured by standardized tests for curing applications.

ASTM Specification for Sheet Materials for Curing Concrete (C 171) covers burlap, polyethylene sheeting, and poly-bonded burlap, and was revised in 2003 to simplify the required testing. ASTM Specification for Polyethylene Sheeting for Construction, Industrial and Agricultural Applications (D 4397) covers all the necessary properties of polyethylene sheeting used for concrete curing and therefore no additional tests are required for material meeting this specification. Other sheet material is to be tested by ASTM Test Methods for Water Vapor Transmission of Materials (E 96) rather than C 156. This change recognizes that while the water loss from liquid membrane-forming curing compounds is a combination of loss during film formation and loss through the dried film, for sheet materials the loss rate should be constant. In practice, test results using Method C 156 for sheet material are more likely to depend on the quality of the seal of sheet to specimen than on the inherent water transmission rate of the product under test. The new test limit of 10 g/m^2 loss in 24 h is set in terms of Method E 96, Procedure E (37.8°C, (100°F)), and represents a more stringent loss rate requirement than previously required in recognition of the known characteristics of polyethylene sheet (D 4397 lists 5.5g/m^2 loss in 24 h as the requirement for 4 mL polyethylene). The new test limit corresponds to 0.03 kg/m^2 in 72 h, which is below the level practically measurable by the C 156 procedure. The equivalent AASHTO specification for sheet material is M 171, which does not yet reflect these changes.

Liquid Membrane-forming Curing Compounds

Liquid membrane-forming curing compounds are paint-like products that can be applied to freshly placed concrete by spray, brush, or roller, and dry to form a membrane or film that retards the evaporation of water. They offer the advantages of ease of application, low material and labor costs, and ready availability. However, there are wide variations in the quality of available products and they are sensitive to the time of application and the thoroughness of the applicators. Despite years of effort by many researchers, the performance of any given batch of curing compound as measured by C 156 is still not accurately predictable by any other measurable property.

ASTM Specification for Liquid Membrane-forming Curing Compounds (C 309) covers the workhorses of curing technology. They have evolved under pressures of environmental concerns so that the products used in highest volumes are water-borne emulsions with little or no VOC content, capable of providing protection to large areas of concrete at low cost in both labor and materials. AASHTO M 148 is a corresponding specification, but revisions lag several years behind C 309. The problems of assuring the quality of the products and the workmanship of the applicators remain.

The specification includes requirements for water retention, reflectance of white-pigmented compounds, and drying time. The water retention requirement calls for mass loss of no more than 0.55 kg/m^2 of surface in 72 h when applied at 5.0 m^2/L (200 ft^2/gal) when the testing is done according to ASTM C156. This is, by far, the most important requirement, even though measurement is difficult and the validity of the qualifying value is still under debate. The requirement for a minimum reflectance of 60 % is related to the prevention of heat buildup in concrete exposed to solar radiation. The pigment also helps to visually assess completeness of coverage. The maximum drying time requirement is to guard against the concrete having a tacky or slippery surface and to make sure the curing compound does not track off when walked on. The major classes of film formers used for concrete paving are based on petroleum waxes or hydrocarbon resins. For commercial floors styrene-acrylic resins are widely used because they can also serve as sealers for in place concrete. Chlorinated rubbers were once widely used for their excellent water retention properties, but have fallen from favor because of their low UV resistance (they darken in sunlight) and more recently problems of resin supply related to environmental and economic issues. Styrene-butadiene resins also have excellent water retention but low UV stability. Acrylic resins as solutions or latexes show excellent UV stability but inferior water retention properties.

Corps of Engineers Specification CRD-C300 is similar to C309, but calls for water retention of 0.31 kg/m^2 in seven days when tested by CRD C 302 (which is similar to the ASTM method). However, the spec is not being actively maintained by the Corps and good quality cures manufactured to meet C 309 are generally acceptable for Corps applications.

Many state DOTs have generated variants or extensions of these specifications in response to their perceived needs. Likewise most European countries and members of the British Commonwealth have established specifications and test methods for curing compounds. A review of these is beyond the scope of this chapter.

Liquid Membrane-Forming Compounds with Special Properties for Curing and Sealing Concrete (C 1315), a new specification, was adopted in 1995 in response to a perceived need to replace General Services Administration Federal Specification Curing Compound, Concrete, For New and Existing Surfaces, TT-C-800, which was withdrawn in 1978. Compared to C 309, the new specification calls for more stringent water retention performance (0.40 kg/m^2 in 72 h when applied at 7.4 m^2/L (300 ft^2/gal)) and minimum solids content ($>$25 %) and includes requirements for resistance to UV degradation (non-yellowing), chemical resistance, and compatibility with adhesives used to bond tile or carpet to concrete. These properties are important in a range of applications including commercial floors, bridge decks, and high performance concrete (HPC) in general. The Environmental Protection Agency (EPA) has adopted a regulation [14] allowing products classified as curing and sealing agents and defined by compliance to C 1315 to have a Volatile Organic Compound content of 700 g/L, whereas curing compounds meeting C 309 are allowed only 350 g/L. Practically speaking, this regulation allows solution-based products to be sold for curing and sealing whereas only emulsion based products can realistically meet the VOC limit set for curing compounds.

A few state DOTs have adopted material specifications calling for the use of α-methyl styrene polymer in liquid membrane-forming curing compound formulations, especially for bridge decks. The specifications call for lower water loss than in C 309, typically around 0.3 kg/m^2 in 72 h. There is little evidence that the specified resin yields products that are really superior to equivalent formulations with similar resins.

Effects of Curing on Concrete Properties

For any concrete mix, proper curing allows for maximum hydration of cementitious materials resulting in the greatest possible reduction in capillary porosity of the initial mixture by the formation of products of hydration. This process, in turn, results in minimum permeability and maximum strength and durability. Consequential effects include reduced shrinkage and maximum protection from rusting of reinforcing steel. For detailed discussion of these factors see for instance ACI 308R [6]. A recent article by Erlin, Nasvik, and Powers [15] summarizes the current understanding of curing with particular reference to low w/c mixes.

The initial w/c of the concrete sets a limit on the ultimate results since at w/c $> \sim 0.4$ the excess water limits the initial density on placement, and at w/c $< \sim 0.4$ incomplete hydration resulting from internal desiccation may lead to high capillary porosity. The necessity for special curing attention for high performance concrete arises from this relationship. These effects have been extensively discussed and analyzed elsewhere [6,7].

A frequently reported failure is plastic shrinkage cracking, which results from rapid surface drying before the concrete has hardened sufficiently to resist shrinkage stress. This cracking radically reduces the durability and increases the permeability of the surface. It is especially likely to occur in mixes with low w/c, and low bleed rates, for instance those with silica fume or other pozzolans. Wojakowsi of the Kansas DOT [10] has demonstrated that careful attention to maintenance of curing conditions by use of fogging, evaporation reducers, and sheet products, each applied at the appropriate time, allows production of crack-free bridge decks from high performance concrete mixes. Failure to attend to any of the details results in shrinkage cracking of the surface. Similar results have been reported from the Virginia and Iowa DOTs.

A more subtle effect of poor curing has been proposed by Shotwell [16] to explain scaling of driveways and sidewalks during the first season of exposure to freezing and thawing. Petrographic examination of poorly cured mortar with a porous surface layer showed that a relatively dense band of carbonated matrix is formed just beneath the surface and inhibits penetration of water into the underlying concrete. The result is concentration of water in the porous top layer leading to freeze-thaw scaling. Typically, in otherwise good concrete, the deterioration decreases after the initial scaling.

Studies of curing focus primarily on whether desired properties such as low permeability or other measurable qualities of concrete products are attained. An alternate approach is to determine if the materials or processes used perform so as to maintain the conditions required for optimum hydration. This requires careful monitoring of temperature, ambient and/or internal humidity, and air flow as well as evaluating the ability of liquid membrane-forming curing compounds and sheet material to impede or prevent the evaporation of water.

Effects of Ambient Conditions on Curing

The need to take deliberate steps to ensure proper curing of concrete is dependent on the ambient atmospheric conditions. In certain environments where the relative humidity and temperature are favorable, no deliberate action may be needed to cure the concrete. But, usually conditions require action for some portion of the curing-sensitive period. Control of ambient conditions may involve application of energy rather than materials, but nevertheless it seems appropriate for discussion in this chapter.

Although there are no standard definitions, ambient conditions may be put into three temperature categories; hot, $> 32^\circ$C (90°F); cold, $< 10^\circ$C (50°F); and normal, ($10-32^\circ$C) ($50-90^\circ$F). ACI Committee 306, Cold Weather Concreting, continues to struggle with defining cold weather in a way that is useful to construction professionals [17]. The severity of the environment as it concerns curing is affected by relative humidity and wind speed. Therefore, by determining the ambient temperature, relative humidity, wind speed, and concrete temperature, the rate of evaporation of water from the exposed surface of fresh concrete can be estimated. A nomograph [18] has been commonly used for this purpose. With the advent of readily accessible computer power, the equations upon which the nomograph is based have been reduced to programs that output the evaporation rate given the ambient conditions, or allow calculations of the effects of actions such as reducing the initial concrete temperature [19]. As has been pointed out in the ACI Guide to Curing Concrete [6], at low ambient temperatures evaporation rates from fresh concrete surfaces may be even greater than at any other conditions because the concrete is warmer than the air, providing an added driving force for evaporation. Thus, early measures to protect concrete surfaces may be even more important in cold than in hot weather. Because the original nomograph was based on models for evaporation from bodies of water and wind speeds measured at a height of six feet above the surface, Jeong and Zollinger [20] have taken the additional step of modifying the evaporation model on which the nomograph is based to more accurately reflect real-world values in concrete. Their model and experimental data indicate high sensitivity of evaporation rates to wind speed at the concrete surface and suggest that the ACI nomograph overestimates evaporation from concrete surfaces after bleeding has stopped.

High temperature curing, commonly referred to as accelerated curing, but better described as heat curing, has received considerable attention in the wake of failures of cast products attributed to excessive temperatures during the curing cycle. In the precast industry, heating concrete forms, usually with steam, increases the turn over rate for reuse of the forms by rapidly producing sufficient strength to allow form removal. Increased temperature generally increases the rate of all chemical reactions, but a number of precautions apply. At high temperatures some equilibrium reactions may actually be reversed, and all of the complex hydration reactions involved in hardening of concrete are not affected equally by temperature. Perhaps even more importantly, thermal expansion stresses introduced in the plastic concrete must be relieved in the hardened products to prevent cracking. The probable cause of some failures is the formation of deleterious hydration products at higher temperatures. Agreement on maximum allowable temperatures, and heating and cooling cycle rates is being sought in ACI 308 T.G. on accelerated curing.

Heat curing of high-performance concrete has been studied by Freyne, Russell, and Bush [21]. Based on experiments involving 31 different HPC mixtures and six different heating schemes, and the measurement of 1 day and 28 and 56 day strengths they concluded that heat curing was damaging to ultimate strength potential and sometimes even failed to accelerate early strength development. Intense heat (60–71°C (140–160°F)) was found to be more damaging to ultimate strength than moderate heat (30–42°C (86–108°F)). They concluded that heat curing might be useful in a business model emphasizing speed of construction, but not always pragmatic in a model emphasizing life-cycle cost.

Other Materials Applied to New Concrete

Silicates

Application of alkali silicate solutions to new concrete has been practiced for many years. In 1998 a note was added to C 309 specifically excluding silicates from the specification since they are not membrane formers. No known silicate formula reduces water loss in the C 156 test to the level specified by C 309. However, when properly applied, silicates do harden and/or densify concrete surfaces. Under certain conditions, they are used to produce highly polished, nearly impermeable floors. Subcommittee C09.22 is attempting to produce a specification covering performance requirements for silicate products, but to date all available information is manufacturer specific and formulations are proprietary. Nasvik [22] has reported the use of silicates prior to diamond polishing of floors to enhance surface hardness, and various manufacturers recommend their use in conjunction with less aggressive polishing techniques.

Bond Breakers

Bond breakers for tilt-up construction are another target for specification writing. Some products are presented as acting both as curing compounds and bond breakers, while others perform only the bond breaking function. In this area too, no test methods exist to characterize the performance of bond breakers and therefore users must depend on manufacturers' claims. A range of products are established in the market, but formal comparisons are not possible without an agreed on test method. A task group within C09.22 is drafting a specification and exploring test procedures.

Dry Shake Hardeners

Granular materials, including natural silicate minerals, ferrosilicates, and iron may be broadcast on freshly placed concrete and trowelled into the surface to produce exceptionally wear resistant surfaces. Here again a multitude of products are available but no specification literature exists beyond that supplied by the manufacturers. A draft specification is currently being circulated in C09.22.

New Developments

Internal Curing

Increasing use of high performance concretes has brought with it several new concerns and response to new problems. Aside from the need for special attention to early curing conditions [7] the possibility of internal desiccation leading to autogenous shrinkage arises in low w/c mixes. Mather and Hime [23] calculate that for concrete made with w/c below 0.4, not all of the original mixing water-filled space can be filled with hydration product. They observe that the critical feature of the chemical reaction between the constituents of the cement and the mixing water is the ratio of their volumes, and that for w/c of less than 0.4 some of the cement will remain unhydrated. In practice, externally supplied water is ineffective in penetrating into mass concrete because of the rapid development of low permeability of the mass. A process described as internal curing has been developed to help reduce cracking in HPC slabs, and provides a solution to the w/c dilemma.

Internal curing differs from traditional curing in that it involves an addition to the concrete mix before it is placed rather than action taken after placement. Internal reservoirs of water are created by adding high moisture content structural lightweight aggregate (see chapter in this volume on Lightweight Concrete and Aggregates) or water absorbing polymers to the mix. This water is more strongly held than the free mix water, but available to become water of hydration or gel water throughout the mass as needed. Strictly speaking this is curing by means of an admixture rather than action taken to retain water. Both AC I and RILEM (International Union of Laboratories and Experts in Construction Materials, Systems and Structures) [24] are actively developing specifications around these products. Bentz and Snyder [25] have developed equations for calculating the level of saturated lightweight fine aggregate required for complete curing of HPC.

Curing Meter

The Curing Meter devised by Hansen and Jensen [26] continuously shows a readable value of the local integrated water loss in kg/m^2. The unit, composed of an evaporation cell and a capillary tube mounted on a thin aluminum plate is shown to register the total loss from a fresh concrete surface over a time period of interest, avoiding separate measurements of temperature, relative humidity and air velocity. The device is effective even with continually varying atmospheric conditions. Unfortunately, it does not appear to be appropriate for measuring the effects of materials applied to the concrete surface since calibration of the device assumes evaporation from a free-water surface. It might be useful for measuring the curing environment in test chambers.

Maturity Testing

Maturity testing methods as a means of predicting concrete strength have been around for over 60 years. For a discussion of these methods see the chapter in this volume titled Prediction of Potential Concrete Strength at Later Ages. Maturity testing methods are based on calculation of a maturity index associated with the relationships between a concrete mixture's temperature changes, its curing (hydration) history, and its rate of strength development [27]. ASTM Standard Practice for Estimating Concrete Strength by the Maturity Method C 1074, has been developed for regulating and ensuring the proper use of the maturity testing method for determining in-place concrete strength. The method generally assumes that best practicable curing procedures have been applied. In practice the index may be used to decide when curing procedures may be terminated and/or when forms or bracing may be removed. Commercial systems are available and a system is under development by Tikalsky and Tepke [28] at Penn State.

Research on Hydration

Instrumental analytical methods for study of the early stages of the hydration process include X-ray absorption, nuclear magnetic resonance, microwave resonance and resistivity measurements. All of these have been applied to give a more detailed understanding of the early stages of hydration of cementitious mixtures.

An X-ray environmental chamber at the Technical University of Denmark has been used to measure drying/hydration of cement pastes at various depths within the specimens under carefully controlled conditions. X-ray absorption is proportional to the density of the material through which the X-rays are passing, so a high w/c ratio specimen will absorb less X-rays than a lower w/c specimen, and a dried specimen will absorb less than the same specimen in its initial saturated condition. These experimental data have been used in combination with the NIST CEMHYD3 computer model to verify that, unlike nonreactive porous materials in which a drying-front can be observed to proceed from the surface inward, these specimens initially appear to dry uniformly throughout as water is consumed in hydration [29]. This work contributes to understanding of the mechanisms of water movement in the drying/hydration of cement and may be useful in designing curing systems for concrete.

Proton Magnetic Resonance would seem to offer a way to follow the kinetics of transfer of water from the free to gel and combined states since the chemical shift of the proton in each state should be distinctive allowing continuous measurement of the proportion of water in the sample of each type. However, the only work found in this field is the use of nuclear relaxation NMR experiments (T2 relaxometry) to follow the hydration process in internally cured HPC as a change in the average bulk property of cement paste or mortar [30].

Time-Domain-Reflectometry microwave spectrometry [31] has been applied to cement samples to show signals assigned to free and bound water allowing the monitoring of hydration progress as the microstructure of the product develops.

Following changes in the resistivity of fresh concrete is difficult because with direct current, polarization effects at the electrodes introduce errors, and if high frequency alternating current is used to avoid polarization, uniformity of contact and evolution of gases are still issues. Li, Wei, and Li [35] have devised a noncontacting method of measuring concrete resistivity, which employs a transformer principal. Mortar or concrete is cast in a ring shaped mold, which becomes the secondary of a transformer. Voltage is applied to the primary of the transformer, and voltage and current in the toroidal mold are measured to provide a plot of resistivity vs. time. Their data for mixtures ranging from 0.3–0.5 w/c allowed them to identify four periods in the early maturation of cementitious mixtures: (I) dissolving period (initial hydration) wherein resistivity falls as soluble components of the cement saturate the mix water; (II) competition period wherein resistivity begins to rises as dissolution and precipitation compete; (III) setting period wherein resistivity slowly increases as hydrates are formed; and (IV) hardening period wherein resistivity increases as hydration continues to immobilize water in the mix. All of these effects are seen in less than 24 h, providing a profile of the cement hydration process. Although impractical for fieldwork, the device should be useful for laboratory studies of various cements and admixtures, as well as the effects of curing procedures on hydration.

Needs for Future Work

Some of the work called for in the previous version of this chapter has been accomplished, and there has been an increase in effort and attention to curing in general. Active programs at NIST and within the USACE are aimed at better understanding and better practical control of all the factors involved in curing. The ACI Guide (308R) and Specification (308S) offer extensive guidance. Since rapid construction schedules are frequently important, efforts to find methods to establish how long curing measures must be applied in specific situations or to specific structures will undoubtedly continue. Reliable curing monitoring methods that can be used by inspectors at job sites, and new test procedures for evaluating the properties of concrete at the end of the curing period to determine the effectiveness of the curing are much desired.

An array of instrumental data on hydration is appearing. There is an opportunity for someone to integrate all of this information in a multidimensional understanding of the complex chemistry of cement hydration.

Another essential component of future developments to improve the curing of concrete is the developments of effective educational programs to enable people to develop a real appreciation for the value of curing. Unless one understands the need for curing and its profound impact on the properties of concrete, the steps prescribed to cure the concrete merely become requirements that one must satisfy without concern for the end result. With Senbetta [1], this author feels that, in the construction of concrete structures, curing of the concrete continues to be an under appreciated step. For a great deal of concrete, durability problems arise as a result of poor concrete surface properties because of the lack of adequate curing.

From the curing materials point of view, the primary means of curing, as stated earlier, is preventing the drying of concrete by covering the surface of the concrete with cloth, paper, plastic, or a membrane-forming curing compound. The effectiveness of these materials depends on the quality of the materials; the time of application of the materials, that is how soon after the concrete is placed; and how well they are applied and kept intact and in place for the specified length of time. The special properties of sheet materials for insulation, water absorption, and reflectivity need to be characterized and their contribution to curing evaluated. Finally, because liquid membrane-forming curing compounds used in volume are low-cost, low-margin items, the technology of their formulation and manufacture remains rudimentary. If available technologies for studies at near molecular scale are applied to study manufacturing techniques and the film formation process, it may be possible to understand the origins of batch-to-batch variability and develop products with predictable reliability and special properties.

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Air-Entraining Admixtures

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Preface

TO PREPARE THIS CHAPTER, THE CONTENTS OF THE 4th edition were drawn on. The author acknowledges the authors of the prior three editions: *ASTM STP 169* (1956) by Carl E. Wuerpel, U.S. Army Corps of Engineers, and *ASTM STP 169A* (1966), *B* (1978), *and C* (1983) by Paul Klieger, Consultant–Concrete and Concrete Materials. The current edition will review and update the topics addressed by the previous authors, and introduce new relevant technology with corresponding up-to-date references.

Introduction

Air-entrainment in concrete has been universally accepted and is a well-established means for greatly enhancing the ability of concrete to resist the potentially destructive effect of repeated cycles of freezing and thawing, as well as alter the workability and yield of cementitious mixtures. Air-entrainment should be mandatory when concrete is to be exposed to such harsh environments, particularly when chemical deicers are being used, as on pavements and bridge decks.

A thorough survey of the early development of air-entrainment is presented by Gonnerman [1]. The following paragraph from Gonnerman's report is of particular significance:

These projects (test roads constructed in 1935–1937) showed no relationship between surface scaling and composition of the cement, but they did show clearly that portland cement that inadvertently contained "crusher oil" reduced surface scaling as did many of the blends of portland and natural cement that contained tallow added during grinding of the natural cement. Laboratory tests disclosed that the beneficial effect of the crusher oil and tallow was due entirely to the additional air entrapped in the concrete by these air-entraining agents.

Other investigators [2,3] came to similar conclusions. In these early instances, the air-entrainment was not intentional but resulted from the presence of the crusher oil or the use of the tallow as a grinding aid during the production of the cement. These were the forerunners of materials called air-entraining additions, now used to produce air-entraining cements. Materials similar to presently used additions are called air-entraining admixtures when added with the other concrete ingredients at the time of mixing, the more widely used method for obtaining intentionally entrained air. This chapter is concerned with this class of materials.

What are these materials; how do they function, both as to the process of entraining air, enhancing durability, and effecting workability; how can they be specified and tested to ensure adequate performance; what are the effects of concrete material properties including other chemical admixtures, production procedures, field conditions, and construction parameters on air content and hardened air-void parameters? These are some of the topics that will be addressed in this chapter.

Definitions

The following definitions can be useful in discussing air-entrainment in mortars and concretes:

1. Air-entrainment–The introduction of air in the form of discrete air-voids or bubbles dispersed throughout the mixture as a result of the use of air-entraining materials.
2. Entrained Air–The air, made up of discrete air-voids, that becomes part of a mixture during the process of air-entrainment.
3. Entrapped Air-voids–Air-voids not resulting from intentional air-entrainment. Such voids are larger than those resulting from intentional air-entrainment and are at times referred to as natural air-voids.
4. Entrained Air-voids–Air-voids resulting from the use of intentional air-entrainment. Such voids are generally spherical in shape and considerably smaller than the natural air-voids.
5. Air-Entraining Admixture–A material added to cementitious mixtures at the time materials are batched for mixing, the use of which results in intentional air-entrainment. (See ASTM Terminology Relating to Concrete and Concrete Aggregates (C 125) for the definition of an admixture.) A material conforming to the requirements in ASTM C 260 can be regarded as an air-entraining admixture.
6. Air-Entraining Addition–Air-entraining material interground with hydraulic cement. (See ASTM Terminology Relating to Hydraulic Cement (C 219) for the definition of an addition.)

Materials Used as Air-Entraining Admixtures

There are many materials capable of functioning as air-entraining admixtures. In an extensive evaluation program, the Bureau of Public Roads [4] separated 27 commercial air-entraining admixtures submitted for test into the following classifications: (1) salts of wood resins (pine wood stumps); (2) synthetic detergents (petroleum fractions); (3) salts of

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sulfonated lignin (paper pulp industry); (4) salts of petroleum acids (petroleum refining); (5) salts of proteinaceous materials (processing of animal hides); (6) fatty and resinous acids and their salts (paper pulp and animal hide processing); and (7) organic salts of sulfonated hydrocarbons (petroleum refining).

Several reports (Rixom [5], Dodson [6], Ramachandran [7], Paillere [8], Kodama [9], and Bauverlag GmbH [10]) provide an excellent description of the chemistry of air-entraining admixtures and discuss their function in freshly mixed and hardened concretes. Whiting and Nagi [11] classify current air-entraining agents into five broad categories and provide general performance characteristics for each group, and includes gum and wood rosins, which are reported to provide similar performance to neutralized Vinsol Resin®. A study [12] on the various fractions for one class of materials, alkylsulfonates, indicates optimal air-void and freeze-thaw performance are obtained from the C8 to C10 fraction. As Dodson [6] has previously noted, modern air-entraining agents continue to consist of anionic materials based on their track record of providing cost-effective and predictable air-entrainment.

Function of Entrained Air in Freshly Mixed and Hardened Concrete

The major reason for the use of intentionally entrained air is to provide concrete with a high degree of resistance to freezing and thawing, particularly when chemical deicers are used. (The discussion to follow will also be applicable to the use of air-entraining cements, in which the air-entraining agent is used as an addition during the grinding of the cement clinker.) There are numerous other advantages, also, to the use of intentionally entrained air. For example, plasticity and workability are increased, enabling a reduction in water content. Uniformity of placement and consolidation can be achieved more readily, thus reducing segregation, and bleeding is reduced. Another study [13] found that the internal surface area of air-entrained concrete plays a significant role on sound absorption. Wang and Gillot [14] report that alkali-silica reaction can be partially mitigated with air-entrainment. These and other advantages are discussed in detail by Lerch [15] and Bruere [16].

To achieve the improvement in frost resistance, the intentionally entrained air must have the proper total volume of air, spacing factor, and size and distribution of the air-voids to provide efficient protection to the cement paste. Powers' [17–19] contributions to the understanding of how entrained air functions in providing increased frost resistance have been outstanding.

Powers developed the concept that internal hydraulic pressure created by the resistance to flow or movement of excess water volume produced during the freezing process is responsible for distress to cementitious mixtures. To keep this internal pressure below the tensile or rupture strength of the paste, Powers showed that the air-voids must be well-distributed throughout the cement paste and sufficient in number so that each void provides protection to the adjacent cement paste, and the protected volumes overlap to leave no unprotected paste.

Later work by Powers and Helmuth [20] indicated that, in addition to the generation of hydraulic pressure during freezing, another important factor may be the diffusion of gel water to capillary cavities contributing to the growth of ice bodies in these cavities resulting in the development of additional expansive forces. A modified and expanded version of Helmuth's model of ice penetration in concrete has been proposed by Chatterji [21], who attributes the specific efficiency of air-entraining agents by the different degree of hydrophobicity they impart to the surface of the entrained air-void. The hydrophobic surface is reported to minimize the ice-paste bond, which promotes ice growth within the void causing water to be withdrawn from the surrounding paste by suction. Water movement under suction is therefore unable to produce expansive pressure.

Powers [18] developed the concept of air-void spacing factor to characterize an air-void system and laboratory freezing and thawing data available at the time to show that the void spacing factor for frost resistance should be about 0.01 in. (0.25 mm) or less. The void spacing factor is defined by Powers as the average maximum distance from any point in the cement paste to the nearest air-void, and is the most important air-void parameter for frost resistance. This is an indication of the distance water would have to travel, during the freezing process, to reach a protective air-void. Work by Mielenz et al. [22] indicates that an upper limit of about 0.006–0.008 in. (0.152–0.203 mm) is required for extreme exposures. Extensive freezing and thawing tests by Klieger [23,24] provided further substantiation of the void spacing factor concept. These tests called attention to the need for different volumetric air-content requirements for concretes made with different maximum sizes of coarse aggregate. Table 1, adapted from Ref 22, shows this effect of maximum size of aggregate on the optimum air content along with void spacing factors. Although the total air contents of the mixtures shown in Table 1 vary through a wide range, the air content of the mortar fraction is essentially

TABLE 1—Air-Void Parameters at Optimum Air Contents of Concretes (Adapted From Tables 17 and 18 of Ref 14)

Maximum Size of Aggregate		Cement Content, 5½ bags/yd^3		$\bar{L}$		Cement Content, 7 bags/yd.^{3}		$\bar{L}$	
in.	(mm)	Optimum Air Content, %[a]	Mortar Air Content, %	in.	(mm)	Optimum Air Content, %	Mortar Air Content, %	in.	(mm)
2½	(63)	4.5	9.1	0.007	(0.18)	4.5	9.2	0.007	(0.18)
1½	(37.5)	4.5	8.5	0.008	(0.20)	4.5	8.4	0.008	(0.20)
¾	(19)	5.0	8.3	0.009	(0.23)	5.5	9.2	0.007	(0.18)
⅜	(9.5)	6.5	8.7	0.011	(0.28)	7.0	9.6	0.008	(0.20)
No. 4	(4.75)	9.0	9.0	0.012	(0.30)	10.0	10.0	0.008	(0.20)

[a] Optimum air contents determined from the relationship between expansion during 300 cycles of freezing and thawing and air contents of concretes.

constant at about 9 %. As the maximum size of coarse aggregate increases and workability and cement content held constant, less mortar is required in the mixture; therefore, a reduction in total concrete air content with an increase in maximum size of coarse aggregate is to be expected.

Most of the early field and laboratory work on air-entrained concretes dealt with paving-type concrete in which the coarse aggregate was generally about 11/2 in. (38 mm) maximum size. In concretes without intentional air-entrainment, the air content may range up to as high as 1 or 2 % by volume. However, this air is composed of entrapped air-voids that are too large to be effective with respect to improving frost resistance or workability. The provision of an additional 3 % of intentionally entrained air by the use of an air-entraining admixture will provide an air-void system well-distributed throughout the matrix and containing a sufficient number of air-voids to meet the void spacing factor requirements for adequate resistance to freezing and thawing, even in the presence of deicing chemicals.

The importance of size and distribution of air-voids, as contrasted with total volume of voids alone, can be seen in the results of a study made some years ago in the laboratories of the Portland Cement Association. Air-entrained concretes were prepared using an acceptable proprietary air-entraining admixture and four nonproprietary materials that exhibited a potential for entraining air. A non-air-entrained concrete was also included in these tests. In addition to the determination of air content of the freshly mixed concrete as described in ASTM Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231), the air content, void spacing factor, specific surface, and number of voids per lineal inch of traverse were determined on the hardened concretes as described in ASTM Practice for Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete (C 457). Additional refinements of the technique enabled the determination of the total number of air-voids per unit volume of concrete. The results of these measurements and the performance of the concretes when frozen and thawed while immersed in water are shown in Table 2. It is apparent from these data that an air-entraining admixture must not only be capable of entraining some volume of air but also that the air-void system must be characterized by a large number of small, well-distributed air-voids to provide a high degree of frost resistance.

Recently, a number of refinements to the interpretation of various microscopic analysis have been proposed to allow better correlation of air-void systems in hardened concrete to freeze-thaw durability. Pleau et al. [25] found that the flow length concept, whereby the flow length is defined by the authors as the distance that freezable water must travel through the cement paste to reach the perimeter of the nearest air-void. The authors found that the size distribution of circles intercepted by a plane and chords intercepted by a line of traverse provide an indication of the actual spatial distribution of air-voids in concrete. The two studies [26,27] probed the consequence of in-filling of air-voids by the deposition of ettringite crystals. The degree of in-filling is reported to be associated with the duration of moist curing coupled with wet/dry cycling, and may increase the effective spacing factor, thus possibly rendering the concrete less durable than otherwise indicated by the spacing factor.

Factors Influencing Amount and Character of Entrained Air In Freshly Mixed Concrete

General

Mielenz et al. [22] and Bruere [16,28–30] have made significant contributions to the understanding of the mechanism by which air-entraining admixtures function and the influence of a number of different variables. More recently, Whiting and Nagi [11] have prepared a comprehensive summary of the various factors–concrete materials and mix design, handling operations, construction practices, and field conditions–that can affect air content and quality. Mielenz and his co-workers dealt extensively with the origin, evolution, and effects of the air-void system in concrete. They showed that in the concentrations normally used in concrete, air-entraining admixtures are adsorbed at air-water interfaces and that the surface tension of the water is decreased about 25 %. This adsorption at air-water interfaces produces a “film” of air-entraining admixture that influences the air-retention properties of discrete air-voids formed during mixing. For some air-entraining admixtures, the

TABLE 2—Influence of Air-Void Characteristics on Resistance of Concrete to Freezing and Thawing

		Air-Void Characteristics (ASTM C 457)						
				Void Spacing Factor (L),				
Air-entraining Admixture	Air Content % Pressure Meter (C 231), %	Air Content, % C 457	Number of Voids/in.	in.	(mm)	Specific Surface, in.^{2}/in.^{3} of air	Number of Voids/in.^{3} Concrete, millions	Freezing and Thawing Cycles for 0.10% Expansion
None	1.8	1.1	0.8	0.031	(0.79)	302	0.08	19
A	6.0	4.1	4.0	0.013	(0.33)	387	0.11	29
B	6.0	4.1	4.9	0.010	(0.25)	480	0.22	39
D	5.0	3.2	3.3	0.013	(0.33)	416	0.26	82
E	5.8	3.5	5.1	0.009	(0.23)	577	0.78	100
F[a]	5.2	3.9	9.6	0.006	(0.15)	990	3.79	550

[a] A commercial air-entraining admixture meeting the requirements of ASTM C 260.

calcium salt of the active constituent in the admixture may be only slightly soluble in water. In such instances, the film at the air-water interface may include a precipitated solid or gelatinous film enclosing each air-void.

The amount and character of the air-entrained in concrete is influenced by numerous factors, some of which are: (1) concentration and type of the air-entraining admixture and its influence on surface tension; (2) use of other admixtures in the concrete mixture; (2) the fineness and composition of cement and supplementary cementitious materials; (3) amount of mixing energy (time and shear rate); (4) flow and slump of mortar or concrete mixture; (5) temperature, water-cement ratio, and water content of the mixture; and (6) gradation of the fine and coarse aggregates. Mielenz et al. [21] have theoretically concluded that both the total volume of air and the size distribution of the air-voids can change in the unhardened concrete due to interchange of air between air-voids and dissolution of air. Bruere [30], however, has shown that such changes do not take place to any significant degree in air-entrained pastes after cessation of mixing, although such interchange is of significance during the mixing process and may be for a few minutes thereafter. Moreover, the overall experience with air-entrained cementitious mixtures is consistent with the observations by Mielinz that as long as concrete in the plastic state and in some state of motion, evolution of the air-void system can occur, thus possibly accounting for both air gain and air loss.

Type and Amount of Air-Entraining Admixture

Mielenz and his co-workers theorize that the type of organic ingredient in the air-entraining admixture influences the amount and character of entrained air-voids by its effect on: (1) surface tension, (2) the elasticity of the film at the air-water interface, (3) transmission of air across the air-water interface, and (4) adhesion of the air-voids to particles of cement or aggregate. All of these factors will be operative during the mixing operation. Based on extensive field experience with the common classes of air-entraining agents, the author prepared Table 3 (cited in [11]), which associates general performance characteristics with the various types of agents.

At the same volumetric air content, different air-entraining admixtures will produce air-void systems having different specific surfaces, number of air-voids per unit volume, and void

TABLE 3—Classification and Performance Characteristics of Common Air-entraining Agents

Classification	Chemical Description	Notes and Performance Characteristics
Wood derived acid salts Vinsol Resin®	Alkali or alkanolamine salt of a mixture of tricyclic acids, phenolic, and terpenes	Rapid air generation, especially in low slump mixes. Minor air gain some air loss possible with continuous mixing. Mid-sized air bubbles. Compatible with all admixtures. Some air loss possible.
Tall Oil	Fatty acid—major component; tricyclic acids—minor	Slower air generation. Air may increase with prolonged mixing. Smallest air-voids among common agents. Compatible with all admixtures.
Vegetable oil acids	Alkali and alkanolamine salts of coconut fatty acids	Relative to wood rosins, slower air generation, both air gain and air loss possible with continuous mixing. Small to mid-sized air-voids, and compatible with all admixtures.
Synthetic detergents	Alkyl-aryl sulfonates and sulfates (e.g., sodium dodecylbenzenesulfonate)	Rapid air generation. Minor air loss with mixing. Coarser bubbles. Not compatible with naphthalene sulfonate-based HRWR. Applicable for cellular concretes.
Synthetic workability aids	Alkyl-aryl ethoxylates	Primarily used in masonry mortars.
Miscellaneous	Alkali/alkanoloamine acid salts of lignosulfonate Oxygenated petroleum residues Proteinaceous materials Animal tallow Saponin	Older technologies not currently used as concrete air-entraining agents.

spacing factors. Furthermore, the rate at which air-voids are entrained in various concrete mixtures can be affected by the class of air-entraining admixture. Moreover, one can expect the air-void system to be influenced by interaction between the class of air-entraining admixture and the various mixing, handling, placing, and consolidating operations.

An almost universal guide for the use of air-entraining admixtures is that an increase in the dosage rate will increase the volume of air-entrained and decrease the void spacing factor.

Chemical Admixtures

A large variety of other chemical admixtures may be used in air-entrained concrete. These include normal, mid-, and high-range water reducers, set accelerators and retarders, corrosion and ASR inhibitors, pumping aids, pigments, and shrinkage control agents (SRAs). Little information has been published that discusses the interaction of these admixtures on air content and the air-void system in concrete. Whiting and Stark [31] note that chemical admixtures such as water-reducers, retarders, and accelerators may increase air content somewhat when used in normal, recommended dosages, requiring adjustments in dosages of air-entraining admixtures. This would especially apply to lignosulfonate-based water reducing admixtures and polycarboxylate-based superplasticizers. The authors state further that high-range water reducers may possibly result in an altered void size distribution of the entrained air, manifested by higher void spacing factors than those normally considered acceptable for adequate resistance to freezing and thawing. Nevertheless, excellent freeze-thaw durability has been obtained both in laboratory and field concretes with these admixtures. Guidance on how to assure acceptable air-void systems in concrete admixed with SRAs has been reported by Berke et al. [32]. Several studies [33–36] involving high-performance concrete mixtures, with and without superplasticizers, found that superplasticizers can both increase and decrease air contents with the spacing factor either remaining unchanged or increasing slightly; moreover, the various superplasticizers tested affected air-entrainment differently. Several reports [37,38] found that the use of viscosity modifying agents in air-entrained concrete has no significant effect on freeze-thaw durability, suggesting minimal impact on air-void quality.

Fine Aggregate

Changes in grading of sand may alter the volume and nature of air in the mortar [39]. An appreciable increase (>3 %) in the quantity of sand particles passing the No 200 sieve (75 μm) will decrease the amount of entrained air, and consequently require higher doses of air-entraining agent to achieve specified air contents [11]. The maximum and median size of the individual air-voids may decrease [40]. Sand in the middle fractions, No 30–100 (600–150 μm) is most effective stabilizing entrained air. Sand gradation is of more importance in leaner versus richer mixes, where the influence of gradation on entrained air is not as marked. Other aggregate properties that can affect the performance of air-entraining admixtures include surface texture and shape and organic contaminants [11,41]. On the one hand, air stability can be enhanced with angular sand particles, but decrease when air-entraining admixtures adsorb on rough and cracked surfaces. Nonpolar contaminants such as oils can decrease air content, whereas oxidized decayed vegetable matter can have the opposite effect.

Cement

As the cement content increases, the air-entraining potential of an admixture will tend to diminish, and an increase in the fineness of cement will result in a decrease of the air-entrained in the mortar [42–44]. Some regular (non-air-entraining) cements naturally entrain more air than others, and these require less air-entraining admixture to develop a given mortar air content. Soluble alkalis in cements will influence the required amount of air-entraining admixture dosage [11,45]. At fixed dosage levels, higher air contents will be obtained with higher alkali cements. Therefore, if more than one cement is being used and soluble alkali contents differ significantly, care must be exercised to adjust dosages when necessary. The ASTM C 185 mortar test can be useful to detect possible changes in air content associated with cement properties.

Water

An increase in water-cement ratio is likely to result in an increase in air content. Although the volume of air-entrained may increase, the specific surface of the air-voids generally decreases and the void spacing factor increases [21]. Nondegradable detergents present in water can result in excessively high and variable air contents. When air-entraining agents are added to recycled wash water or potable water with relatively high hardness, the active components may partially precipitate, thus requiring an increased dosage to achieve target air content.

Fibers

Synthetic and steel fibers may possibly increase air content if treated with surface-active coatings.

Workability and Flowability

Within the normally used range, increase in initial slump up to approximately 6–7 in. (150–175 mm) is accompanied by an increase in air content in concrete mixtures [11,15,42,44]. At higher slumps (Fig. 1), the viscosity of the mix becomes insufficient to retain the air-voids and prevent coalescence [11]. Furthermore, when retempering water is used to restore workability to the target slump, air contents will most often increase with a far lesser probability of some air loss. Work by Klieger

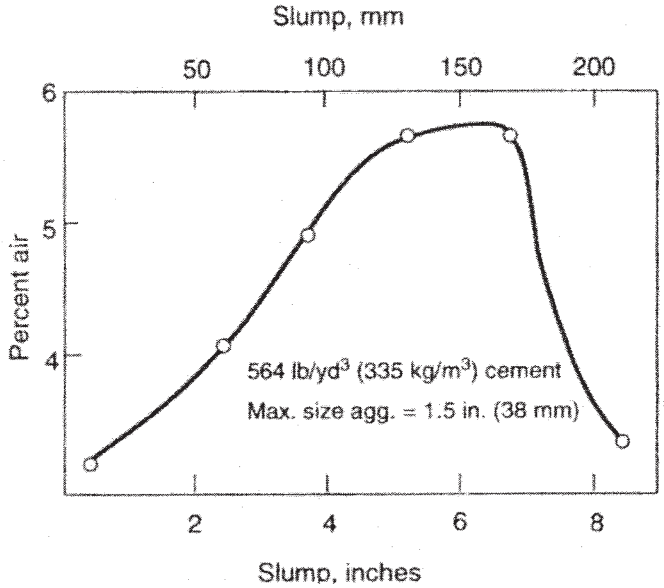

Fig. 1—Effect of slump on entrained air content (Ref 11).

[23] indicates that the optimum mortar air content remains at about 9.0 %. The impact of slump on air generation can be most significant at the far ends of the workability range for modern day concrete operations, namely, no slump and self-consolidating concrete mixtures. With no slump concrete (as in the case of paving and extruded concrete applications), the use of up to ten times the normal dose of air-entraining agent is not uncommon to achieve target air contents. In one study [46], considerable irregularly-shaped, entrapped voids can be included in the entrained air-void system from compaction operations. As indicated in Table 3, field experience has found the Vinsol Resin®, gum rosin, and wood rosin-based air-entraining agents to be more effective for these applications. With regard to self-consolidating (SCC) and self-leveling (SLC) concrete mixtures, a number of investigators [47–52] have confirmed that these highly workable mixtures exhibit comparable freeze-thaw durability to similar corresponding mixtures with lower workability. Khayat et al. [49,50] have cautioned that entrained air can lower the viscosity of SCC mixtures thus increasing the risk of segregation and the coalescence of air bubbles. The use of viscosifying admixtures or fillers such as limestone can restore the required viscosity to minimize these potential concerns.

Supplementary Cementing Materials

Supplementary cementing materials, such as pozzolans (fly ash, silica fume, etc.) and ground granulated blast-furnace slags, generally require increased amounts of air-entraining admixtures to attain the proper volume of entrained air [53–55]. Some ASTM C 618 class C ashes may actually lower the dosage of air-entraining admixture dosage. Klieger and Gebler [56] called attention to the effect of certain fly ashes in reducing the stability of the entrained air-void system as a function of time after mixing. Further details concerning this phenomenon and techniques for control are described in an ACI Committee Report [57]. In order to minimize unexpected changes in air content due to changes in fly ash properties such loss-on-ignition, several screening tests have been proposed to predict relative changes in the dosage of air-entraining agent. The “Foam Index Test” [6], which has become a common practice by many concrete producers, is a relatively simple procedure involving adding increments of an air-entraining agent to an aqueous slurry of fly ash until a stable foam is produced. A reported improved version of the test, which claims improved predictability to concrete and less variability, calls for using a reagent-grade surfactant and including cement in the fly ash slurry [58]. Other investigators feel that fly ash uniformity and changes in air content are possible by measuring the surface area of the carbon content in fly ashes [59]. Metakaolin, unlike silica fume, is reported to have no significant effect on air-entraining agent dosages to achieve a target air content [11].

Batching: Sequence of Material Addition

The air content of concrete mixtures can be affected by the order of material addition to a truck mixer. Although higher air contents have been reported when the air-entraining admixture is added after the cement has been batched [11], lower values are also not uncommon. Preferably, air-entraining agents should be dispensed separately from the other chemical admixtures. A common mode of batching is adding the air-entraining agent to the sand either contained in the weigh hopper or on the conveyor belt. Alternately, the air-entraining admixture can be introduced near the discharge of the water line prior to the water reducers and the addition of cement and supplementary cementitious materials.

Mixing

The amount of air-entrained can vary with the type of mixer, condition of blades, mixing speed, and mixing time [11,15,60]. The amount of air-entrained by any given mixer will decrease appreciably as the blades become worn, or as the mixing action is impaired if hardened mortar is allowed to accumulate in the drum and on the blades. An increase in entrained air will occur if the mixer is loaded to less than rated capacity, and a decrease will result from overloading the mixer. A central drum stationary mixer, a paving mixer, and a transit mixer may develop significant differences in the volume of air-entrained in a given concrete mixture. The air content will increase with increased time of mixing up to about 2 min in central stationary [15] or paving mixers (and up to about 15 min in some transit mixers), after which the air content may remain approximately constant for a considerable period before decreasing. The reduction in air may result from an increase in very fine particles in the mixture with prolonged mixing action, from an increase in the ratio of air-escape to foam-generation in the latter portion of the mixing period, from adsorption of the chemical by unburned carbon in fly ashes, or from adsorption of the chemical on rapidly hydrating aluminate phases of the cement. The air-void system, as characterized by specific surface and spacing factor, does not usually appear to be harmed by prolonged agitation. Different air-entraining admixtures may require significantly different mixing periods to reach maximum and constant air content. In some cases, air contents may possibly increase with prolonged mixing, especially when the concrete is re-tempered with water or normal and high range water reducing agents at the jobsite.

Temperature

For a constant amount of air-entraining admixture, less air will be entrained at 100°F (38°C) than at 70°F (21°C) and more will be entrained at 40°F (4.4°C). In other words, everything else being equal, air-entrainment varies inversely with temperature [15,43]. One general rule of thumb, an approximate 30 % increase in air-entraining agent dosage would correspond be required for a roughly a 30°F (15°C) change in concrete temperature. Furthermore, the rate of air content loss increases with higher slump [11]. Changes in concrete temperature do not generally affect the hardened air-void system, as long as the air content remains unchanged by adjusting the air-entraining agent dosage.

Vibration

Intensive internal vibration applied to freshly mixed concrete will cause air-voids to rise to the surface and be expelled. The larger natural voids are most readily expelled [21,61]. Moderately small air-voids may tend to work upward if the vibration is intense and prolonged. There is increasing evidence, however, that the critically important spacing of small entrained-air-voids in the matrix is not significantly disturbed, even by intense vibration. If vibration is applied as required, with just enough intensity and duration to effect consolidation, and if the mixture is designed properly, removal of the effective portion of the entrained air will not occur. The concrete slump will influence the extent to which vibration can alter air content [11]. For concrete slumps above 5 in. (125 mm), significant air loss (up to 4 %) can occur after 30 s of vibration. In some instances, externally applied vigorous vibration may cause an increase in air content. However, in this case, the added air is in the form of relatively large natural voids. As a general guide for slip-form operations, a vibration frequency of 8000 vpm is recommended for paver speeds greater than 3 ft/min (0.9 m/min), with lower frequencies at lower paver speeds.

General Comments

One of the most frequent and pronounced causes for variations in air contents may possibly result from variation in the amount, type, or condition of the air-entraining admixture. This last cause, as with many of the other variations in concrete materials and production practices, is a function in turn of the alertness and adequacy of the control and inspection given the work. Since air-entraining admixtures are generally incompatible with other admixture types, care must be exercised to prevent them coming in direct contact while being dispensed into the concrete during mixing.

Any influence that would maintain or even actually improve the distribution of the air-voids (number, or spacing factor) within the paste fraction or increase the ratio of air boundary surface to air volume (specific surface) would be desirable.

Methods for Determining Air-Void Characteristics

Freshly Mixed Concrete

Up until recently, only the total air content could be determined in freshly mixed concrete either by the gravimetric method described in ASTM Test Method for Unit Weight, Yield, and Air Content (Gravimetric) of Concrete (C 138), the pressure method described in ASTM C 231, or the volumetric method (ASTM C 173), which is most useful for determining the air contents of concretes made with lightweight aggregates. With the introduction of the AVA (the so-called “air-void analyzer”) instrument [62,63], originally developed by European researchers in the early 1990s, both total air content and some indication of the quality of the air-void distribution (specific surface and spacing factor) can be estimated while the concrete is still in the plastic state. The method calls for sieving the mortar fraction from a concrete mixture, and injecting into a viscous fluid contained within a column. The viscous fluid retains bubble size, and allows the bubbles to rise and contact a buoyancy recorder. Migration rate of the air-voids is a function of bubble size, where the larger bubbles rise faster than smaller ones. The change in buoyancy is recorded as a function of time, which can be related to bubble size distribution. While this technique holds some promise as a means of applying additional quality checks on air-entrained concrete prior to placement, extensive correlation of results with the established ASTM C 457 method needs to be performed before the AVA method can be recommended for acceptance testing. Furthermore, the delicate buoyancy measurements are intolerant of vibration and temperature fluctuations; and thus the AVA, as currently designed, is not expected to be operated under field conditions.

Regarding the ASTM C 231 Pressure Air Meter method, this procedure is generally adequate for use with all ordinary types of mortar or concrete mixtures containing reasonably dense aggregate. Large errors may be introduced where highly vesicular or porous aggregates are used due to the inability of differentiating between the air in the aggregate particles and the entrained air in the paste. The volumetric method described in the C 173 Test Method for Air Content of Freshly Mixed Concrete eliminates the possibility of significant errors in differentiating between air in the aggregate particles and air in the paste. However, this method is time consuming and yield inaccurately low air content readings due to insufficient time to expel the air.

Specifications and control tests will continue to be primarily based on the volume of air-entrained in the concrete, rather than on the size and distribution of the air-voids in the cement-paste matrix, until techniques such as the AVA can reliably indicate air-void parameters directly on the freshly mixed concrete in the field. Until such a test has become accepted by standards organization such as ASTM, concrete producers and specifiers will continue to rely on the total air contents in the range of the optimum air contents shown in Table 1, and will be assured that the size and distribution of air-voids will provide adequate resistance to freezing and thawing when produced by air-entraining admixtures conforming to the requirements of the ASTM C 260 Specification for Air-Entraining Admixtures for Concrete.

Hardened Concrete

The important characteristics of the entrained-air-voids can most readily be determined in hardened concrete by microscopic examination of sawed and ground surfaces of a sample of the hardened concrete. ASTM C 457 is a procedure for determining the total air volume, specific surface, and spacing factor of air-voids by either a linear traverse method or a modified point-count method. Additional refinements of the linear traverse equipment enable the measurement of chord-size distribution of air-voids from which the total number of air-voids per unit volume of concrete can be calculated, as shown in Table 2.

Mielenz and his co-workers [21] show the results of measurements of air-void characteristics of cores taken from a wide variety of structures. Although such measurements are time consuming, they can provide reassuring evidence of the effectiveness of the air-entraining admixture in providing the desired air-void system. As an improved means of better ascertaining the accuracy of spacing factor to predict the distance water must travel to the nearest air-void, a computer program was used to place nonoverlapping spheres, representing air-voids, in a cubic cell of cement paste [64]. Points in the paste were then chosen at random and the distance to the nearest air-void surface tabulated for each point, thus providing a spatial distribution. Attiogbe [65] feels that the air-void parameters as measured and calculated by ASTM C 457, specifically a single spacing factor, do not correlate well with ASTM C 666 freeze-thaw data because the predicted air-void distribution does not adequately represent the actual random distribution of air-void. A mean spacing model for air-voids is proposed whereby three zones of durability—probable, intermediate, and nondurable—can be predicted.

Progress has been made with various automated techniques for measuring the air-void parameters in hardened concrete [66–69]. One of the more promising methods is based on the RapidAir 457 Automated-Air-Void-Analyzer, which consists of comprises a computerized control unit (PC) with a 19 in. color monitor, a video camera, and a microscope objective mounted on a moving stage. Following the traditional grinding and polishing steps of ASTM C 457, a contrast enhancing technique (described in EN 480-11) is used to obtain a surface of the concrete plane section where air-voids are bright white, and the rest of the surface is black. The plane section is mounted onto the moving stage placed under the video camera. After scanning, the air-void parameters are immediately calculated. The accuracy of the RapidAir system was verified recently by an extensive round robin test program conducted by 13 European laboratories [69].

Status of Current Specifications and Test Results for Air-Entraining Admixtures

During the early 1940s, the size and distribution of the air-voids in concrete mixtures was recognized to be a major factor with respect to the effectiveness of the entrained air in enhancing durability. Since there was no ready and reliable means for determining these air-void characteristics and since there was a need to evaluate the influence of these air-entraining materials on other concrete properties, a performance-type specification (ASTM C 260) was developed in 1950 by ASTM. It remains essentially the same as it appeared initially.

ASTM C 260 currently evaluates the effects that any given air-entraining admixture under test may exert on the bleeding, compressive and flexural strength, resistance to freezing and thawing, and the length change on drying of a concrete mixture, all in comparison with a similar concrete mixture containing a reference air-entraining admixture, neutralized Vinsol Resin®. The methods by which these effects may be tested are given in ASTM Methods of Testing Air-Entraining Admixtures for Concrete (C 233). The criteria of ASTM C 260 afford assurance that if, under the conditions of the specified mixtures and conditions in ASTM C 233, the particular sample of the admixture under test exerts satisfactory influence on certain properties of the laboratory concrete, it will be reasonable to expect that the quantity of the air-entraining admixture represented by the sample will develop satisfactory air-entrainment in field concrete. A recent modification to ASTM C 233 and C 260 eliminates the need for later age (6 and 12 month) compressive strength tests, which have been found from statistical analysis of numerous C 260 certification reports to have no additional significance versus the 28-day results.

The testing required by ASTM C 260 reduces simply to a very indirect method of determining whether: (1) the particular admixture under test will produce relatively stable air-voids that will become widely dispersed throughout the matrix of field mortar or concrete so as to produce an air-void system having the proper characteristics for enhancing durability; and (2) that the admixture contains nothing that will have a deleterious effect on other properties of such mixtures. This specification, with its attendant test methods, has provided a means for evaluating air-entraining admixtures on a performance basis. The wide variety chemically of materials that can function as satisfactory air-entraining admixtures precludes the inclusion of chemical requirements.

A further consideration that is receiving attention is the possibility that the freezing and thawing tests, which are probably the most costly and time-consuming part of the testing procedure, can be supplemented by an examination of the characteristics of the air-void system produced by the admixture under test and a comparison with the system produced by the reference admixture. The CSA Standard A23.1, Concrete Materials and Methods of Concrete Construction (Canada), has adopted this alternative and considers the concrete to have a satisfactory air-void system if the average of all tests of the hardened concrete shows a void spacing factor not exceeding 0.009 in. (0.23 mm) and no single test greater than 0.010 in. (0.26 mm).

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Chemical Admixtures

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Preface

THE FIRST EDITION OF THE CHAPTER ON "CHEMICAL Admixtures" was issued in 1966 and was prepared by Dr. Bruce Foster [1]. The chapter in *ASTM STP 169B* [2], published in 1978, was an updating of Dr. Foster's paper prepared by Bryant Mather. In 1994, the chapter was again updated by Bryant Mather and appeared in *ASTM STP 169C* [3]. In the preparation of this chapter, the contents of the previous editions were drawn upon and some duplication of information from the previous edition exists. We acknowledge the authors of the previous editions and their contributions to the literature on this topic. The current edition will update the topics addressed previously, provide up to date references, and focus primarily on new technologies that have been developed. The review period has been limited to contributions made during the last decade. Attempts have been made to uncover all relevant research, but undoubtedly, important research may not be covered or may be unknowingly omitted in this review.

This discussion is limited to certain features of chemical admixtures regarded as most appropriate to the scope of this volume. For a more comprehensive review of knowledge in this field, reference should be made to the following additional works:

a. In 1991, the American Concrete Institute (ACI) published its most recent report of its Committee 212 on Admixtures for Concrete, entitled "Chemical Admixtures for Concrete" (ACI 212.3R-91) [4].
b. In 1993, Committee 212 published its most recent report on Superplasticizers for Concrete, entitled "Guide for the Use of High-Range Water-Reducing Admixtures (Superplasticizers) in Concrete" (ACI 212.4R-93) [5].
c. In 1995, V. S. Ramachandran published the second edition of the Concrete Admixtures Handbook: Properties, Science, and Technology [6].
d. In 2002, the Portland Cement Association published the fourteenth edition of *Design and Control of Concrete Mixtures*, where Chapter 6 discusses Chemical Admixtures [7].
e. In 2003, ACI published the most recent report of its Committee E-701 on Materials for Concrete Construction, entitled *Chemical Admixtures for Concrete* (ACI E4-03) [8].

Introduction

Most chemical admixtures react chemically with the cement in concrete. The reports of unfavorable behavior of some admixtures with certain cements and under certain conditions of use are counterbalanced by a record of successful use under controlled conditions in many concreting operations. However, when experience with specific admixture-cement combinations under similar job conditions is not available, tests with specific materials should precede a decision for use in construction.

The use of chemical admixtures has become an integral part of everyday concrete production. Newer levels of concrete performance are now possible that previously were not [9–12]. Economic benefits are available for the concrete producer by the use of chemical admixtures, creating a win-win situation for manufacturer and user [13].

Many of the admixtures currently have a valid ASTM specification (refer to the 2003 edition of the *Annual Book of ASTM Standards* [14–22]) by which to demonstrate conformance against. Many of the newer admixtures, though, do not and various levels of activity in committee are underway to develop them. It seems prudent that this document should discuss both categories of chemical admixtures, as both are currently used in the field.

Types of Materials and Their Action in Concrete

Admixtures Currently With an ASTM Specification

Water-Reducing and Set-Retarding Admixtures

Water-reducing admixtures may be used in at least three different ways: (1) to produce concrete with a lower water to cementitious materials ratio (w/cm); with no change in cement content or slump; (2) to produce a higher slump, with no change in cement content or w/cm; or (3) to produce concrete with reduced cement content, with no change in w/cm or slump. In the first case, the usual benefits accruing from the use of a lower w/cm normally will be obtained, and, in many

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cases an increase in strength greater than that normally produced by the reduction in w/cm alone may result. In the second application, easier placing of concrete may be obtained. In the third application, a reduced cost should result.

Set-retarding admixtures may be used in at least two different ways: (1) to delay the time of setting, and (2) improve compressive strength development. In the first case, the benefits are typically realized when concrete temperatures are greater than room temperature. In the second case, the benefits are from changes in the chemical hydration and not necessarily from additional water reduction.

Materials used as conventional set-retarding and/or water-reducing admixtures are some of the following: (1) lignosulfonic acids and their salts; (2) modifications and derivatives of lignosulfonic acids and their salts; (3) hydroxylated carboxylic acids and their salts; and (4) modifications and derivatives of hydroxylated carboxylic acids and their salts. In each of these, the primary component has both water-reducing and set-retarding properties. In the formulation of products of Classes 2 and 4, these admixtures may be modified by the addition of other components to give various degrees of retardation, no significant change in setting time, or acceleration, while at the same time preserving the water-reducing properties. They also may be modified by the addition of an air-entraining admixture. The water reduction resulting from the use of conventional water-reducing admixtures is typically from 5–12 % [8]. A part of the water reduction found with lignosulfonate water-reducers may be the result of the additional air entrained by these materials. In addition to varying with the particular cement employed, the amount of water reduction by a given admixture is also influenced by dosage, cement content, type of aggregate, and the presence of other admixtures, such as air-entraining agents or pozzolans. Water-reducing admixtures are effective with all types of portland cement, portland blast-furnace slag cement, portland-pozzolan cement, and high-alumina cement.

The extent of retardation in time of setting caused by retarders depends on the cement characteristics, the temperature, the admixture dosage, and other factors. Overdosage may produce times of setting in excess of 24 h or more, but in such cases, if the concrete finally sets and has been protected from drying, ultimate strengths developed may be satisfactory if forms are left in place for a sufficient length of time. Severe overdosage of lignosulfonate admixtures may produce excessive air contents and consequently reduced strength. Lignosulfonate water-reducing retarders usually entrain 2–3 % of air when used in normal dosages. The hydroxylated carboxylic admixtures do not entrain air. However, both classes of material enhance the effectiveness of air-entraining admixtures from the standpoint of volume of air produced, so that less air-entraining admixture may be required when added to concrete containing one of these other admixtures. It is important to note that while the air-void spacing obtained with water-reducing retarders is slightly greater than that for an equivalent amount of entrained air produced by typical air-entraining admixtures, the performance of concrete containing water-reducing retarders, as measured by freezing and thawing tests, often has been found to be better than concrete of the same air content, but without the water-reducing retarders. This increase might be the result of the reduction in w/cm.

Contrary to expectations, water-reducing retarders usually have not been found effective in reducing slump loss resulting from substantial delays in placing mixed concrete. The use of retarders with some cements may actually produce an early stiffening. The outcome may be highly dependent on the chemistry of both the cement and the set-retarding admixture used [23].

The effects of molecular weight fractionation on the retardation and dispersing efficiency of lignosulfonates have been reported [24–26]. Reknes and Gustaffsson [24] found that the high molecular weight fractions of a softwood sodium lignosulfonate obtained from the sulfite process gave better dispersing efficiency and slump retention, as well as less retardation, than the low molecular weight fraction. Maximum achievable water reductions were increased from 10–20 %. Zhor and coworkers [25,26] reported investigations on a methylsulponated organosolv lignin that was also fractionated. They found that the minimum level of retardation occurred at the maximum molecular weight, but an optimum in dispersing performance was observed in the middle of the molecular weight range.

Accelerating Admixtures

Accelerators have their primary application in cold weather concreting. They may be used to permit earlier starting of finishing operations. They reduce the time required for curing and permit earlier removal of forms because of more rapid early-strength development.

The accelerating admixture that is most widely used is calcium chloride [27]. The addition of recommended amounts of calcium chloride, which are typically 1–2 % by cement weight, has been found to reduce the water requirement by a small amount over that required to produce the same slump with no calcium chloride added. The magnitude of the effect of calcium chloride on time of setting depends on the dosage, the particular cement used, the temperature, and other factors. The recommended maximum dosage has a substantial effect on time of setting at normal temperatures and can produce a very rapid set at high temperatures, as is also the case with a very large dosage at normal temperatures.

Calcium chloride does not entrain air but its use enhances the effectiveness of air-entraining admixtures so less air-entraining admixture is required for a given air content. Calcium chloride may result in early stiffening and in many cases, therefore, is not added until after mixing has commenced. Calcium chloride used at maximum dosages can affect the color of the hardened concrete.

The addition of calcium chloride to reinforced concrete has the potential to promote corrosion. As such, other non-chloride accelerators are used in applications where corrosion of embedded steel is a concern. Common materials are inorganic sodium or calcium salts of nitrites, nitrates, thiocyanates and thiosulfates [6], of which thiocyanates are the only class with a potential for promoting corrosion. At the dosages used in commercial accelerators, though, they have been shown not to promote corrosion [28]. Alkali metal salts are very rapid accelerators and often used in shotcreting applications [29,30]. Lithium-based salts are commonly used to accelerate calcium aluminate cements [31]. Other materials are organic accelerators, such as triethanolamine or calcium formate.

Nonchloride accelerators, such as nitrites, nitrates, and thiocyanates are most effective at temperatures below 21°C, performing much better at 5–10°C [6,32,33]. ASTM C 494 specification for Type C admixtures requires testing at nominally 21°C, which does not fully elucidate the effectiveness of these materials. Consideration should be given to testing these admixtures under more appropriate conditions [34].

High-Range Water-Reducing Admixtures (HRWR)

High-range water-reducing (HRWR) admixtures were added to ASTM C 494 in 1980 and their use to produce flowing concrete is covered by ASTM C 1017, adopted in 1985. They have been used to produce high-strength concrete by taking advantage of their ability to substantially lower the w/cm. Alternatively, they can be used to produce "flowing" concrete (200 mm slump) or self-consolidating concrete (550–750 mm slump flow) discussed in Chapter 58. Early generation HRWRs based on melamine or some versions of naphthalene generally had poor workability retention, often requiring addition of the HRWR at the job site. New generation HRWRs often maintain adequate workability retention over time, thereby they may be added at the batch plant.

The addition of HRWRs to air-entrained concrete may increase the spacing factor and decrease the specific surface area of the air-void system [5]. However, a reduction in the resistance of such concretes to freezing and thawing has not been observed, provided the spacing factor is generally not more than double the maximum recommended limit [35]. Some reduction in resistance to salt scaling has been observed with concrete exhibiting increased spacing factors [7], but on other occasions concrete with poor air void systems has performed adequately [36]. Nevertheless, it would be prudent to evaluate the effect of a specific high-range water reducer on the frost resistance of a concrete mixture if this is a significant factor for the application of the concrete.

Seven CANMET/ACI Symposia have been held on the topic of Superplasticizers and Other Chemical Admixtures to date [37]. This document focuses exclusively on the developments that have been discussed during the last four meetings, which covers the period from 1994 to 2003.

The most significant new development in HRWRs in the last decade has been the introduction of a class of materials known as polycarboxylate ethers (PCE) [38–50]. These polymers consist of an ionic backbone grafted with pendant nonionic side chains. These polymers provide dispersion by means of both electrostatic and steric repulsion, the latter of the two believed to be the dominant mechanism [51,52]. As a result, these polymers are highly efficient dispersants, typically 2 to 3 times more effective on a mass basis than naphthalene or melamine-based HRWRs, and exhibit excellent workability retention. Water reduction is essentially linear with increasing dosage of polymer and can exceed 40 %. Another advantage of this dose efficiency is the lack of retardation with these polymers, since less of the surface of the cement grain is covered with adsorbed polymer.

Another key aspect to the performance benefits of the PCEs is the ability to tailor the polymer to the needs of specific applications. Modifications to conventional HRWRs are limited primarily to changes in overall molecular weight or substitutions of other monomers [53,54]. Variations to the structures of PCEs, on the other hand, are considerable and often result in a performance difference when used in concrete [55–57]. Variations in length and graft density of the side chains, charge density in the backbone, and molecular weight of the overall polymer are all examples of molecular modifications that can affect such performance characteristics as early strength development, time of setting, workability retention and viscosity of the cementitious system. Additional functional chemistries, such as shrinkage-reducing admixtures, can be grafted to the polymer to provide further enhanced performance [58].

The primary disadvantage of the PCEs is a propensity to entrain some amount of air in the absence of an air-entraining agent. The amount of air entrained increases with increasing dosage, similar to a lignosulfonate. This is the result of the surfactant-like structure of the polymer, namely a hydrophopic backbone with hydrophilic side chains. To offset this deficiency, many commercial products are formulated with defoamers or other components to minimize air entrainment.

Admixtures Currently Without an ASTM Specification

Mid-Range Water-Reducing Admixtures (MRWR)

Mid-range water-reducing (MRWR) admixtures were first introduced to the market in the mid-1980s [59,60]. The first generation products contained lignosulfonate as the primary dispersing component. The retardation, which typically increased as the dosage of water reducer increased, was offset by the additions of the appropriate amount and type of accelerating additives. Newer generation admixture formulations may contain polycarboxylate ethers as the primary dispersants. It is possible to achieve water reduction levels of 5–12 % or more with these admixtures or slump levels of 125–200 mm. MRWRs are also noted for the improvements they provide to the workability of the concrete. Pumping pressures have been shown to decrease by the addition of these admixtures [59]. The finishing characteristics of the concrete containing MRWRs are improved, making these admixtures highly desirable for flat work applications [61].

At this time, there is no ASTM specification for MRWRs. Most are certified to meet the requirements of a Type A, whereas some are able to meet the requirements of a Type F. There was considerable effort put forth in ASTM Committee C09.23.3 in the late 1990s and into the early 2000s to develop a specification for these types of admixtures. First efforts were to develop a modified version of ASTM C 1017, where the target slump was adjusted down from 215 mm (±25 mm) to a value of 150 mm (±12 mm). The time of setting and compressive strength of the MRWRs were compared against that of a Type A water reducer. It was found that it was not possible to adequately differentiate the MRWRs from a Type A based on these characteristics. The next efforts were again to use an approach based on ASTM C 1017, where the starting slump was decreased from 100 mm to 50 mm and increasing dosages of the admixtures were used to increase the slump while monitoring the time of setting. This was continued until differences in the time of setting as a function of slump increase were observed. No differences in setting time at a fixed slump increase were observed until the retardation levels were on the order of 2.5 h beyond a control, which were well beyond the 1.5-h limit in the standard. The final efforts focused on using the differences in finishing characteristics as a means of distinguishing the MRWRs from Type A water reducers. Data collected using a finishing machine [61] were inconclusive and the pursuit of a specification was abandoned.

Corrosion-Inhibiting Admixtures (CIA)

Steel reinforcing bars embedded in concrete form a passive oxide film, due to the high pH created by hydroxyl ions. This oxide film can be destroyed by a sufficiently high concentration of chloride ions or because of a decrease in pH from carbonation, resulting in corrosion. The products of the corrosion reactions create a volume expansion, causing cracking and spalling of the concrete structure. Reduction in chlorides or water ingress can be achieved by a number of approaches. Addition of mineral admixtures decreases the permeability of the concrete internally, while surface sealers or membranes will limit penetration at the surface. Neither of these approaches may be sufficient, though, if a crack forms from the surface, providing a low resistance pathway for the chloride ions to the steel.

Chemical admixtures are another means for delaying or eliminating the onset of corrosion in concrete structures. HRWRs can reduce the w/cm, thereby decreasing the permeability of the structure. In addition, the use of corrosion-inhibiting admixtures (CIA) has been shown to be effective [62–65]. One classification method is to define the CIAs by their chemical nature, namely whether they are inorganic, organic, or vapor-phase. The more common method is to classify them by the electrochemical reaction that they predominantly affect, which are the anodic or cathodic reaction, or both (mixed). Some CIAs can be applied topically [65,66], though the majority of the literature discusses the use of integral CIAs.

Calcium nitrite is one of the most widely used CIAs and is believed to act as an anodic inhibitor [70]. Typical dosages range from 5–30 L/m^3 of concrete for a 30 % solution. It also accelerates hydration and must be used in combination with a retarding admixture to achieve acceptable times of setting under summer conditions. It has been reported to meet the requirements for an ASTM C 494 Type C – Accelerating Admixture [67].

Alkanolamines are also used in formulating CIAs [65]. Specific details are not readily available in the literature, as these compounds are components in many proprietary CIA formulations. Brown et al. reported on the performance of these materials [68,69] and compared them to calcium nitrite and other organic CIAs.

Nmai et al. [70] discussed an organic-based CIA, consisting of a mixture of amines and esters in a water medium. The admixture was characterized as a mixed inhibitor and has a recommended dosage of 5 L/m^3 of concrete. It imparts corrosion protection by a dual mechanism: (1) inhibition by amine technology already used in the petrochemical industry, and (2) protection by film formation on steel reinforcing bar from fatty esters. Tests with precracked concrete beams subjected to chloride ponding showed significantly longer times to corrosion for samples treated with this organic inhibitor than those treated with calcium nitrite.

Tourney and Berke [71] discussed the need for an ASTM specification for a corrosion-inhibiting admixture. Their contention was that specifications like ASTM C 494 are designed primarily to demonstrate harmlessness and do not evaluate the performance of the CIA for what it is claimed to do—delay or prevent corrosion. In 2003, the final form of a specification was approved at the main committee of C09 and is due for publication in 2004. A key aspect of the specification is the requirement that the amount of chloride required to initiate corrosion must be higher in the presence of the CIA than in its absence. Therefore, materials that only slow the rate of chloride ingress will not meet the specification.

Admixtures for Suppressing Alkali-Silica Reactions

The deleterious effects of alkali-silica reaction (ASR) are well known [72–89] and expounded on in Chapter 34 of this volume. In short, the reaction requires two main components: (1) reactive siliceous aggregate, and (2) a sufficient concentration of hydroxyl ions, i.e., high pH. Since sodium and potassium oxides are the main species affecting the pH of the pore solution, the latter requirement is met by the presence of soluble alkalis. Sources of the alkali can be internal—cement, aggregate or pozzolan, or external—seawater, deicing salts or the like. The alkali-silica reaction itself is not destructive. The product of the reaction, though, is a gel that is susceptible to swelling in the presence of water. Due to the low tensile strength of concrete, the expansion forces generated by this swelling can result in cracking and spalling [73].

Various strategies have been employed to mitigate or suppress ASR [72]. The first is to eliminate reactive aggregates from the cementitious mixture. Good quality aggregate is not available in many regions and it is often economically or physically impractical to ship in appropriate aggregates. Another approach is the use of mineral admixtures, namely low-alkali pozzolanic materials like metakaolin, class F fly ash or silica fume, as well as hydraulic materials like ground granulated blast furnace slag. All have been shown to provide reduction in expansion [72]. Class F fly ash has been stated to be the most cost effective approach, but may not completely mitigate ASR in some instances [72].

Two primary classes of chemical admixtures are discussed in the literature to mitigate ASR. The first is the use of air-entraining admixtures (AEAs). This approach is somewhat effective but has created concerns that the air voids cannot provide sufficient space for both the expansive gel and an advancing ice front when used in freeze-thaw environments [72,74]. The second is the use of lithium salts, i.e., LiOH, LiCl, Li_2CO_3, $LiNO_3$. There have been numerous reports on the ability of these materials to virtually eliminate expansion due to ASR over long periods of time [75–83]. The preferred salt form in most instances is $LiNO_3$. This is primarily because it does not exhibit a concentration that must be exceeded to prevent expansion, referred to as the pessimum limit. Compounds such as $LiOH_2$ will increase the pH of the pore solution and can actually cause expansion if an insufficient amount is used [84]. $LiNO_3$ has also been shown to have insignificant effects on the plastic and hardened properties of either air or non air-entrained concrete when used at the dosages typically required for ASR suppression [85].

The recommended dosage of lithium is based on the alkali content of the cement and a molar ratio of $[Li]/[Na+K]>0.7$ to 0.8 should be exceeded [77]. This translates to dosages in the range of 3–5 L/m^3 of a 30 % solution of $LiNO_3$. Combinations of class F fly ash and $LiNO_3$ have been shown to be particularly effective, both from an economic and technical perspective [72].

Yet another approach to minimize ASR is to limit the ingress of water into the structure. This is typically a strategy used for existing structures that do not contain concrete designed to address the potential for ASR. Application of lithium salt solutions to the exteriors of structures has only been marginally successful. Another approach is to dry the surface of the structure and apply a sealer to prevent additional ingress of moisture.

No ASTM specification exists at this time for these admixtures, though a task group has been formed to develop one. The primary focus of the group is to develop an acceptable test method from which to develop specifications for expansion. The current ASTM C 1260 method requires some modification, especially to prevent leaching of lithium salt from the mortar bar during testing. This is potentially being addressed by addition of lithium salt to the sodium hydroxide bath during testing [86]. Modifications to the molds are also potentially required to address issues discussed by Ong and Diamond [87]. An AASHTO method that is similar to ASTM C 1260 is of relevance, as well [88].

Viscosity-Modifying and Anti-washout Admixtures

Many applications for concrete (underwater concreting, for example) require that the mixture remain highly cohesive during transport, pumping, placing and prior to setting. Sufficient cohesion may not always be achieved by mixture proportioning alone. Concrete contains cementitious materials, which are fine powders and can therefore easily be washed out of the concrete mixture during placement in water or in the presence of moving water. In this application, the viscosity-modifying admixture (VMA) acts as an anti-washout admixture (AWA). Another application is in the production of self-consolidating concrete (SCC) discussed in more detail in Chapter 58. These mixtures are highly fluid, yet must remain stable–i.e., resist segregation in both a dynamic and static state, until hardened. Early approaches to maintaining stability and minimizing bleeding were to increase the proportion of fine materials (cement, sand, filler, etc.) in the mixture. These mixtures are more susceptible to creep and shrinkage [90]. In contrast, addition of a VMA makes it possible to use a more conventional mixture design, while still maintaining the desired stability.

Organic versions of these admixtures are based on a number of different chemistries [91–98]. One class is the natural polymers. These include such materials as polysaccharide gums (welan, xanthan, etc.), starches (potato, corn, etc.), and proteins. A second class is semi-synthetic polymers like cellulose ether-based materials (hydroxy methyl, hydroxyl propyl, carboxymethyl, etc.). A third class is the synthetic polymers like polyethylene glycols, polyvinyl alcohols, or polyacrylamides. Inorganic versions can include swelling clays (bentonite), as well as high surface area materials like silica fume or colloidal silica [99].

Some of the materials impart cohesiveness by binding water, thereby increasing the viscosity of the water phase. Others, like gums, are believed to affect the interaction between cement grains through polymer entanglement and hydrogen bonding, thereby increasing the viscosity of the mixture [91]. Yet others, such as silica fume, significantly increase the surface area of the mixture and the physical binding of the water.

Bury et al. [100] discussed a modified cellulosic polymer in the form of a fluidized suspension that binds the free water, minimizing bleeding and imparting viscosity modification. Use of the polymer as an AWA decreased the mass loss to roughly one-tenth of the amount lost in the absence of the AWA. Unlike some other cellulose ethers [101], this particular polymer exhibited little change in time of setting from the control mixture.

Khayat and coworkers have published extensively on the effects of VMAs on the plastic and hardened properties of concrete [102–108]. In one study, it was found that the use of welan gum or HPMC lowered the flexural strength by 10–30 % and gave similar or slightly better compressive strength relative to reference concrete. In another study, it was found that the resistance to freezing and thawing cycles of concrete containing VMA was acceptable. The best air void system was obtained when the AEA was added after the VMA and a HRWR were added.

At this time, there is no ASTM specification for VMAs. The need for a specification has been discussed at recent meetings, but no activity has occurred to date. Many of the materials mentioned above have been tested in a manner consistent with ASTM C 494, with no water reduction taken into account, and found to be harmless to the concrete properties.

Shrinkage-Reducing Admixtures (SRA)

One of the mechanisms of cracking in concrete is volume change due to drying shrinkage. One means of minimizing this type of shrinkage, and subsequently the tendency of structures to crack, is by the use of a shrinkage-reducing admixture (SRA). These materials first appeared in Japan in the early 1980s and the US patent literature in the mid-1980s [109]. The admixtures consist primarily of low molecular weight polyoxyalkylene aklyl ethers. They are liquids at room temperature and soluble in aqueous systems and some hydrocarbon solvents. Typically they are treated as water in the cementitious mixture, so the amount of added mix water is reduced by the amount of SRA used. Dosages of 0.75–2 % by cement weight are used resulting in typical reductions in unrestrained drying shrinkage of as much as 80 %.

The primary mechanism of action of these materials is the reduction of capillary tension inside the pores that develops during drying [110]. This tension pulls on the interior walls of the pores, resulting in shrinkage of the bulk specimen. The reduction in capillary tension is achieved by decreasing the surface tension of the water in the pores, which results by addition of the SRA to the mixtures. Pore sizes in the range of a few to tens of nanometers are believed to be the main sizes contributing to shrinkage [110].

Additions of SRAs have been shown to decrease the compressive strength of concrete at 28 days by up to 15 % [111]. This can be offset best by using a water reducer to decrease the w/cm and still maintaining workability. Depending on the specific chemistry of the SRA, a slight increase in air content of 0.5–1 % can be observed on occasion [109], as well as some retardation of time of setting [112]. It has also been reported that because these materials affect the surface tension of the pore water, they can potentially impede the ability of an air-entraining admixture (AEA) to develop an adequate air-void system [113]. As such, the resistance of concrete containing a SRA to freezing and thawing cycles can be negatively impacted. This is especially true when the SRA is used in combination with a rosin-based AEA and a naphthalene-based HRWR [114]. Kamimoto et al. [113] showed that the resistance of concrete to freezing and thawing cycles could be improved by delaying the addition of the SRA versus adding it initially to the mixing water.

Despite the lack of an ASTM specification [111] and some of the disadvantages discussed above, SRAs are now finding widespread usage in concrete applications in Japan and North America [115–117]. Shah et al. [118–120] have published a number of works on the benefits of this class of admixtures. See et al. [121] have demonstrated the benefits of an SRA using ring tests, where both the shrinkage rate and shrinkage potential were decreased in mixtures containing the admixture. Tests by the Hawaii Department of Transportation reported positive benefits in the field when used in a bridge application [122].

Cold Weather Admixtures

Accelerating admixtures have been discussed and are used to increase the rate of hydration, particularly as the concrete temperature approaches the freezing point of water. While these materials accelerate time of setting and early strength development, they do not necessarily prevent the concrete from freezing. When temperatures below the freezing point of water are experienced, additional precautions may be necessary to ensure that the concrete does not freeze and cease to gain strength. Examples of traditional approaches to minimizing the potential for freezing are: (1) heating the concrete before leaving the batch plant, (2) using higher cement contents and/or Type III cements, and (3) heating and insulating the area of placement. Option 1 can increase the potential for plastic shrinkage cracking [123], while in-place costs using Option 3 can be twice that of summer concreting [124].

Another approach is the use of cold weather admixtures (CWA), sometimes referred to as antifreeze or freezing-protection admixtures. CWAs have a dual mechanism of action: they depress the freezing point of the pore solution and accelerate hydration of the cement. Most of the chemicals that are used are inorganic salts that exhibit eutectic (easily dissolved) temperatures significantly below 0°C [6]. Organic materials such as propylene glycol are also mentioned [125].

Senbetta and Scanlon [28] discussed test results of concrete containing a CWA that would prevent the concrete from freezing at temperatures as low as 20°F (−7°C). The CWA was found to meet the requirements of ASTM C 494 for Types C and E. Because the admixture contains sodium thiocyanate, it was evaluated for its effect on corrosion of steel and was found not to promote corrosion at a dosage of 8L/100 kg of cement. Korhonen and Brook [126] documented a more comprehensive study on this commercial CWA. Similar work on another commercial admixture and a developmental product was published the following year [127].

Farrington and Christensen [128] have recently published results on a further improvement to the original CWA developed by Brook et al. [129]. Concrete was batched at 11–13°C and cured at temperatures as low as −11°C. Acceptable times of setting and early compressive strength development were possible with combinations of the CWA and a HRWR. Korhonen and coworkers [124–127,130–132] have published extensively on the performance of CWAs and the benefits of using them for cold weather concreting. Additional studies have been conducted in Japan [133], as well as field data reported by Nmai [134].

Most CWAs will meet the requirements for ASTM C 494 Type C and/or E. As such, the harmlessness of the material with regard to the parameters of the standard is demonstrated. At this time, though, there is not a specification for a CWA that demonstrates its ability to provide freezing point depression. Therefore, a new section, ASTM C09.23.5, has recently been created to oversee the development of an appropriate test method and specification for this class of chemical admixtures.

Hydration Controlling Admixtures (HCA)

The production of ready-mixed concrete results in waste from wash water and returned plastic concrete. Disposal of these materials is an economic burden for the producer, as well as an environmental concern for the surrounding community. Reuse of the material into freshly batched concrete generally results in accelerated hydration and slump loss, making its use impractical in many instances. One means of making this waste concrete reusable is with the use of a hydration-controlling admixture (HCA), sometimes referred to as a hydration-stabilizing or extended-setting admixture.

HCAs are retarding admixtures that act on all the cement phases to delay hydration, in contrast to conventional sugar-based retarders, which act primarily on the silicate phases [135]. Hydration can be arrested for weeks at a time with an HCA, if desired, and then when the effect of the HCA wears off, setting and strength development proceed as normal. Hydration that is still arrested by an HCA can be re-activated by the addition of an accelerating admixture meeting the requirements of ASTM C 494 Type C. This attribute is particularly useful for long hauls, overnight or weekend stabilization of plastic concrete. High dosages of conventional retarding admixtures, on the other hand, can permanently arrest hydration and make the concrete unusable. They can also cause rapid stiffening, exhibiting similar signs to flash or false setting.

Dosage rates of HCA, and the corresponding amounts of accelerator to reactivate, are highly variable and depend on a number of factors [136]. These include the age of the concrete, length of stabilization time required, temperature of the concrete, and time of setting required after reactivation, etc. HCAs are formulated using carboxylic acids and/or phosphorus-containing organic acids and/or their respective salt forms [6,140].

Senbetta and Scanlon [28] reported that concrete in which an HCA was used and reactivated after 18 h of storage, when tested for resistance to freezing and thawing, gave equally good results as concrete not so treated. The same HCA was studied by Senbetta and Dolch [138] for effects on paste as revealed by X-ray diffraction, thermogravimetric analysis, differential thermal analysis, scanning electron microscopy, and determination of nonevaporable water content, surface area, and pore size distribution. No significant differences between treated and untreated pastes were noted. Additional studies by Ragan and Gay [139], as well as many others [140–143], have showed the utility of these admixtures and the general lack of degradation in properties of the stabilized concrete once activated.

No specific ASTM category exists for HCAs. Most commercial products meet the requirements for ASTM C 494 Type B or Type D. No recent discussion has occurred in the ASTM C09.23 subcommittee around the need for a separate specification.

Other Chemical Admixtures

Other types of admixtures not discussed in this document include gas-forming admixtures, grouting admixtures, expansion-producing admixtures, bonding admixtures, flocculating admixtures, fungicidal, germicidal, and insecticidal admixtures, damp-proofing admixtures, permeability-reducing admixtures, and color-conditioning admixtures. Air-entraining admixtures are discussed in a previous chapter of this book, hence are not discussed here, as well.

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Supplementary Cementitious Materials

Scott Schlorholtz[1]

Preface

THIS WRITER IS INDEBTED TO PRIOR AUTHORS OF this chapter [1–4] because their work provides the background for this version. I have borrowed from their ideas and specific words. However, following the format provided by Cain [4], this chapter will present less discussion of the use of these materials in concrete and more on the characteristics of the various materials and the significance and use of their acceptance tests. For discussion of the use of these materials in concrete, the reader is referred to three reports from the American Concrete Institute (ACI): ACI 232.1R "Use of Natural Pozzolans in Concrete" [5]; ACI 232.2R-96 "Use of Fly Ash in Concrete" [6]; and ACI 234R-96 "Use of Silica Fume in Concrete" [7]. Updated reports on these materials are currently being prepared by the appropriate ACI Committees.

Introduction

This chapter will touch on the history of the use and interest in fly ash, natural pozzolans, and silica fume. It will chronicle the paths that the various specifications for these materials have taken through ASTM task groups, subcommittees, and committees. A discussion of tests and specification limits will follow at that point in the chapter.

Supplementary Cementitious Materials

Readers of this chapter will note that a change in terminology has taken place. Prior versions of this chapter all used the term "mineral admixture" throughout the title and text of the document to describe the wide variety of materials that are commonly added to concrete to increase the paste content of the mixture. These same materials will now be referred to as "supplementary cementitious materials (SCMs)" throughout this chapter. This change was needed because the ASTM Subcommittee on Terminology (C09.90) could not reach a consensus on an adequate definition of the term "mineral admixture." Hence, part of this section will be devoted to explaining the reasons for the change.

Definitions

The term admixture is defined in ASTM Standard Terminology Relating to Concrete and Concrete Aggregates (C 125-03) as follows:

admixture, *n* — a material other than water, aggregates, hydraulic cementitious material, and fiber reinforcement that is used as an ingredient of a cementitious mixture to modify its freshly mixed, setting, or hardening properties and that is added to the batch before or during its mixing.

The definition continues to list the various types of admixtures that are commonly available. The verbiage given for the term mineral admixture is as follows:

mineral admixture, *n* − deprecated term.

DISCUSSION − This term has been used to refer to different types of water insoluble, finely divided materials such as pozzolanic materials, cementitious materials, and aggregate. These materials are not similar, and it is not useful to group them under a single term. The name of the specific material should be used; for example, use "pozzolan," "ground granulated blast-furnace slag," or "finely divided aggregate," as is appropriate.

Hence, the use of "mineral admixture" has been discouraged. However, what will now be used to denote a generic reference to this broad class of apparently dissimilar materials that were all used for similar reasons? The discussion given above indicates that the specific term (i.e., "pozzolan") should be used; however, that is lengthy and awkward when referring to several materials at the same time. For example, who will pay attention when a speaker indicates that the talk will be about Class C fly ash, Class F fly ash, calcined natural pozzolans, and ground granulated blast-furnace slag rather than simply mineral admixtures? This has led to the use of another generic term for these materials. That term is supplementary cementitious material (SCM). The term is not currently defined in ASTM C 125. The American Heritage dictionary [8] gives the following definition for the word "supplement":

supplement, *n* − something added to complete a thing, make up for a deficiency, or extend or strengthen the whole.

The new terminology is not perfect but it does give a better indication of why the various materials are added to concrete mixtures. It also gets rid of the word "admixture" which is of questionable merit when high replacements (dosages) of the supplementary cementitious material are used. The overall thrust of the change in terminology appears to be a positive step forward. It gives a clearer indication that the materials are being added to supplement (i.e., strengthen) both physical and chemical properties of the mixture; and hence, the materials are not simply being used to replace portland cement. The new verbiage also pertains when the SCM is sim-

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ply being used to reduce the cost of the concrete mixture (i.e., extend).

Fundamental Properties

According to Mielenz [9], "mineral admixtures include finely divided materials that fall into four types: those that are (*a*) cementitious, (*b*) pozzolanic, (*c*) both cementitious and pozzolanic, and (*d*) those that are nominally inert chemically." They include natural materials, processed natural materials, and artificial materials. They are finely divided and therefore form pastes to supplement portland cement paste. This is in contrast to soluble admixtures that act as chemical accelerants or retardants during the hydration of portland cement or otherwise modify the properties of the mixture. The chemical compositions of the four types of finely divided materials vary widely, both within and between the various groups, and this has made categorization difficult. This is illustrated in Fig. 1. The data for the figure were extracted from a variety of historical papers and reports on fly ash [10–16]. The deprecation of the term "mineral admixture" was related to the fact that it was difficult to define. Figure 1 suggests that this may be partially due to the fact that the materials traverse such a wide range of chemical composition.

For instance, silica fume is currently specified to contain at least 85 % SiO_2. This restricts its overall (bulk) composition to the apex of the triangle shown in Fig. 1. Natural pozzolans exhibit a very wide range of chemical composition. However, they typically contain only small amounts of calcium and magnesium [17–19], and this means that they will plot very near the $SiO_2 - (Al_2O_3 + Fe_2O_3)$ axis of the triangle. Fly ashes, shown as data points in the figure, appear to break into different groups; however, there is considerable overlap between the groups. Class F fly ashes roughly plot parallel to the $SiO_2 - (Al_2O_3 + Fe_2O_3)$ axis. In contrast, Class C fly ash plots roughly perpendicular to the $SiO_2 - (Al_2O_3 + Fe_2O_3)$ axis. These general trends in bulk chemical composition fail to indicate the similarities between the various materials. However, the materials from the SCM types denoted above as a, b, and c do have a property that makes them all similar. The common property is the lack of crystalline structure–all of these finely divided materials are primarily composed of glass. This fact is illustrated in Fig. 2, where the crystalline compounds and the glass scattering halos have been labeled on the various X-ray diffractograms. This is not a new observation; rather it is an old observation that is often forgotten [20–27].

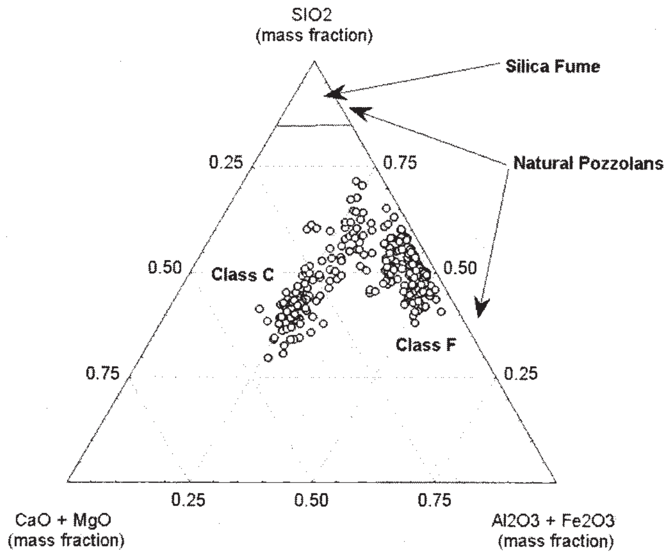

Fig. 1—Ternary diagram illustrating the bulk composition of fly ash, natural pozzolans, and silica fume.

The composition of the glass varies considerably between the materials as do the crystalline minerals that are commonly observed (refer to Fig. 2). For example, the "reactivity" of fly ash has been linked to the amount of amorphous material, the thermal history, the presence of specific crystalline components, the chemical composition of the glass phase(s), and the particle size (fineness) of the bulk material [27–29]. All of these complicating factors have made it difficult to devise a single (simple) categorization scheme that works for all SCMs. Hence, the current thrust in Subcommittee C09.24, the subcommittee that has jurisdiction over the specifications pertinent to their use, has been to push towards the use of a series of performance tests that isolate the key properties that the SCM is being used to enhance.

Cementitious Materials Not Discussed

Cementitious materials such as natural cements, hydraulic limes, portland-pozzolan cements, ground granulated blast-furnace slag, and slag cements were discussed in the previous versions of this chapter. They will not be dealt with here because specifications for those materials are no longer under the jurisdiction of ASTM Subcommittee C09.24.

Low-Reactivity Materials Not Discussed

Such materials as ordinary clay, ground quartz, ground limestone, bentonite, hydrated lime, and talc were once used to improve workability and reduce bleeding in concrete. However, they are admixtures with low reactivity. Now, more suitable and available materials such as fly ash and slag have largely taken their place for these purposes. Therefore, such materials will not be dealt with further in this chapter. Interested readers should refer to prior chapters in this series [1–3] for additional information on these types of materials.

Fly Ash and Natural Pozzolans

History and Use

Although slag cements and natural pozzolans had been used in concrete in local areas for many years, it was the use and acceptance of fly ash in concrete in the national market in the late 1940s and early 1950s that created the need for national specifications governing the use of these materials. It was recognized that the use of fly ash in concrete would have the following benefits:

(*a*) improved workability,
(*b*) lower heat of hydration,
(*c*) lower cost concrete,
(*d*) improved resistance to sulfate attack,
(*e*) improved resistance to alkali-silica reactions (ASR),
(*f*) higher long-term strength,
(*g*) opportunity for higher strength concrete,
(*h*) equal freeze-thaw durability,
(*i*) lower shrinkage characteristics, and
(*j*) lower porosity and decreased permeability.

Good results were being obtained with fly ash [10–13,30,31] and natural pozzolans [20,32], and therefore there was a need for an ASTM specification for their use. A very simplistic timeline of events pertinent to the development of the various specifications is given in Fig. 3. For completeness, the figure also includes events related to the development of the specification for silica fume.

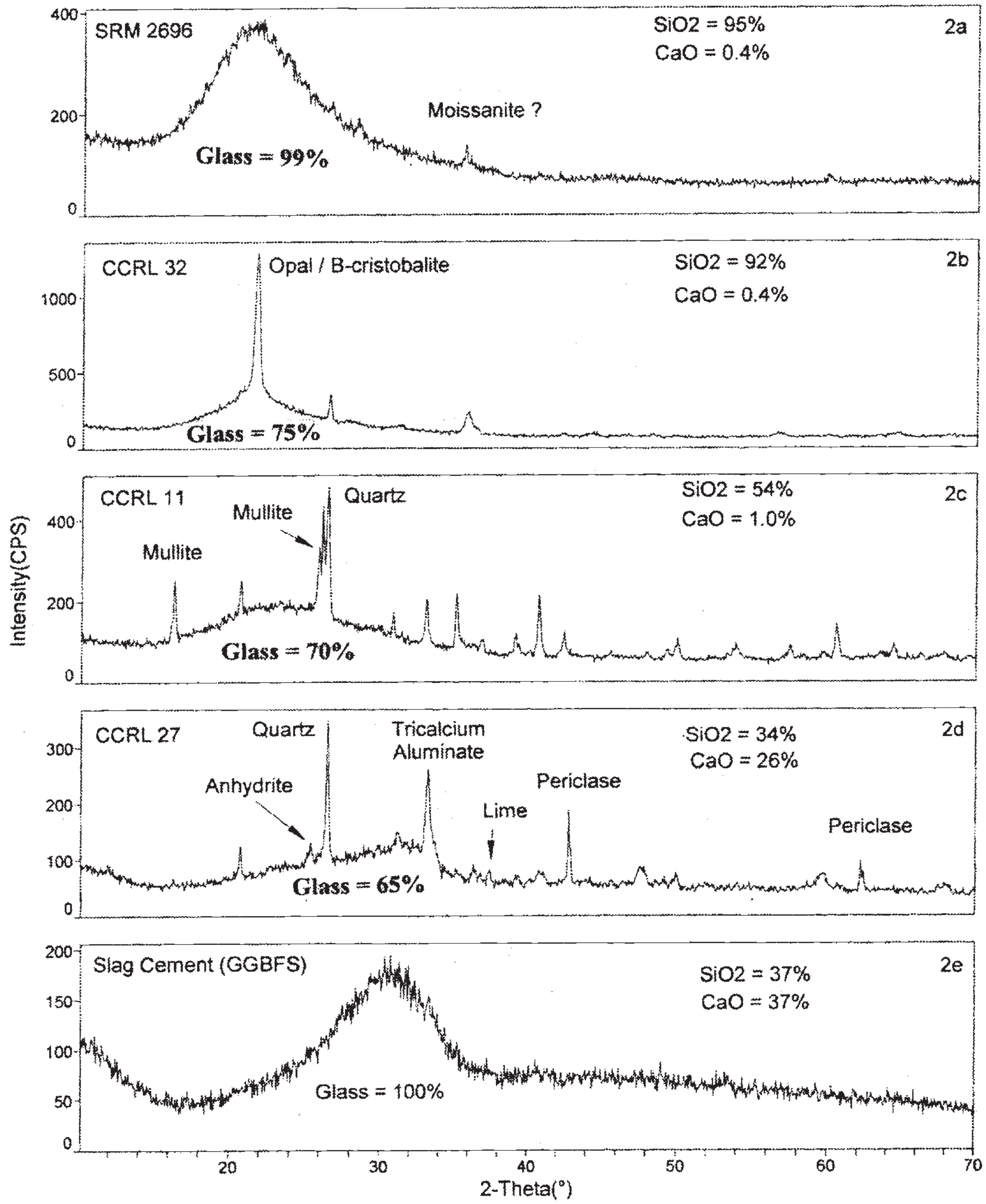

Fig. 2—X-ray diffractograms for common supplementary cementitious materials: (2a) Silica fume, SRM 2696; (2b) Class N pozzolan, CCRL 32; (2c) Class F fly ash, CCRL 11; (2d) Class C fly ash, CCRL 27; (2e) GGBFS (slag cement).

The preparation of a specification for fly ash for use in portland-cement concrete was begun in 1948 by ASTM Committee C-9 on Concrete and Concrete Aggregates. This work was done by Subcommittee III-h dealing with all admixtures, chemical and mineral, later designated as Subcommittee C09.03.08. In early 1953, the Subcommittee proposed two standards on fly ash. One of these dealt with the methods of testing and was published as ASTM Tentative methods of Test for Fly Ash as an Admixture for Portland Cement Concrete (C 311-53T). The other proposed standard dealt with specifications for fly ash with various chemical and physical limits. This latter proposal met with serious opposition from some members of the cement industry and was not approved by Committee C-9 until several changes were made. In point of fact, these changes lumped all fly ash into one class where a class distinction would have allowed the better fly ashes better recognition. It was published as ASTM Tentative Specifications for Fly Ash for use as an Admixture in Portland Cement Concrete (C 350-54T) and covered only the use of what is now designated as Class F fly ash in concrete "where the use of increased quantities of fine material may be indicated to promote workability and plasticity." The fly ash was to be treated only as a portion of the fine aggregate and not as a partial replacement or substitute for portland cement. Notes of caution on the use of fly ash in concrete were inserted in the early versions of the specification and dealt with the amount of sulfur, magnesium oxide, and effect on autoclave expansion. Experience has shown them to be of less concern than expected, but many of the reflected limits are still in the specification.

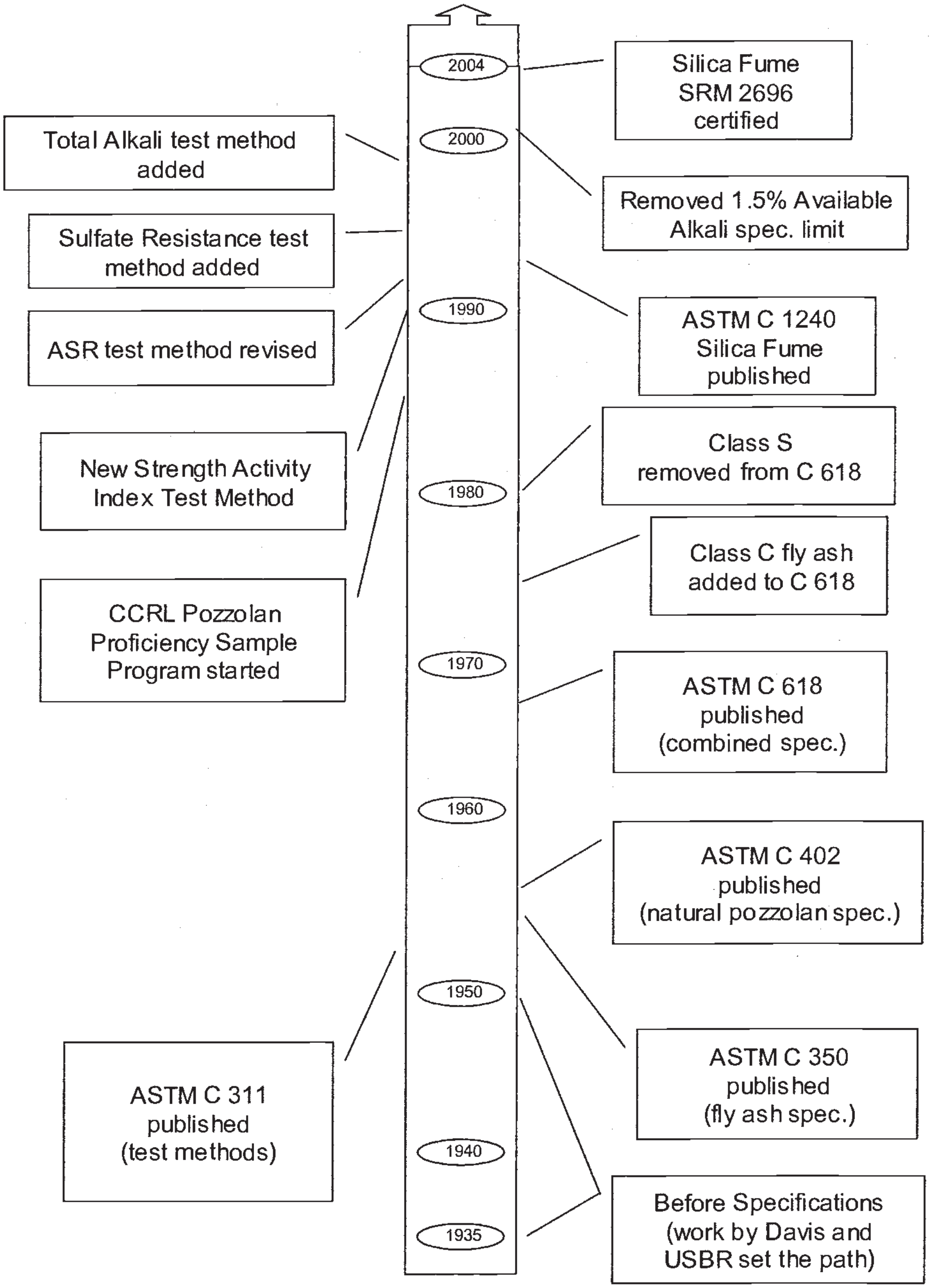

Fig. 3—Time line of important events for natural pozzolans, fly ash, and silica fume.

ASTM C 350 was revised repeatedly based on new experience and research. In 1960, the scope of ASTM C 350 was extended to cover fly ash as a pozzolan in concrete acknowledging the prospect that the use of fly ash in the mixture might result in a reduced amount of portland cement.

In 1957, ASTM Tentative Specifications for Raw or Calcined Natural Pozzolans for Use as Admixtures in Portland-Cement Concrete (C 402-57T) was published. At that time differences between natural pozzolans and fly ash were recognized by different requirements on drying shrinkage, loss on ignition, fineness, and reactivity with cement alkalies. Then in 1968, ASTM C 350 was combined with ASTM C 402 resulting in ASTM Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use in Portland Cement Concrete (C 618-68T). Three classes of material were dealt with in three columns. Class F was the fly ash from ASTM C 350. Class N was the natural pozzolan from ASTM C 402. A new Class S having the same requirements as Class F allowed for other materials that could not meet the requirements for natural pozzolans, but could contribute desirable qualities to the concrete mixture.

In 1977, Class C fly ash was added to ASTM C 618 to recognize it as a material with cementitious characteristics compared to those of the original Class F. There was no apparent use of Class S pozzolan in the construction industry, so the designation was dropped from ASTM C 618 in 1980.

In 1980, ASTM Committee C-9 formed a new subcommittee, ASTM C09.03.10 on Fly Ash, Slag, Mineral Admixtures, and Supplementary Cementitious Materials, with Robert E. Philleo as chairman. The number designation of the subcommittee was changed to C09.24 in 1992. The scope of ASTM C09.24 was "to develop and maintain test methods and specifications for finely divided mineral materials other than cement and pigments, used in substantial proportions in concrete." This new subcommittee now had no concern for chemical admixtures and instead had new interest in slag and eventually silica fume.

In 2001, the name of the subcommittee was changed to Supplementary Cementitious Materials. The scope of the subcommittee was changed to reflect the formation of a new subcommittee (C09.27) for slag cement. The new scope for C09.24 is "to develop and maintain standards for supplementary cementitious materials other than ground granulated blast furnace slag." Hence, the main materials currently under the jurisdiction of C09.24 are fly ash (Classes F and C), natural pozzolans, and silica fume.

Classification

There have been many attempts to classify natural pozzolans and fly ash into subgroups that reflect how a specific source of material will perform when mixed with hydrated lime or cement. Performance in most instances is normally interpreted as the ability of the material to enhance the ultimate strength of the mixture. These classification schemes have been based on bulk chemistry, mineralogy, and geological origin. None of the classification schemes has been adopted for practical use.

Since 1968, when ASTM Specification for Natural Pozzolans in Concrete (C 402) was combined with the fly ash specification, ASTM C 350, to form the current ASTM C 618, there have been no changes to the requirements for natural pozzolans that were not brought on because of experiences with fly ash. However, in 2000, there was enough interest in the use of natural pozzolans that users requested the formation of a new task group in C09.24 to review the status of test methods and specification limits for natural pozzolans. The task group was disbanded in 2002 after four consecutive meetings failed to produce any items to be balloted. Hence, much of this section will deal primarily with fly ash. Comments about natural pozzolans will be interjected when the specification limits differ from those for fly ash.

In 1977, when Class C fly ash was added to ASTM C 618, there was recognition of the fact that this was a new material made available because utilities had begun to burn larger quantities of low-sulfur coal from the western United States. This new fly ash exhibited both chemical and mineralogical differences from the Class F fly ash around which the earlier version (C 350) of the specification had been written [33,34]. Class C fly ash often exhibits self-cementitious characteristics when mixed with water. It contains higher amounts of elemental calcium (commonly expressed as calcium oxide, CaO) and is less of a pozzolan [30,35,36]. Mielenz [37] was among the first to note that "Class C fly ashes that display cementitious properties when gauged with water do not conform to the definition of pozzolans." This created a dilemma that we are still dealing with today—fly ash is generically (classically) attributed with enhancing the properties of concrete summarized in the History and Use section of this chapter. How do we deal with the divergence of properties manifested by test specimens containing these different classes of materials? After all, Class C fly ash generally exhibits greater hydraulicity and makes a greater contribution to early strength than that obtained from the Class F fly ash. Also, as noted by several authors [38–44], there is considerable evidence that Class F fly ashes perform better than Class C ashes for controlling ASR or enhancing sulfate resistance. Currently the only differentiation made between the two classes of fly ash in ASTM C 618 is coal source and bulk chemistry. ASTM C 618 states that Class F fly ash is "normally produced from burning anthracite or bituminous coal" and Class C fly ash is "normally produced from lignite or subbituminous coal." This distinction has been controversial. The subcommittee has not been able to make a better one. Recently, the Canadian Standards Association (CSA) has split fly ash into three groups based on analytical calcium content [45]. Attempts have been made to ballot similar fly ash classification schemes in ASTM C 618; however, to date all attempts have failed.

Chemical Requirements

Table 1 of ASTM C 618 gives the chemical requirements for fly ash and natural pozzolans. There is a limit on the sum of three elements, expressed as oxides, namely, silicon dioxide (SiO_2) plus aluminum oxide (Al_2O_3) plus iron oxide (Fe_2O_3). There are also limits on the sulfur trioxide (SO_3), moisture content, and loss on ignition. ASTM C 311, specifying the methods to be used in the determinations, refers to ASTM Test Methods for Chemical Analysis of Hydraulic Cement (C 114) with modifications for a material with a high insoluble residue. Rapid and instrumental methods may be employed similar to those in C 114 or in ASTM Test Method for Major and Minor Elements in Coal and Coke Ash By X-ray Fluorescence (D 4326).

The earliest versions of ASTM C 618 assumed that the pozzolanic contribution had to come from its makeup of SiO_2, Al_2O_3, and Fe_2O_3. The minimum requirement placed on these three constituents is based on that assumption. However, variations in the total of those three constituents for any material have not been observed to correlate directly with results in concrete. At one point in the development of the specification, there was a suggestion to remove the requirement with the rationale that it served only to define the material as a pozzolan [46]. At that time, the expense and delay in running these tests was significant and not altogether worthwhile. Now, with quicker instrumental chemical testing procedures, the determination of the total SiO_2, Al_2O_3, and Fe_2O_3 (plus many additional elements of interest) is not a concern.

The only specified difference between fly ash Classes F and C is the total of SiO_2 plus Al_2O_3 plus Fe_2O_3. Class F has always required a minimum of 70 % and this limit served well for many years. Class C requires a minimum of 50 %. Actually, this is the subcommittee's way of recognizing that Class C may have 20 % more analytical calcium (expressed as an oxide, CaO) content than Class F with the corollary result of lowering the sum of the other three oxides. This is the only chemical difference recognized between Classes F and C in the specification. Any material passing the Class F requirement will also pass as Class C. Therefore, this is not a helpful distinction.

Sulfur in fly ash and natural pozzolans is reported as SO_3. The first editions of this specification limited the SO_3 to 3 %. Subsequent research showed that a fly ash with up to 17 % SO_3 did not give significant delayed volume changes or setting problems. Hence, the SO_3 limits for fly ash were raised to 5 % (maximum). The SO_3 content for natural pozzolans is currently listed at 4.0 % (maximum). Higher amounts of sulfur trioxide, especially when associated with soluble alkali, can contribute to efflorescence, so when they are present, testing for discoloration can be considered.

The moisture content is limited to 3 % maximum because the material would become sticky and hard to handle if too much moisture were encountered. The test is easy and inexpensive to run and the values determined are needed in other parts of the test methods.

The limit on loss on ignition (LOI), now set at a maximum of 6.0 %, was originally set at 12.0 %. At that time, many of the fly ashes available in the eastern United States could not pass limits

TABLE 1—CCRL Test Results for the Available Alkali Test

Sample	Year	Class	Number of Labs	Available Alkali (as %Na_2O_e)	Standard Deviation	Coefficient of Variation, %
CCRL 1	1987	C	12	0.97	0.28	29.0
CCRL 2	1987	C	12	1.52	0.60	39.5
CCRL 3	1988	F	20	0.56	0.21	37.3
CCRL 4	1988	F	20	0.74	0.35	47.1
CCRL 5	1989	F	27	2.21	0.57	25.8
CCRL 6	1989	F	27	2.69	0.50	18.6
CCRL 7	1990	F	29	0.47	0.14	30.0
CCRL 8	1990	F	29	0.40	0.12	29.8
CCRL 9	1991	C	28	3.77	1.80	47.7
CCRL 10	1991	C	28	6.18	2.70	43.7
CCRL 11	1992	F	27	0.54	0.18	33.6
CCRL 12	1992	F	27	0.48	0.19	39.6
CCRL 13	1993	C	23	1.33	0.25	18.8
CCRL 14	1993	C	23	1.40	0.59	42.3
CCRL 15	1994	F	27	0.50	0.10	19.8
CCRL 16	1994	F	27	0.79	0.19	24.1
CCRL 17	1995	C	29	1.23	0.40	32.4
CCRL 18	1995	C	29	1.11	0.38	34.1
CCRL 19	1996	F	28	1.24	0.32	25.8
CCRL 20	1996	F	28	1.14	0.44	38.8
CCRL 21	1997	C	23	2.43	0.65	26.7
CCRL 22	1997	C	23	2.21	0.81	36.7
CCRL 23	1998	C	25	1.33	0.24	18.1
CCRL 24	1998	C	25	1.24	0.25	20.1
CCRL 25	1999	F	20	0.45	0.10	23.0
CCRL 26	1999	F	20	0.38	0.10	25.4
CCRL 27	2000	C	25	1.37	0.15	10.9
CCRL 28	2000	C	25	1.46	0.15	10.3
CCRL 29	2001	F	20	0.70	0.15	21.4
CCRL 30	2001	F	20	0.52	0.13	24.9
CCRL 31	2002	N	23	0.36	0.13	36.1
CCRL 32	2002	N	23	0.30	0.13	43.3
CCRL 33	2003	C	20	1.15	0.19	16.5
CCRL 34	2003	C	20	1.43	0.22	15.4

much lower, and they were giving satisfactory results in concrete. With the advent of better (more complete) combustion in electric power plants and with the coming of Class C fly ash to the market, it was found that many agencies were dropping the limit to 6.0 %. Subcommittee C09.03.08 was divided in the action to lower the limit. The maximum LOI was already at 6.0 % for Class C fly ash. The note under ASTM C 618, Table 1, was added providing for a 12.0 % maximum LOI if acceptable performance records were provided. Everyone concedes, however, that the lower the loss on ignition, the easier will be the control of air entrainment content in the concrete. Class C fly ash is usually below 1 % LOI. Agencies frequently lower this maximum when they are sure there are competitive materials available in their marketplace. LOI for natural pozzolans is set at a maximum of 10.0 %. This is the same value given in the initial version of C 402, issued in 1957. It is important to note that the LOI of natural pozzolans is not normally associated with the presence of free carbon. Rather it is more commonly attributed to the presence of minerals that decompose during heating at 750°C (e.g., clays or carbonates).

Historically, loss on ignition in fly ash is directly related to carbon content (see Fig. 4). When the specification was first written, the determination of LOI was chosen because it was an available test method from ASTM C 114 and was preferable to the determination of free carbon. LOI is run at 750°C rather than 950°C, the temperature given in ASTM C 114. Currently, instrumental tests for carbon are frequently run instead of LOI and are quicker and more desirable [47]. This is especially true when additional fuels such as petroleum coke are burned along with coal. In that instance, the LOI test method given in C 311 typically fails to burn off all of the carbon present in the fly ash.

Supplementary Optional Chemical Requirements

Currently there are no supplementary optional chemical requirements for fly ash or natural pozzolans. The supplementary optional chemical requirements were removed in 1983 (bulk magnesium oxide) and 2001 (available alkali). The removal of these optional requirements was associated with the replacement of prescriptive chemical requirements with performance tests.

The bulk magnesium oxide content (analytical Mg, expressed as an oxide) of fly ash and natural pozzolan has historically been limited to 5 % (maximum). This was initially a mandatory requirement (1968 through 1976) and then an optional chemical requirement (1977 through 1983). This meant that specifiers could limit usage based on an optional chemical requirement even if the SCM met the mandatory physical requirement by passing the autoclave test. Originally, this limit

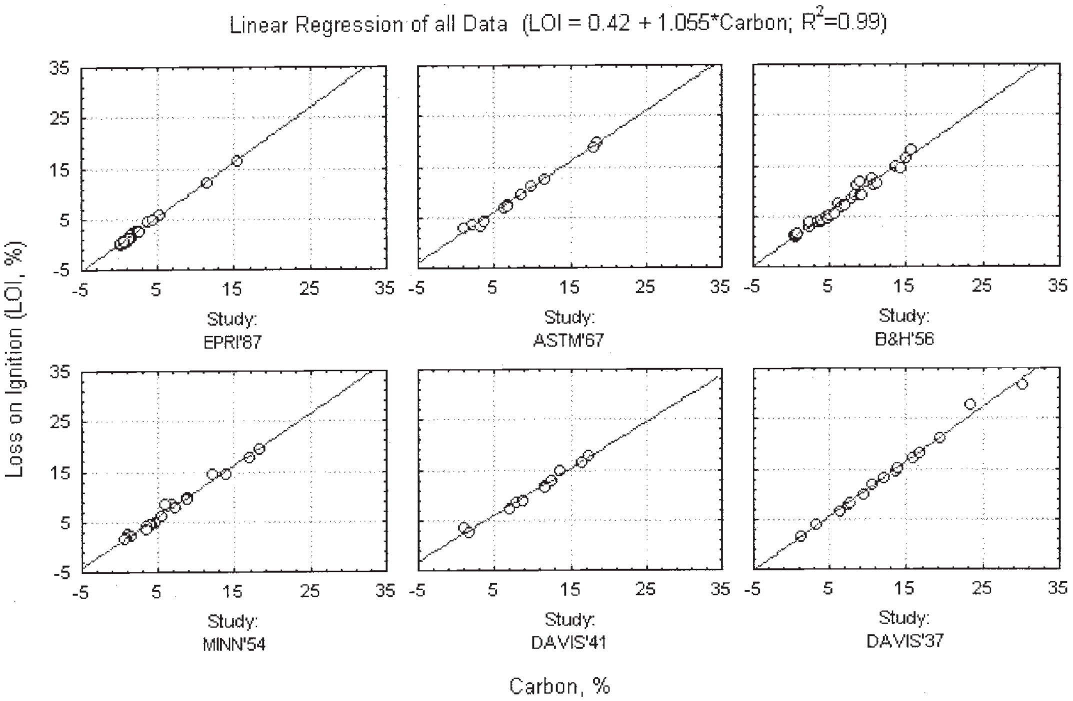

Fig. 4—Relationship between carbon content and loss on ignition for fly ash. Historical data extracted from Davis [10,11], Minnick [12], Brink and Halstead [13], ASTM [14], and EPRI [15].

was based on the concern that the presence of magnesium would indicate the presence of periclase (MgO) in the SCM. Periclase, while only sporadically identified in Class F fly ash and natural pozzolans, is very common in Class C fly ash (compare Fig. 2b, 2c and 2d). Hence, after the subcommittee became more comfortable with the conservative results produced by the autoclave test, the redundant specification limit was removed. Later work indicated that the relationship between bulk magnesium oxide content and the presence of periclase in fly ash was considerably more complex than had been anticipated [16].

Up until 2001, the available alkali (or available "alkalies" in older versions) content was limited to 1.5 % (maximum) in the chemical or optional chemical requirements. This initially only applied to fly ash but later the limit was also applied to natural pozzolans. A note to this supplemental chemical requirement stated that the limit was "Applicable only when specifically required by the purchaser for mineral admixtures to be used in concrete containing reactive aggregate and cement to meet a limitation on content of alkalies." Since about 1989 there had been much discussion in the subcommittee about the available alkali test and its relevance to the ongoing battle against alkali-silica reaction (ASR). Concerns were voiced about both the reliability of the test method and the significance of the 1.5 % limit. Since no member of the subcommittee could remember why the limit was originally set at 1.5 % the historical significance and rationale behind the limit remains a mystery.

The available alkali test method dates back to about 1950 [48]. At that period in time the test method was most probably considered highly innovative because it employed new technology (flame photometry) that was greatly enhancing the ability of analysts to determine the alkali content of a wide variety of materials. Prior to the use of the flame photometer the determination of sodium and potassium was not trivial–it was laborious and required highly trained technical staff. Hence, when the method proved fruitful in the determination of water soluble and total alkali in portland cements [49], it rapidly became the method of choice for measuring alkali content. However, fly ash and pozzolans are only partially soluble in acid so another method had to be devised to put the alkali into solution. That method became the "available alkalies" test. The method summarized in ASTM C 311-53T is very similar (but not identical) to one described by Moran and Gilliland [50] (see Appendix B, page 126), which was used by the U.S. Bureau of Reclamation during the 1940s.

The purpose of the available alkali test is to estimate the amount of alkali (sodium and potassium, normally expressed as equivalent sodium oxide) that a fly ash or pozzolan can release when it is reacted with calcium hydroxide. The test is long and tedious. The specimens are cured for 28 days at a temperature of 38°C and then the alkalis are washed from the pulverized specimens. In concept, this was assumed to give an estimate of how much alkali the fly ash could contribute to alkali-silica reaction. However, the test method typically exhibits poor interlaboratory precision (see Table 1) and this limits the applicability of the test results. This is especially true when the test results approach the 1.5 % specification limit (note, if the precision values for CCRL samples 9 and 10 are ignored, that the D2S limit is about 0.9 %, absolute, for this test method). The poor precision has been linked to a variety of different experimental variables, such as: (a) calibration standards do not adequately represent the unknown samples; (b) specimen grinding and washing during the alkali extraction phase are poorly defined; and (c) carbonation of the calcium hydroxide has a major impact on the test results. Also, alkali solubility data for a small number of natural pozzolans and Class F fly ashes indicated that the release of alkali could continue out to 90 days of curing [50]. Using a larger suite of fly ash samples, Brink and Halstead [13] showed that the release of alkali from Class F fly ash often continued past the 28-day curing period specified in the available alkali test. Similar observations were reported for Class C fly ash

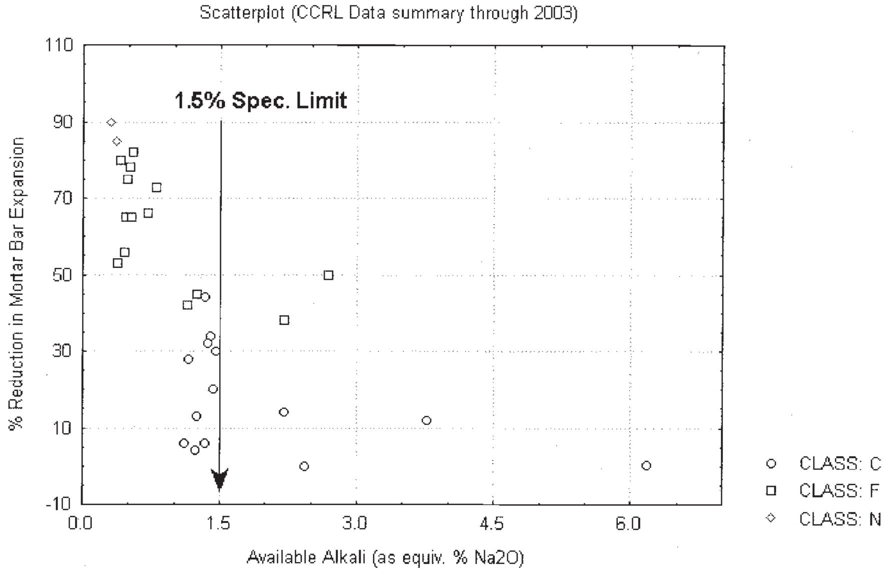

Fig. 5—Relationship between available alkali content and the reduction of mortar bar expansion for data from the Cement and Concrete Reference Laboratory (CCRL) Pozzolan Proficiency sample program.

in 1986 [51]. Work by Buttler, Morgan, and Walker [52] indicated that the selection of the fly ash to calcium hydroxide ratio used in the test method also plays a critical role in the amount of alkali that was liberated during the 28-day curing period. Their test results indicated that the amount of alkali extracted increased when the fly ash-calcium hydroxide ratio was decreased. In addition, test results from the CCRL Pozzolan Proficiency testing program show little indication of a strong relationship between available alkali content and the reduction in mortar bar expansion (see Fig. 5). There is a considerably better relationship between the sum of the oxides ($SiO_2+Al_2O_3+Fe_2O_3$) and the reduction in mortar bar expansion (see Fig. 6). These facts made the 1.5 % limit very difficult to support. Since the subcommittee could not remember the rationale for setting the limit at 1.5 % in 1957, they reached a consensus to remove the limit.

The strategy for removal of the specification limit was composed of three steps. First, a performance test was adopted that would differentiate between SCMs based on their ability to reduce the expansion caused by ASR. The test method that was adopted will be described later in this chapter. Secondly, a test method for measuring the total sodium and potassium content was provided so that the alkali content of the SCM could be measured and the total alkali burden of the cementitious material could be calculated. The determination of total alkali content is fast and allows one to perform testing on a routine basis with a much better turnaround time than was possible with the available alkali test. Finally, the 1.5 % specification limit for

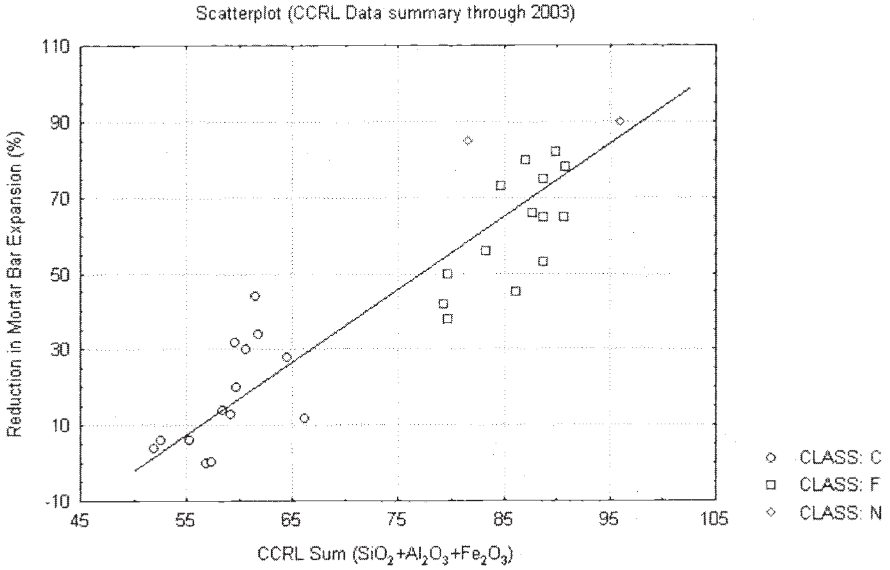

Fig. 6—Relationship between the oxide sum (Si+Al+Fe) and the reduction of mortar bar expansion for data from the Cement and Concrete Reference Laboratory (CCRL) Pozzolan Proficiency sample program.

available alkali was removed. No maximum limit was placed on the total alkali content of the SCM because the subcommittee could not reach a consensus on an appropriate value for the limit. Instead, they indicated that the performance test results should guide the selection and replacement level appropriate for any given mixture of SCM and cement.

Physical Requirements

The mandatory physical requirements, set forth in Table 2 of ASTM C 618, consist of fineness, strength activity index, water requirement, soundness, and uniformity of fineness and density. This section will deal with them in that order.

Fineness of a SCM is important in its contribution to a concrete mixture. Although there is not a direct correlation, the amount of water needed to produce a concrete mixture of adequate workability and the apparent reactivity of the SCM are both dependent on fineness. The first versions of this specification used specific surface as a measure of fineness. Specific surface was measured by ASTM Test Method for Fineness of Hydraulic Cement by Air-Permeability Apparatus (C 206) (this is the Blaine test adjusted to compensate for the density of the material being analyzed). Then, mean particle diameter was substituted in 1965. The consensus went back to specific surface in 1968. At that time, a new fineness requirement was added for Class N pozzolans that consisted of fineness as determined by wet washing through a 45 μm (No. 325) sieve. A maximum of 12 % retained was the original limit. Fly ash added the new fineness test and a new specification limit in 1971. The limit was initially set at 20 % (maximum, % retained) but was later raised to 34 % (maximum) in 1973, when the procedure in ASTM Test Method for Fineness of Hydraulic Cement by the 45-μm (No. 325) Sieve (C 430) began to call for the use of the new electroformed sieves [4,53,54]. These changes are mentioned to indicate the difficulty experienced measuring fineness in a way that correlates with results in concrete. In the C 206 test, higher carbon contents can give readings indicating higher surface area, which would assume a finer product when actually the material is coarser or less fine [55]. The percent-retained test does not measure the fine material that is actually contributing to the concrete reaction, but it generally acts as an accurate indicator of the amount of fine material present. The test is simple, quick, and inexpensive and can be run at a high frequency for quality control purposes. The sieve calibration procedure was changed in 1994 to utilize an additive correction factor rather than the multiplicative correction factor given in C 430. This was done in response to concerns voiced about both the calibration procedure and the calibration standards used in the test method [56]. The new calibration procedure appeared to effectively remove the bias in the test results reported for CCRL Pozzolan Proficiency samples 7 and 8 [57]. This is in good agreement with the work by Butler and Kanare [56], which indicated that the selection of a calibration standard was less critical when the additive correction factor was used. Their test results indicated that the cement calibration standard (SRM 114n) "gave results comparable to the best SRM fly ash results." Hence, this appeared to fix the fineness calibration problem and still allowed laboratories to use only a single SRM (cement standard 114) for both cement and fly ash fineness determinations.

Strength tests have always played a fundamental role in the acceptance of fly ash and natural pozzolans for use in concrete. Strength testing is normally conducted on mortar specimens containing specific proportions of lime and SCM (pozzolanic activity index with lime) or on mortar cubes containing specific amounts of portland cement and SCM (compressive strength of mortar cubes, pozzolanic activity index with portland cement, strength activity index with portland cement). The history of early changes in the test methods and associated specification limits has been discussed by Mielenz [37,48,53]. The mortar tests and associated specification limits have always been subject to criticism because they do not directly correlate with the performance of the SCM in concrete. However, the reader must bear in mind that the purpose of standardized testing is to ensure that the product (fly ash or natural pozzolan) meets minimum requirements listed in the standards. The tests are meant to be used as product acceptance tests on a nationwide basis and not as a guide to mixture designs for specific projects. Hence, a note stating this disclaimer has been used since the early versions of the specifications.

The strength activity index test with lime was controversial for some time prior to being removed from the specification. Part of the reason for the controversy was probably related to the very poor interlaboratory precision of the test method (see Table 2). The exact reason for the poor precision is difficult to pinpoint; however, the test was felt to be as much a test of the lime (fineness, purity, etc.) as it was of the fly ash or natural pozzolan. The requirement was dropped for Class C fly ash in 1985. Class C fly ash makes its own contribution of lime to the mixture, so this test was irrelevant. The requirement was dropped for Class F and Class N pozzolans in 1992. Part of the reason the method had stayed around so long was because it was quick; it

TABLE 2—CCRL Summary of Test Results from the Lime Pozzolanic Strength Index

Sample	Year	Class	Number of Labs	Mean (psi)	Standard deviation (psi)	COV (%)
CCRL 5	1989	F	21	1042.3	267.5	25.7
CCRL 6	1989	F	21	960.3	222.5	23.2
CCRL 7	1990	F	25	782.4	188.0	24.0
CCRL 8	1990	F	25	595.4	211.0	35.4
CCRL 9	1991	C	23	2144.9	405.5	18.9
CCRL 10	1991	C	23	2851.7	937.0	32.9
CCRL 11	1992	F	26	1068.7	193.5	18.1
CCRL 12	1992	F	26	947.7	200.1	21.1

was the only strength test that produced a result in seven days. The other tests all required 28 days of curing prior to producing a test result. Hence, it was retained for that reason alone.

During the mid to late 1980s, the pozzolanic activity index test with portland cement was subjected to considerable scrutiny. The method was subjected to several round robin studies and extensive changes in procedure and evaluation resulted from the studies (ASTM report RR: C09-1001). The changes were substantial so the revised test method was given a new name to avoid confusion with the old method. Also, the name was changed to reflect the subcommittee's opinion that any test conducted at room temperature, with a maximum duration of only 28 days, was too short to ascertain the "pozzolanic" nature of the SCM. Rather, the method would simply indicate how a SCM behaved with any given cement. The new test method was named the strength activity index with portland cement and it was published in 1990. The changes in the method pertained to SCM replacement level, curing temperature, flow properties, and the alkali content of the lab cement that would be used in the testing. The changes to the flow properties of the mortar will be described later with the discussion on the water requirement limit. In the new method, the replacement of portland cement is done by mass (20 %) rather than volume (35 % in the old method) to reflect field usage. This also helped to eliminate the need to conduct density tests prior to proportioning the mortar. The curing temperature was reduced from 38°C (sealed containers) to standard laboratory conditions (23°C, submerged in lime water). This greatly simplified the curing requirements of the test method. One side effect of the reduction in curing temperature was that the alkali content of the portland cement needed to be constrained within a specific range (0.50 % to 0.80 %, as equivalent Na_2O). High alkali cements tended to increase the early strength gain of the test specimens. In addition, low alkali cements tended to decrease the early strength gain of the test specimens. Hence, for standardization purposes the alkali content of the lab cement needed to be constrained so that test results could be compared across the country. This did not mean that job cement of any arbitrary composition could not be used in the evaluations. Rather it simply meant that the subcommittee was striving to reduce the interlaboratory variation in the test method. The most important addition to the new test method was a 7-day alternate with the previous 28-day requirement. The availability of a 7-day result from the strength activity index test allowed the subcommittee to delete the 7-day lime pozzolanic activity test.

One complaint about the strength activity index with portland cement test has been that it fails to consistently reject finely ground materials that exhibit marginal to negligible pozzolanic properties (for example, pulverized quartz). This can be partly attributed to the low dosage of SCM (20 % replacement) that is used in the mixture. During the development of the test method a coarse Class F fly ash was used to simulate a material that would consistently fail to meet the specification limit; however, this approximation apparently failed to cover an adequate range of behavior. The subcommittee has discussed this issue several times and is currently waiting to see how frequently testing labs report this anomalous behavior. To date, after using the new test method for a little over a decade, two labs have reported instances where materials have passed the specification limit but exhibited little potential as a pozzolanic material. Hence, this is still an active issue in the subcommittee.

The water requirement limit gives some measure of the contribution to workability that the SCM will impart to a concrete mixture. The test is performed using the mortar that is prepared for the strength activity index with portland cement test. The control mortar is proportioned at a fixed water-cement ratio of 0.485 and the flow for the mixture is determined. The flow of the test mortar (containing 20 % SCM) is constrained to be ± 5 % of the flow value that was obtained for the control mortar. Ultimately, by trial and error, this dictates the amount of water that will be used for the test mortar. For fly ash, the limit is set at a maximum water demand of 105 %, relative to the control cement. For fly ash, this limit will depend on fineness, carbon (or LOI), and, to a lesser extent, on the glassy spherical particles in the fly ash. For natural pozzolans this limit is set at a maximum of 115 %, and depends primarily on the fineness and surface characteristics of the material. When the pozzolanic activity index test was replaced with the strength activity index test in the late 1980s, the water requirement limits were held constant. This was done even though the amount of SCM used in the test mixture was significantly reduced. Hence, this has impacted the usefulness of the water requirement limits because it has reduced the range of test results that are obtained. Good fly ashes, which often reduced the water demand about 8 % to 10 % in the pozzolanic activity index test, now only reduce the water demand about 5 % for the current test method. Of more concern are tests conducted on fly ash or pozzolans that have high water demands. Reducing the quantity of SCM that is added to the test mortar makes the method less sensitive to problematic behavior [58]. However, such behavior may no longer be relevant to field applications because chemical admixtures (water reducers) have become so commonly used in the construction industry.

Soundness, as measured by the autoclave expansion test, will indicate any possible delayed expansion problems related to periclase and free lime, if those materials are present in deleterious quantities. Extensive testing has indicated that the autoclave test provides a conservative approach to identifying potential soundness problems related to periclase and/or free lime in fly ash [16,59,60]. Helmuth and West [61] have reported concerns about the validity of the autoclave test when evaluating blended cements; however, these concerns were attributed to the influence of unsound cements containing pozzolans, not unsound pozzolans. The drier consistency of the paste used in the autoclave test constrains the hydration of periclase and lime when they are present in a "hard burned" state. Some Class F fly ash has failed this test due to the lime, even though the lime would have been adequately hydrated when used in concrete at normal water-to-cementitious-materials ratios. Users still need to pass the autoclave test when using higher SCM replacements. Note *D* of Table 2 states that "If the fly ash or natural pozzolan will constitute more then 20 % by weight of the cementitious material in the project mix design, the test specimens for autoclave expansion shall contain that anticipated percentage. Excessive autoclave expansion is highly significant in cases where the water to fly ash or natural pozzolan and cement ratios are low, for example, in block or shotcrete mixes."

The mandatory physical requirements for uniformity include limits for fineness (± 5 percentage points) and density (± 5 %). Both requirements are compared to the average of the ten prior test results. The uniformity of the fineness is specified since it is an easily determined indicator of a change in the product. Such a change is important because it might affect water demand in concrete. The other uniformity requirement in Table 2 of ASTM C 618 is on density. However, a change of 5 % in the uniformity of density is not thought to be significant either as an indicator of product change or in effect on concrete mixture design. This is probably because most agencies currently limit SCM replacements to about 15 % to 25 % of the cement in the mixture. Hence, the 5 % uniformity limit on the SCM tends to be obscured by the variability present in the other raw materials [62].

Supplementary Optional Physical Requirements

ASTM C 618, Table 3, lists the supplementary optional physical requirements as multiple factor, drying shrinkage, uniformity requirement for air-entraining solution demand, effectiveness in controlling alkali-silica reactivity, and effectiveness in contributing to sulfate resistance.

The multiple factor is defined as the sieve fineness multiplied by the LOI (or free carbon content). For Class F fly ash the multiple factor has exhibited good correlation with the relative water requirement; and hence, has been linked to strength parameters in mortar and concrete [58]. The multiple factor helps to avoid the situation where increasing fineness values and carbon content (LOI) lead to an increase in water demand. This relationship does not apply to Class C fly ash because the LOI is generally below 1 % and is, therefore, too low to act as a multiplier. In addition, the relationship does not apply to natural pozzolans because the LOI is not related to carbon content and the fineness can be adjusted by manufacturing processes.

Increase of drying shrinkage is a requirement because some natural pozzolans will cause an unacceptable increase in drying shrinkage. This is typically related to the increased water demand of the mixture. Fly ash will generally reduce drying shrinkage [63,64]. The test method, which dates back to the 1950s, originated from a time when the SCM was commonly used to replace a portion of the aggregate. This is in contrast to modern practice in which the SCM is treated as part of the cementitious material. Hence, the paste content of the mortar containing the SCM is greater than in the control mortar. Since total cementitious material content and water content both have a significant influence on shrinkage, this tends to put the test specimen containing SCM at a disadvantage. Hence, the test method is currently being re-evaluated and may be revised in the future.

The test procedure for the uniformity of air-entraining agent demand has recently been challenged as not being sensitive enough [65]. The problem with the test method is related to the high air content (18 ± 3 %) used in the evaluation. The subcommittee will consider a lower air content that would make the test more sensitive. In the meantime, this optional test will deal with the concern that some fly ash causes problems with control of entrained air in the concrete mixture.

Class F fly ash and natural pozzolans have been used to reduce the expansion caused by alkali-silica reaction for over 50 years. Early work by Hanna [32], Davis [20], Lerch [66], Stanton [67], and Pepper and Mather [68] set a firm foundation upon which nearly countless other authors have built. Mielenz et al. [69] have indicated that natural pozzolans typically perform better than fly ash in controlling expansion related to ASR (also, refer to Fig. 5). However, the reactivity with cement alkalies test has not been a satisfactory method for selecting fly ash that can help to reduce expansion caused by alkali-silica reaction. The required "reduction of mortar expansion" does not apply to fly ash and the required mortar bar expansion at 14 days (0.020 %, maximum) is too low for many otherwise acceptable fly ashes (especially when the replacement is limited to 25 %). Therefore, this has served to cause the rejection of materials that might have been helpful in correcting alkali-silica reaction in concrete. The 0.020 % limit was set assuming that low-alkali portland cements (less than 0.60 %, expressed as equivalent Na_2O) could meet that limit [69]. However, recent changes in the storage containers for the test procedure have increased the sensitivity of the test and this limit can now only be reached by cements with very low alkali contents [70,71]. Extensive work by many researchers has clearly indicated that fly ash replacement level is a major factor in controlling expansion caused by ASR [32,42,44,67,68]. This is especially true when the replacement level is near the pessimum level for the cementitious system. Also, Dunstan [41] has hypothesized that fly ash class designation should impact the ability of a fly ash to reduce the expansion caused by ASR (see Fig. 7; note that the trend is also evident in CCRL test results illustrated in Fig. 6). These inconsistencies forced the subcommittee to improve the test method and specification limit for the test.

The new test procedure is called the Effectiveness of Fly Ash or Natural Pozzolan in Controlling Alkali Silica Reactions and it was adopted in 1993. The new test procedure is accompanied by a commentary describing how the procedure should be used and interpreted (see Appendix X1 of C 311). Briefly, the test procedure is based on the mortar bar expansion test described in the Standard Test Method for the Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction, ASTM C 441. This method was modified to substitute SCM for cement on a mass equivalent basis and a control cement is used to set the level of expansion (performance) that the test specimens will be compared against. This concept is illustrated in Fig. 8.

The control cement for the test is typically chosen with an alkali content between 0.50 % and 0.60 % equivalent Na_2O (Control 1 in Fig. 8, alkali content = 0.52 % as Na_2O). However,

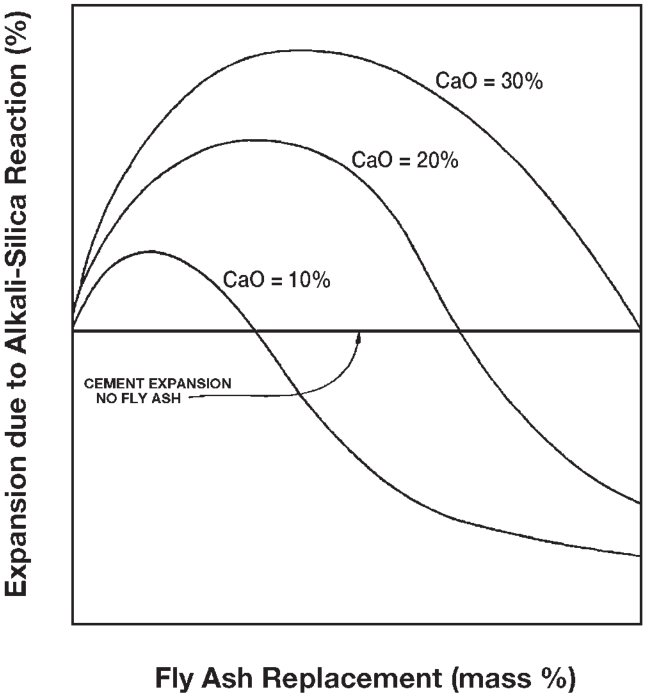

Fig. 7—Theoretical relationship between fly ash replacement level and expansion due to alkali-silica reaction (adapted from Dunstan [41]).

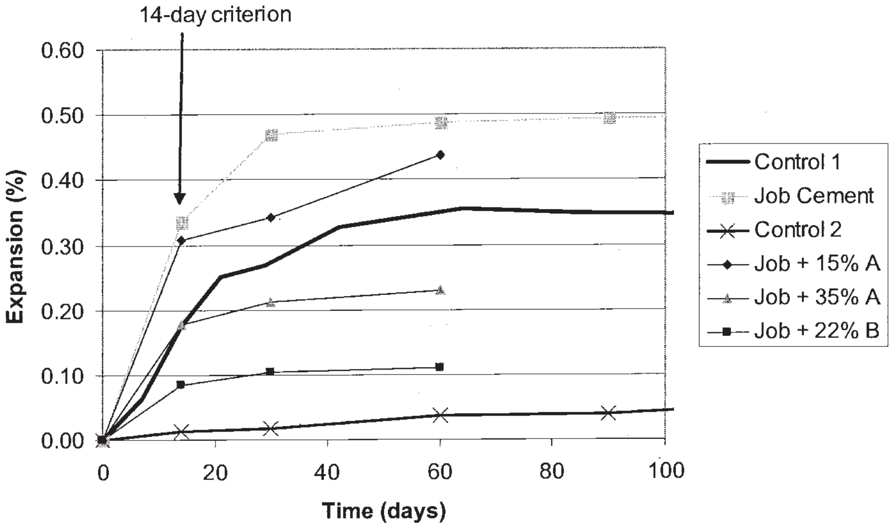

Fig. 8—Illustration of how the new test for the Effectiveness of Fly Ash or Natural Pozzolan in Controlling Alkali-Silica Reactions can be used to select fly ash and replacement levels.

control cements with considerably lower alkali contents may also be used to decrease the expansion of the control specimens (Control 2 in Fig. 8, alkali content = 0.39 % as Na_2O). This tends to make the test more conservative and it also makes the method applicable to regions that only have access to very low alkali cements. The cement used to fabricate the test specimens should have an alkali content equaling or exceeding that of the job cement (Job Cement in Fig. 8, alkali content = 0.75 % as Na_2O). The test is run at several levels of SCM replacement to evaluate how dosage influences mortar bar expansion in the test specimens. This helps to avoid using SCM dosages that are near the pessimum level for that particular cementitious system. In this instance two different fly ashes (A and B in Fig. 8) were used with the job cement at replacement levels of 15 %, 35 %, and 22 %, respectively. A test mixture must exhibit an expansion less than, or equal to, the control specimen (Control 1 in Fig. 8) to be considered "effective." Fly ash B was effective at the 22 % replacement level, while fly ash A was just effective at the 35 % replacement level. In this particular instance, at the replacement levels used in the study, neither fly ash could reduce the expansion below the level of the very low alkali cement (Control 2). In addition, it is apparent that the 14-day criterion tends to provide a conservative estimate of the SCM's ability to reduce expansion.

The new ASR test method was created to provide a better method of selecting good material combinations (those effective at reducing expansion) from the many different sources of material that are commonly available for any given job. The method is relatively quick because test results are available in 14 days. It also provides a more robust solution to the problem of materials selection and optimization than was ever provided by the available alkali test. The method was not designed to provide a mitigative solution to the ASR problem. That is why a joint C09.24-C09.26 Task Group entitled "ASR and Performance Limits" is still actively pursuing more robust methods of eliminating or minimizing the impact of ASR on concrete. The new techniques and associated specification limits will be adopted when they become available.

A new test method was adopted for the effectiveness in contributing to sulfate resistance in 1996. The test method is based on the Standard Test Method for Length Change of Hydraulic-Cement Mortars Exposed to Sulfate Solution, ASTM C 1012. This test method, and the associated specification limits given in C 618, was needed because of the concerns voiced in the literature about how different sources of fly ash influence the performance of concrete exposed to soluble sulfates [38–40,72,73]. Again, the test was adopted to simplify the selection of combinations of materials that could provide the performance needed for specific job requirements. The new test procedure is accompanied by a commentary describing how it should be used and interpreted (see Appendix X2 of C 311). The specification limits can be applied via two different procedures. In both instances the test takes at least six months to complete.

The first method (Procedure A) is very similar to that given in C 1012. The expansion of the mortar bar specimens containing fly ash or natural pozzolan is compared to the absolute expansion limits given in Table 3 of C 618. The absolute expansion limits are selected based on the level of sulfate resistance needed for a particular job. The second method (Procedure B) uses control specimens containing only portland cement to designate the level of expansion that would be acceptable for any particular job. In this instance, test specimens containing various proportions of job cement and SCM are simply checked to make sure that they provide performance equivalent to the control cement. Hence, this is the reason for the 100 % (maximum) expansion limit given in Table 3 of C 618.

Test Requirements Not in the Specification

Various other tests have been proposed and used in research. These have been considered for use in these specifications, but they were not generally applicable or definitive enough to include. These dealt with, among other things, discernment between Classes F and C, heat of hydration, and effect on air entrainment.

This chapter has already suggested that the distinction between Classes F and C fly ash is not properly drawn in ASTM C 618. The subcommittee has worked with a test for hydraulic index in hopes of being able to forecast the possible contribution of a pozzolan to the strength of a concrete mixture. This seemed at least to be one factor that could be isolated. Such a test would help discern between Classes F and C. No such test procedure has proven totally acceptable because the complex contribution to a concrete mixture made by a pozzolan with various cements, in various proportions, at various temperatures, and in various types of concrete mixtures has not allowed any correlations that are acceptable. Manz [74] suggested a performance test measuring strength with no cement in the mixture. If such a mixture gave a measurable strength, the fly ash would be assumed to be Class C; if not, Class F. Various other strength tests of mortars or even of mixtures without aggregate were tried. Temperature rise tests were also tried without conclusive results. Members of the subcommittee have just been slow to accept new testing programs such as those proposed by McKerrall and Ledbetter [75] for Class C fly ash.

These same temperature rise tests on mortars have been used for information on heat of hydration with varying degrees of success. However, in measuring both contribution to strength and heat of hydration, it is more advisable to test a complete concrete mixture rather than a laboratory mortar.

The foam-index test is a quick test to show possible changes in the amount of air-entraining agent required when using fly ash in the concrete. Since it deals with one cement and only the source of fly ash under test, the results apply only to that single combination.

Quality Assurance

Some of the tests discussed earlier could be used as part of a quality assurance program, inasmuch as ASTM C 618 and ASTM C 311 cannot be expected to be a complete quality assurance program. A quality assurance program can better be done by a producer of fly ash than by a user, since the producer will know more about the factors that affect change and will therefore know the tests that really apply to his situation. Fly ash is a by-product of coal combustion in an electric steam power plant and acceptability is therefore more a matter of quality assurance than quality control. Some agencies, such as the U.S. Army Corps of Engineers, once used sealed storage and complete testing before shipment. In 1984, that organization began an effort to avoid the consequent cost and delay by qualifying sources of pozzolan based on prior testing history and producer product certification [76]. Such a program might use some tests not discussed in this chapter that a producer has found to indicate possible changes in product. The ACI Committee reports [5,6] discuss some of those tests that cannot be used in a specification such as ASTM C 618 but can be used effectively for quality assurance.

Silica Fume

History and Use

Silica fume (sometimes referred to as condensed silica fume or microsilica) is a by-product from the production of silicon metal or ferrosilicon metal in submerged-arc electric furnaces [77,78]. According to Malhotra et al. [77], "The first investigations on condensed silica fume began in the late 1940s and since then Norway has made significant advances on various aspects of silica fume." Some of the first field experimentation with silica fume in concrete in the United States was done in Kentucky in 1982. In January 1985, ASTM Subcommittee C09.03.10 organized a task group to develop a specification for the use of silica fume in concrete. According to Richter [79], silica fume can be used to make concrete with the following desirable properties:

(*a*) reduced heat of hydration,
(*b*) retarded alkali-aggregate reaction,
(*c*) reduced freeze-thaw effects and water erosion,
(*d*) high strength,
(*e*) increased sulfate resistance, and
(*f*) reduced permeability.

Silica fume is almost as fine as tobacco smoke. Along with this fineness, it is mostly amorphous (refer to Fig. 2a) and is spherical in shape [80]. The ASTM task group at first thought to add silica fume as a new class or column in the existing ASTM C 618. Test procedures from ASTM C 311 were to be used where possible and modified when needed. Prescription and performance specifications were both to be used when they gave desirable information. By late 1986, it was decided not to try to incorporate silica fume into ASTM C 618. Still, much of the thinking from that specification, as well as ASTM C 311, has guided the preparation of the initial version of the ASTM Standard Specification for Silica Fume for Use in Hydraulic-Cement Concrete and Mortar (C 1240) that was published in 1993. Since then, both the base document and the title of the specification have been subjected to a number of revisions. In 2003, the title of the specification was changed to the ASTM Standard Specification for Silica Fume Used in Cementitious Mixtures, when the deprecated term "mineral admixture" was removed from the document.

Chemical Requirements

Chemical requirements for silica fume are listed in Table 1 of C 1240. They consist of mandatory requirements for silicon dioxide, moisture content, and loss on ignition.

Silicon, reported as silicon dioxide (SiO_2), is usually considered the active constituent in silica fume. The limit was set at a minimum of 85 % when it was found that, with 16 different fumes with a minimum of 85 % SiO_2, no other oxide except potassium oxide (K_2O), which was at 2.27 %, was over 2 % [79]. According to Richter, "most research has been done on silica fume produced from the production of silicon or ferrosilicon alloys. Since very little is known about the other types at the present time, the Canadian specification and ASTM are limiting silicon (expressed as SiO_2) to 85 % and higher" [45,79]. The originating task group from ASTM decided to report elements from the chemical analysis as conventional oxides even though in some fumes none or very little of that oxide is present. Note in Fig. 2a that small amounts of reduced mineral species may occasionally be found in silica fume (moissanite is silicon carbide, SiC). The determination of the bulk chemistry of silica fume had long been hampered because of the lack of high-silica content standards. That limitation was resolved by a round robin conducted in 2003, which yielded a standard reference material (SRM 2696) for silica fume. The remaining two requirements, moisture content and LOI, appear to have been directly adopted from C 618. Moisture content is limited to 3 % maximum to avoid problems in handling. Most of the 6 % LOI is due to "foreign materials" such as carbon and wood chips. These foreign materials do not come from contamination, but are present from the process that produces silica fume.

The early versions of C 1240 had an optional chemical requirement for the available alkali content of silica fume. This requirement was adopted directly from ASTM C 618. However, the requirement was removed in 2000 because it was considered "prescriptive" and a performance test was available for determining the ASR resistance of cement-silica fume combinations. An interesting fact became apparent when the available alkali limit was being balloted for removal. Discussion with both users and producers indicated that few were determining the available alkali content as specified in C 311. Rather most had been determining the total alkali content of the silica fume. They had been conducting this type of testing on a routine basis for as long as they had been testing silica fume. Hence, the current specification allows one to monitor and report the total alkali content of silica fume.

Physical Requirements

Physical requirements are given in Table 2 of C 1240. The physical requirements consist of the determination of oversize, the uniformity of the oversize measurements, the accelerated pozzolanic strength activity index, and specific surface.

Under physical requirements, there is an oversize provision that 10 % (maximum) of the material may be retained on the 45-μm (No. 325) sieve. Normally, all of the silica fume will easily pass through a 45-μm (No. 325) sieve. Material retained on the 45-μm (No. 325) sieve is an indication of foreign materials such as carbon and wood chips in the bulk product. However, Note A of Table 2 warns that agglomerations of extremely fine material can also fail to pass through the sieve. Hence, the task group is currently working on procedural changes to the test method that will provide results that are more concise. The uniformity requirement for the percentage oversize test appears to have been directly adopted from C 618.

With respect to the accelerated pozzolanic activity index test, it should be noted that specimens are cured at 65°C; this is different from any prior methods published in ASTM C 311. The round-robin work conducted for this specification was performed before ASTM C 311 was changed in 1989. The specification limit at seven days, 85 % (minimum) of control, was set with the old C 311 test procedure (pozzolanic activity index, sealed curing at 38°C) but with higher temperatures in mind. The initial version of the test was replaced with the accelerated pozzolanic strength activity index in 2002. This was done to correct some discrepancies in the test method. The major discrepancy in the method was related to the fact that the test specimens (containing 10 % silica fume) were proportioned on a constant flow basis. This caused the test specimens to always have a significantly higher water-to-cementitious-material ratio than the control mixture (formulated at a fixed w/c = 0.485). The new version of the test method allows the use a high-range water reducer in the mixture containing silica fume. This allows the technician to vary the water reducer dosage to make the flow of the test mortar similar to the control mortar, while maintaining a water-to-cementitious-material ratio of 0.485. This change greatly increased the compressive strength of cubes from the test mixture and the specification limit was increased to 105 % (minimum) of the control, to reflect this increase in strength. The note at the bottom of Table 2 in the specification points out that "Accelerated pozzolanic activity index is not to be considered a measure of the compressive strength of concrete containing the silica fume. This is a measure of the reactivity of a given silica fume with a given cement and may vary as to the source of both the silica fume and the cement."

The task group developing the silica fume specification also felt a need to measure particle size or specific surface area because this often appeared to be a property relevant to the use of the material. The air permeability (Blaine fineness) test was evaluated and rejected because of the variation of results between operators and laboratories. Finally, it was decided to measure the specific surface area by the Brunauer, Emmett, and Teller (BET) nitrogen adsorption method. The method is similar to the ASTM Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption, C 1069. A minimum specific surface area of 15 m^2/g is specified in Table 2 of C 1240.

Optional Physical Requirements

Optional physical requirements for silica fume are given in Table 3 of C 1240. The optional physical requirements consist of the determination of the uniformity of air content measurements, the reactivity with cement alkalies, and sulfate resistance. All of the optional physical requirements are currently being re-evaluated by the silica fume task group because they are rather inflexible and, in the instance of the sulfate resistance test, can significantly delay the certification of the material.

The uniformity of air content test was directly adapted from C 311. The test measures the amount of admixture that is required to produce a specific amount of air (18 ± 3 %) in a mortar mixture. The test is meaningful because it reinforces the notion that finely divided materials containing free carbon can have a significant impact on the amount of admixture required to entrain a stable air-void system. However, the test uses mix proportions that consider the silica fume an aggregate addition (rather than a cement replacement). Since the use of a high-range water reducer is not allowed, the water demand of the mixture containing 9 % silica fume tends to be high.

The reactivity with cement alkalies test was directly adapted from C 441. The silica fume replacement is fixed at 10 % by mass of cement. Again, since the water required to produce a plastic mortar mixture is determined using the flow test, the control mix tends to contain much less water than the test mixture containing silica fume. The specification limit is set at an 80 % reduction in expansion as compared to the control specimen. It is uncertain why this value is different from the normal value (75 % reduction), which has been used for natural pozzolans for more than 50 years.

The sulfate resistance test method for silica fume is basically the same as that which was described earlier for fly ash and natural pozzolans (Procedure A, absolute expansion limits). Again, the silica fume content of the mortar mixtures has been set at a replacement of 10 % and the test does not allow for the use of a high-range water reducer to correct for the water demand of the silica fume. The specification limits are also similar to those given for fly ash and natural pozzolans; however, a new expansion category (very high resistance, 0.05 % expansion at one year) has been added to Table 3.

Closing Statements

Enhanced awareness of environmental concerns has led to a resurgence in the use of supplementary cementitious materials. However, it has also changed the way that the materials are employed. In many instances today, especially in the case of fly ash, the user simply wants to be assured that it will not cause any problems in concrete. This is in contrast to the original reason for using the material, which was to enhance specific properties of the concrete mixture. However, in very recent times the push has been to produce high performance concrete on a routine basis for many different applications. These demands require the use of supplementary cementitious materials.

What needs to be done to make the specifications for these materials better? The silica fume specification is only about one decade old and is still in a constant state of revision; hence, that document is currently moving forward at a good pace. However, the specifications for fly ash and natural pozzolans (C 618) are another matter. These specifications are now about 50 years old and sometimes they appear to be firmly rooted in the past rather than looking ahead to future concerns. Current issues, such as co-combustion of biomass with coal, low NO_x burners, precipitator additives, burning of spot market coal, and production of high-reactivity metakaolins, all stretch the specifications to their limit. Many authors have reviewed C 618 and C 311 and many have been critical of them [29,54,74,81–85]. Many of the criticisms have been addressed by the subcommittee; however, much work still needs to be done. The direction that subcommittee C09.24 needs to take to improve these documents is to get back to basics coupled with the use of new analytical strategies or techniques. This suggests a rethinking of many facets of the existing test methods and specifications. There is little doubt of what we need to produce–we need to ensure that the materials are used to produce concrete that meets the needs specified by the user. All efforts must be directed at keeping the new or refined requirements as performance-oriented as possible. This will be necessary because at present it is difficult to predict where changes will occur. For example, some agencies have already developed mixtures of two or more materials (say, silica fume and fly ash) that they now wish to market. What specification, the silica fume or the fly ash, would apply to the composite material? Would either existing specification provide robust enough requirements for the acceptance (or rejection) of the material, or should they even be applied to the new material? If reliable performance limits could be set for the whole group of generic materials covered under the specifications then it would greatly simplify dealing with such mixtures. Mixtures of this type will probably generate great appeal because they have the potential of imparting the benefits from each of the constituents [44,86]. Also, there is no reason to limit the number of materials that can be combined. Performance needs to be the goal.

In conclusion, the supplementary cementitious materials dealt with in this chapter have properties that can provide significant benefits to concrete mixtures. The benefits range from enhanced performance to production of mixtures that are more economical. These benefits have been verified by decades of use in the field. The many tests used in the ASTM specifications discussed in this chapter are significant in controlling and defining the aforementioned properties and their use in the concrete industry. However, these specifications cannot be static. They must be in a continuous state of revision and refinement. This relates directly to the fact that the majority of the materials are by-products from major industries. As these industries change to meet new environmental regulations or the rapidly changing economic climate, they have the potential to alter the quality of the supplementary cementitious materials that are produced.

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Slag as a Cementitious Material

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Preface

IN PREPARING THIS CHAPTER ON SLAG AS A CEmentitious material, the contents of the 4th edition were drawn upon. The author acknowledges the authors of the previous editions, where this subject was included in the chapter on Mineral Admixtures. The authors were C. J. Cain, L. H. Tuthill, and E. C. Higginson, respectively, of each previous edition. This current edition will review and update the topic as addressed by the previous authors, introduce new technology that has been developed, and include up-to-date references.

Introduction

Iron blast furnace slag is used as a supplementary cementitious material in concrete when it is granulated or pelletized, then ground to a fine powder. This chapter will cover the development and requirements of the ASTM standards, a brief history, its use as a blending material with portland cement, and the effects of slag on the freshly mixed and hardened properties of concrete.

The material when used with an appropriate activator to produce mortars or grouts will not be covered in this chapter.

Some of the reasons for using slag as a supplementary cementing material in concrete are [1]:

- higher ultimate strengths with a tendency toward lower early strengths,
- improved resistance to sulfates and seawater,
- lowered expansion from alkali-silica reaction,
- lower temperature rise due to lower heat of hydration,
- better finish and lighter color,
- equivalent durability in freezing and thawing, and
- decreased porosity and chloride penetration.

As a supplementary cementing material, slag replaces a portion of portland cement in concrete at rates generally between 20 % and 80 % by mass of cementitious material, depending on application and desired properties. This replacement can occur by adding slag as a separate material during concrete batching, or can be in the form of a portland-blast furnace slag blended cement. Table 1 shows typical replacement rates for various applications [2].

Definition

Iron blast furnace slag is described by ASTM Terminology Relating to Concrete and Concrete Aggregates (C 125) as "the nonmetallic product, consisting essentially of silicates and aluminosilicates of calcium and other bases, that is developed in the molten condition simultaneously with iron in a blast furnace." ASTM C 125 further defines ground granulated blast furnace slag as "the glassy granular material formed when molten [iron] blast-furnace slag is rapidly chilled as by immersion in water . . . and ground to cement fineness." Like portland cement, ground granulated blast-furnace slag is a hydraulic cement since it sets and hardens by chemical reaction with water, and can do so under water.

Ground blast furnace slag with cementitious properties can also be produced using a pelletization process described in the "Production" section of this chapter.

Ground granulated (or pelletized) blast furnace slag is also commonly referred to as "slag cement" and this term will be used within this chapter to describe both the ground granulated and ground pelletized forms of the product.

History

The first recorded instance of slag being used as a cementitious material was in 1774, when it was combined with hydrated lime and used as a mortar by Loriot. Since the late 19th century, slag cement use has been common in many areas of the world. For instance, slag-lime cements were used in the construction of the Paris underground metro system beginning in 1889. Blended portland blast-furnace slag cements were first produced in the United States in 1896. Portland blast-furnace slag cement was utilized in the Empire State Building's masonry mortar. In the 1950s slag cement as a separate material–added at the concrete mixer–was first developed in South Africa, then gained popularity in many other countries, including the United States and Canada. Separate cementitious slag produced with the pelletizing process was introduced in Canada in the late 1970s. Widespread use of slag cement in the United States began in 1982, with the opening of the granulation and grinding facility at Bethlehem Steel's Sparrows Point, MD steel plant. Popularity of the

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TABLE 1—Suggested Slag Cement Replacement Levels [2]

Concrete Application	Slag Cement*
Concrete Paving	25–50 %
Exterior flatwork not exposed to deicer salts	25–50 %
Exterior flatwork exposed to deicer salts with w/cm ≤ 0.45	25–50 %
Interior flatwork	25–50 %
Basement floors	25–50 %
Footings	30–65 %
Walls and columns	25–50 %
Tilt-up panels	25–50 %
Prestressed concrete	20–50 %
Precast concrete	20–50 %
Concrete blocks	20–50 %
Concrete pavers	20–50 %
High strength concrete	25–50 %
Alkali-silica reaction mitigation	25–70 %
Sulfate resistance	
Type II Equivalence	25–50 %
Type V Equivalence	50–65 %
Lower permeability	25–65 %
Marine exposure	25–70 %
Mass concrete (heat mitigation)	50–80 %

* Percentages indicate replacement for portland cement by mass. These replacement rates are recommended for individual applications and are based on historical performance. Variations in material sources and environmental conditions may require alternate substitution rates. Consult your slag cement supplier for additional assistance. As with all concrete mixtures, trial batches should be performed to verify concrete properties. Results may vary due to a variety of circumstances, including temperature and mixture components, among other things. You should consult your slag cement professional for assistance. Nothing contained herein shall be considered or construed as a warranty or guarantee, either expressed or implied, including any warranty of fitness for a particular purpose.

product in the Mid-Atlantic area of the United States grew quickly, coinciding with publication of ASTM Standard Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortars (C 989) in 1982 and ACI 233-R in 2003 [3].

Use of slag cement expanded rapidly in the following decades with the expansion of granulation and grinding plants in other U.S. regions. Slag cement use has more than tripled in the United States since 1996 to about 3.5 million tons in 2004 (Fig. 1) [4].

Specifications

Slag cement is available as a separate cementitious material and as a component of blended cements. When used as a separate product in concrete, ASTM C 989 is the specification normally cited. The American Association of State Highway and Transportation Officials equivalent specification of the same name is AASHTO M302. If a blended cement is desired, ASTM Standard Specification for Blended Hydraulic Cements (C 595) is specified (or the equivalent AASHTO M240).

In Canada, the applicable standard for all cementitious materials–including portland and slag cement, as well as blended cement–is CSA A3001, Cementitious Materials for Use in Concrete.

Specification Development

ASTM C 989 was originally developed under the jurisdiction of Committee E38 on Resource Recovery in 1982. By starting in ASTM Committee E38, the task group working on the specification was able to avoid unnecessary prescription specifications and instead develop performance-based criteria. That task group succeeded in getting realistic test requirements that had a minimum of long drawn out testing and at the same time included all reasonably effective slags. The task group based its findings on (a) extensive tests of a wide range of ground slags from North America, Japan, England, and South Africa (the last two with extensive use records as admixtures); and (b) all available literature. Once it was developed, jurisdiction for this

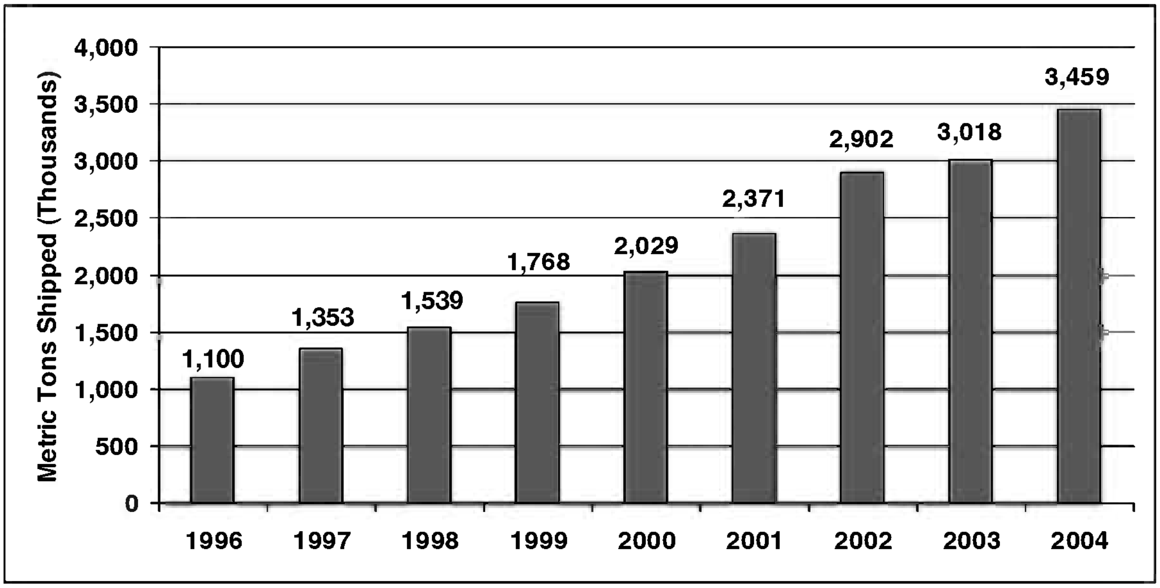

Fig. 1—U.S. slag cement use 1996–2004 [4].

standard was changed to ASTM Committee C09 on Concrete and Concrete Aggregates in November of 1982.

Slag Activity Index and Grade

ASTM C 989 specifies three "grades" of slag cement: Grade 80, Grade 100, and Grade 120. Slag grades are defined by the "slag activity index" (SAI) as shown in Table 2. The SAI is the ratio of the strength of mortar cubes (tested at 7 and 28 days in accordance with ASTM Test Method for Compressive Strength of Hydraulic Cement Mortars (C 109)) that have 50 % slag cement replacement, to the strength of companion cubes made with the reference 100 % portland cement. The reference cement used must have a minimum 28-day strength of 5000 psi and an alkali content between 0.6 and 0.9 %. It was found that performance of slag might vary significantly with other portland cements and that the standard cement provided the best differentiation between grades of slag. Most of the slag cement available in the North America is either a grade 120 or 100 and can be used in virtually all concrete applications. Proportioning of mixtures for field use should be based on applicable concrete tests with project cement.

Laboratory strength test procedures can be misleading in evaluating a slag's performance in the field. Because the test involves curing at room temperatures and the average field temperature of concrete placed in the United States is higher than room temperature, slag is penalized by the lower laboratory temperature. Slag is more sensitive to temperature differences than is portland cement, because slag hydration is accelerated to a greater extent by increases in curing temperature and it is retarded more at lower temperatures. Slag's strength contribution to concrete in the field will often be greater than shown in laboratory tests, but also can be less if field temperatures are low.

It should also be noted that slag is not usually given proper credit for its contribution to better workability in a concrete mixture. Although the ASTM C 109 procedure does adjust for water requirement by equalizing flow, that test and the slump test do not measure the contribution to workability, placeability, or ease of consolidation that slag will make. In concrete, this contribution results in less water required and in better concrete overall. The smooth, dense surface texture and the particle shape of the ground slag account for this benefit [5,6].

TABLE 2—Slag Activity Index (ASTM C 989)

Slag Activity Index Minimum %	Average of last five consecutive samples	Any individual sample
7-Day Index		
Grade 80	—	—
Grade 100	75 %	70 %
Grade 120	95 %	90 %
28-Day Index		
Grade 80	75 %	70 %
Grade 100	95 %	90 %
Grade 120	115 %	110 %

Fineness

ASTM C 989 (Table 2) sets forth not only the slag activity index, but also a fineness requirement of a maximum of 20 % retained on a 45 μm (No. 325) sieve. Specific surface by air permeability is to be determined and reported, but no limits are set for that parameter. One of the benefits of grinding slag separately, as compared to intergrinding with cement at the mill, is the opportunity to grind the slag finer. Slag is harder to grind than portland cement. An interground blend will result in the cement being finer than the slag, which is the opposite of what is desired. By grinding the two materials separately, each can be ground to its optimum fineness. Idorn concludes that the fineness of the slag should be about 5000 to 5500 cm^2/g and portland cement should be about 3800 cm^2/g [7].

Chemical Requirements

Sulfide sulfur is limited to 2.5 % maximum and sulfate ion, reported as SO_3, is limited to 4 % maximum in ASTM C 989 (Table 2). This is to avoid any excess amounts of these products in the concrete, although there is no apparent evidence that this would affect the slag reactions. Some feared that sulfides would produce sulphuric acid and corrode rebars (although it has never happened), and others felt that SO_3 might affect the cement and provide the effect of an over-sulfated cement. The task group did not feel that the specification would be approved without some limits on sulfur. Also, ASTM C 989 provides for the determination of chloride content of the slag but has no limits placed.

Use of Appendicies in ASTM C 989

ASTM C 989 has three appendices. They are X1. Contribution of Slag To Concrete Strength; X2. Sulfate Resistance; and X3. Effectiveness of Slag in Preventing Excessive Expansion of Concrete Due to Alkali-Aggregate Reaction. This nonmandatory information is helpful in interpreting the specification and in evaluating the slag when any of the preceding factors are important to a user of concrete.

It should be noted that the current version of ASTM C 989, Appendix X3, recommends ASTM Test Method for Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction (C 441). This test method utilizes highly reactive pyrex glass as the "aggregate" to determine the effectiveness of pozzolans or slag cement in preventing alkali-silica reaction. In 2004, ASTM approved a new test method, ASTM Test Method for Potential Alkali-Silica Reactivity of Combinations of Cementitious Materials and Aggregate (Accelerated Mortar-Bar Method) (C 1567). This accelerated method utilizes job materials (aggregates, slag and portland cements) and is helpful in determining specific dosages of slag cement required if the job materials are known. ASTM C 1567 may result in lower dosages of slag cement required to control expansion of a specific reactive aggregate than ASTM C 441 (which would only test a specific slag/portland cement combination to control reactivity of a pyrex glass aggregate).

Quality Control Test

In 1985, ASTM Committee C09 gave approval to ASTM Test Method for Hydraulic Activity of Ground Slag by Reaction with Alkali (C 1073). This method originally was designated ASTM E 1085 because its preparation had been started when the originating task group was under Committee E38, but that number was quickly changed by ASTM to a C number so it could be found in the construction volumes of the test methods. ASTM C 1073 states, "This test method can be used as a quality-control test for slag production from a single source after adequate correlation with tests stipulated in Specification C 989." The test is helpful in guiding fineness level required and "in evaluating the hydraulic activity of slags from different sources."

Blended Cement

ASTM C 595 defines three types of blended cement that contain slag:

- Type IS, portland blast-furnace slag cement, which contains between 25 and 70 % ground granulated blast-furnace slag along with portland cement
- Type I(SM), slag-modified portland cement, which contains less than 25 % ground granulated blast-furnace slag along with portland cement
- Type S, "slag cement," which contains at least 70 % ground granulated blast-furnace slag along with portland cement and/or hydrated lime. (Note that Type S "slag cement" is rarely used and that the term "slag cement" in current practice, and according to the American Concrete Institute [3], generally refers to "ground granulated blast furnace slag" as specified by ASTM C 989).

Production

An iron blast furnace (Fig. 2) chemically reduces and converts iron oxides into molten iron. This iron is then subsequently used as a material in steel manufacturing.

When iron is manufactured in a blast furnace, the furnace is batch fed with three materials from the top: iron ore, coke, and limestone. These materials descend–over a 6 to 8 h period–to the bottom of the blast furnace, where preheated air is blown in from the bottom. The final products, tapped periodically from the bottom of the blast furnace, are molten iron and molten slag.

The molten iron is sent to the steel production facility. The molten slag can be processed in three ways, two of which can result in the material that becomes slag cement:

- Air-cooled slag is produced when the molten slag is allowed to cool slowly in ambient air. It can be processed by screening and crushing for use as a construction aggregate, or as a concrete aggregate conforming to ASTM Standard Specification for Concrete Aggregates (C 33). This material has a low glass content, is not cementitious, and cannot be used as a slag cement (Fig. 3).
- Pelletized/expanded slag is cooled quickly through the use of water or steam. Pelletization of slag involves feeding the molen material into a rotary drum which tosses the material into the air resulting in rapid cooling. This product is used as a lightweight aggregate or it can be finely ground into slag cement, if the glass content is high enough.
- Granulated slag is made when the molten material is rapidly quenched in a "granulator" with large quantities of water to produce a sand-like, glassy "granule" that can be finely ground into slag cement. Most of the slag cement available in the United States is produced using a granulator (Fig. 4).

Once the slag has passed through the granulator it is slurried to dewatering bins where most of the granulation water is removed (and recycled in plant operations) and the granules are transported to storage piles.

Slag granules (Fig. 5) are the similar to portland cement clinker, as they both require grinding before they can be classified as hydraulic cements. The morphology of a slag granule at this point is principally a glass (noncrystalline), which allows the material to have cementitious properties, once finely ground. Stockpiled granules are transported to a grinding mill (such as a ball mill or a vertical mill) and ground to a fine powder. The resulting powder is slag cement (Fig. 6).

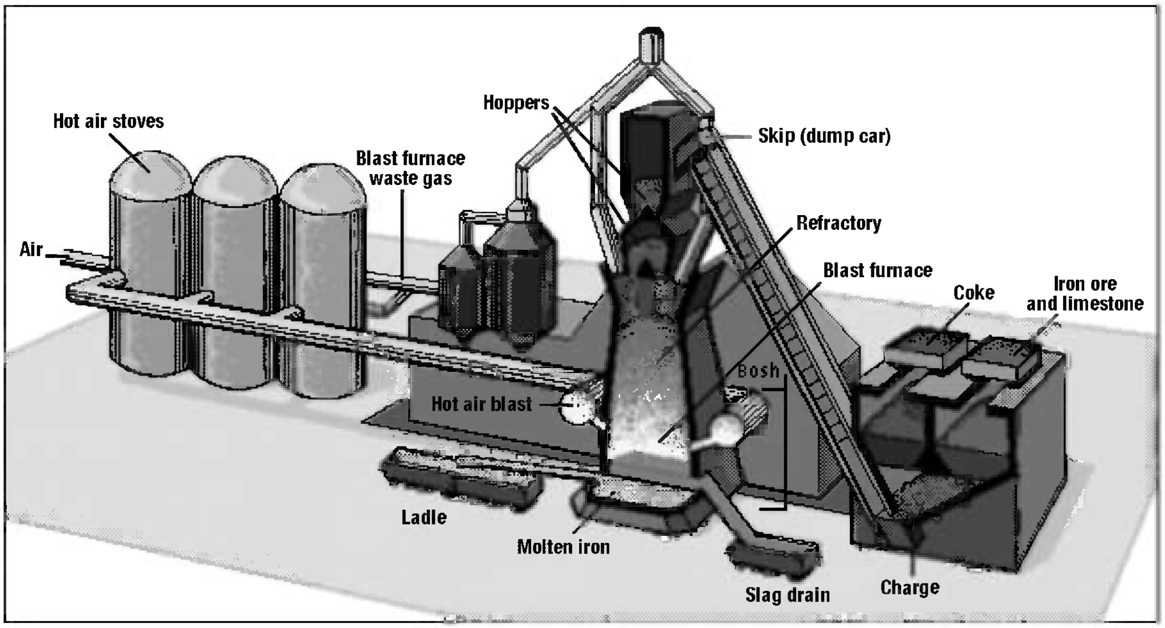

Fig. 2—Iron blast furnace.

Fig. 3—Molten slag being drained into a pit (inset). Cooling in ambient air will produce air-cooled, noncementitious slag. After screening and crushing this slag can be used as a construction aggregate.

Composition and Quality

Typical chemical and physical properties of slag cements available in North America are shown in Table 3 [3]. These properties are determined by the raw feeds into the blast-furnace, the granulation (or pelletization) process, and the grinding operations.

Although slag cement is a by-product of iron production, it is a manufactured material, and is highly consistent in composition and properties because of the quality control and assurance procedures of the blast-furnace and granulation/grinding

Fig. 5—Slag granules in a stockpile (background) waiting to be finely ground into slag cement.

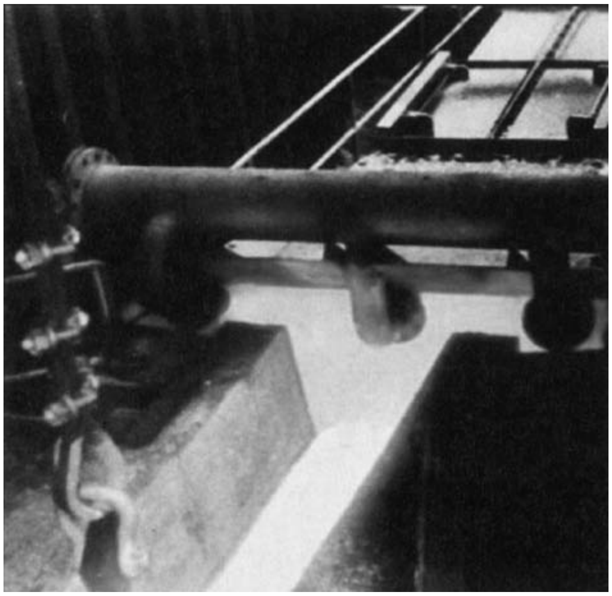

Fig. 4—Molten slag entering a granulator, which will rapidly quench the slag and solidify it in a glassy state. When finely ground, slag with a high glass content (generally greater than 90 %) will have cementitious properties.

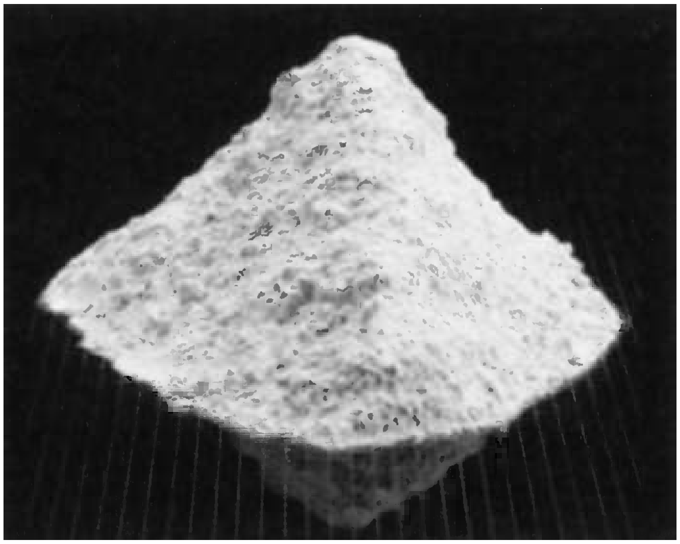

Fig. 6—After grinding, the slag granules become slag cement.

TABLE 3—Typical Composition of U.S. Slag Cements [3]

Chemical Oxides (except sulphur)	Typical Range (% by wt)
SiO_2	32–42 %
Al_2O_3	7–16 %
CaO	32–45 %
MgO	5–15 %
S	0.7–2.2 %
Fe_2O_3	0.1–1.5 %
MnO	0.2–1.0 %
Fineness (Blaine)	4000–6500 cm^2/kg
Specific Gravity	2.85–2.94

operations. During blast furnace operations, the plant operator pays careful attention to the slag chemistry (both composition and variability) as slag behavior is a major consideration in ensuring the quality of hot metal (molten iron). Both the slag granules (before grinding) and slag cement (subsequent to grinding) are closely monitored to ensure high levels of consistency and performance.

Slag reactivity and performance depends on the inter-relationship among numerous variables. The reactivity of slag will depend significantly on glass content, particle size, and slag chemistry; however, many other factors are also involved. To ensure product quality, slag cement manufacturing facilities will each have their own quality procedures, but some of the variables that may be monitored include:

- Particle size and/or Blaine fineness
- Glass content
- Chemical composition
- Color
- Loss on ignition
- Chloride content
- Titanium content
- Basisity

Ultimately, ground granulated blast furnace slag must meet the performance requirements of ASTM C 989 (if a separate material), or the applicable sections of ASTM C 595 (for use in blended cements). As a result of these statistically based specifications and the well-controlled production process, slag cement is found to be one of the most consistent materials used in concrete.

Effect on Hydraulic Activity

As previously mentioned, slag cement is a hydraulic cement, since it chemically hardens by reaction with water and does so under water. The principal hydration product from the slag-water reaction is the same as with portland cement, calcium-silicate hydrate (C-S-H). By itself, slag cement will hydrate at a slower pace than portland cement, but the hydration will continue for a longer period of time.

It is generally recognized that glass content of the granulated slag is a primary factor in determining its contribution to a concrete mixture or its hydraulicity. The granulation process in the slag processing procedure is fundamental. Rapid chilling or quenching of the molten slag inhibits the formation of a crystalline structure. Consequently this quick cooling is necessary so that the slag will be composed largely of glass or amorphous material. One method for determining glass content is by microscopic count. As important as glass content is in slag, investigators have not developed clear-cut relationships between glass content and the strength contribution of slag.

In a portland/slag cement system, both the portland and slag components produce C-S-H when combined with water. However, hydrated portland cement also produces about 15 to 25 % calcium hydroxide, by mass. Calcium hydroxide is a highly soluble mineral; it does not contribute to concrete strength and its presence contributes to increased porosity, efflorescence, decreased aggregate/paste bond, and decreased concrete durability. With a slag/portland system, the relative amount of C-S-H is increased and calcium hydroxide is decreased. This is due to both a dilution of lime-containing portland cement compounds and the reaction of the calcium hydroxide portland cement product with the glass phase of the slag cement (to form more C-S-H). The result of a portland/slag cement system is generally a concrete with significantly decreased permeability, stronger paste-aggregate interface, higher strength, and greater durability.

While the reactivity of slag cement alone is very slow, the reactivity of portland/slag cement binders can be very different. The reactivity of portland/slag cement cementitious systems in concrete (or mortar) depends on the interaction of the slag and portland cements, including the alkali concentration, slag cement glass content and chemical composition, fineness of the portland and slag cements, slag cement proportions and curing temperature.

The hydration of slag cement is significantly activated by alkali hydroxides, calcium hydroxides (among other activators), and heat. Therefore, the addition of even relatively small amounts of portland cement can substantially accelerate the reaction of slag cement. Slag can also be activated by alkali hydroxides alone (alkali-activated concrete) or with hydrated lime (as in some Type S blended cements). Heat increases the solubility of alkali hydroxides, as well as promotes increased reactivity of the portland/slag binder. Therefore, when cured at higher temperature situations (such as heat curing in a precast plant), portland/slag cementitious systems can achieve similar–or even superior–early strengths as portland-only systems [8,9].

Effect on Properties of Freshly Mixed Concrete

Workability, Finishability, and Consolidation

The use of slag cement has several effects on the plastic properties of concrete. It can reduce water demand, increase workability/finishability, slump retention, improve consolidation, and can increase pumpability.

Reduced water demand (or alternatively, increased slump) is shown in Fig. 7, for a specific concrete mixture [10]. As the percentage of slag cement is increased, the amount of water to achieve a given slump is reduced (or the slump for a given amount of water is increased). Generally, the amount of water reduction with slag cement can vary from 0 to 5 %.

Workability and finishability are difficult to quantitatively measure, but are important parameters for concrete contractors, since they often dictate the pace and manpower requirements to perform a finishing task, and also impact the quality of the finished concrete surface. Concrete containing slag cement is often cited as having superior workability and finishability properties. This is attributed by several researchers

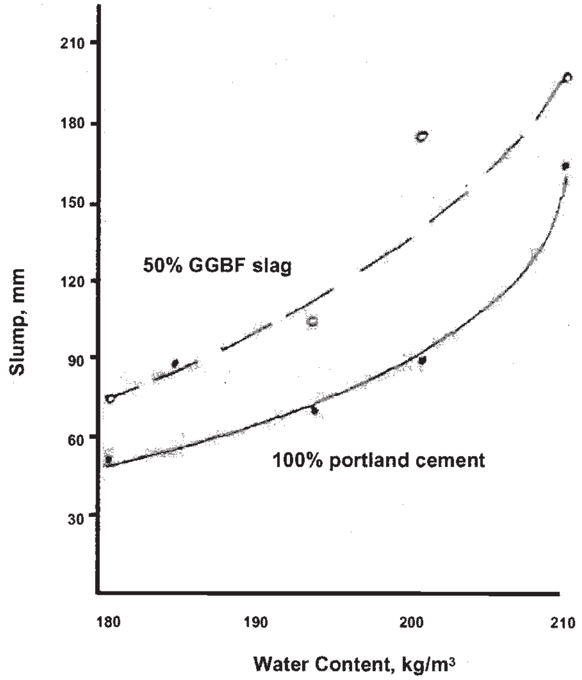

Fig. 7—Slump versus water content for a plain portland cement concrete and the same concrete with 50 % slag cement replacement [10].

[5,6,10,11] to improved rheological properties and particle characteristics, including:

- Increased paste cohesiveness and volume
- Smooth, dense surface of slag cement particles
- Low water absorption after mixing
- Better particle dispersion
- Higher fluidity of paste
- Reduced vibration to achieve consolidation

These improvements may reduce the time of placement of concrete, improve consolidation of concrete, particularly in congested reinforced members, reduce vibration requirements, allow increased speeds for slip-form pavers, improve surface finish of flatwork, and produce finishes with fewer "bugholes" in formed surfaces.

Bleeding

Bleeding capacity and bleeding rate of concrete with and without slag cement are similar. The main difference will be whether the slag is more coarsely or finely ground when compared with the portland cement it is replacing, and whether or not the unit volume of water is the same. Bleeding may be reduced when slag fineness is higher or unit water content is lower. Bleeding capacity and rate are important, particularly with regard to finishing, as concrete finished too soon or too late (with respect to bleed water formation) may result in poor surface durability and plastic shrinkage cracking.

Time of Set

Slag cement at levels above 25 % of cementitious material by mass will generally increase the time of set of concrete at 70°F. This difference will reduce substantially and may become insignificant as the temperature increases to above 85°F; in cooler temperatures, time of set will increase significantly. Figure 8 illustrates, for a specific concrete mixture, the times of set for a range of slag cement percentages and temperatures [12].

In higher temperatures, a longer time of set may be an advantage as it will provide more working time; however, at lower temperatures, chloride or non-chloride accelerating admixtures, and use of heated materials are effective in reducing time of set. Also, because the portland cement setting properties, and the quantity of portland cement in a mixture significantly affect time of set, a reduction in slag percentage or an increase in portland cement content will also be a potential strategy for maintaining appropriate times of set at low temperatures.

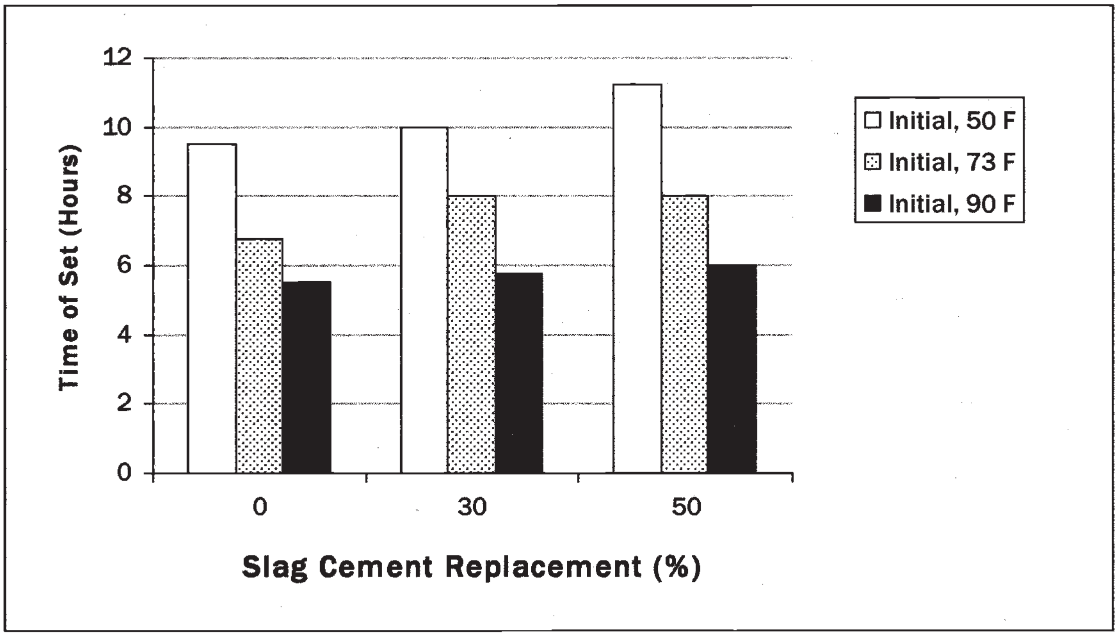

Fig. 8—Time of set characteristics for a typical concrete at various temperatures and slag cement replacement percentages [12].

Curing Requirements

As with all concrete, proper curing practices must be followed to achieve desired concrete properties. Curing is necessary to ensure that the concrete will have sufficient moisture available to develop required properties. Typical curing practices, such as fogging, ponding, wet burlap, polyethylene, and curing compound are compatible with concrete containing slag cement. Duration of curing is dictated by many factors including temperature, rate of evaporation, water/cementitious ratio, and rate of strength gain. Duration of curing may or may not be affected by the presence of slag cement in the concrete. Higher percentages of slag cement (particularly in mass concrete placements, where slag cement percentage may be up to 80 %) may necessitate longer curing times. However, lower percentages may gain sufficient strength and maturity in the time that plain portland cement concrete is normally cured, and curing duration is not extended.

Effect on Properties of Hardened Concrete

Strength

Slag cement will generally have a positive effect on the ultimate strength (both compressive and flexural) of concrete. This is due principally to the higher volume of calcium-silicate hydrate that is generated in a portland/slag cement system, as well as an improved paste-aggregate bond. The initially slower hydration characteristics of a portland/slag cement system will normally cause lower initial strength gain compared to a plain portland cement system. Concrete made with slag cement typically available in North America will generally produce higher strength than an equivalent plain portland cement concrete mixture, between 7 and 14 days when cured at 70° or higher. Early strengths can be significantly affected by several factors including the temperature, the characteristics of the portland cement, the w/cm ratio and the curing conditions. As noted previously, the application of heat, as practiced in some precast concrete production operations, will tend to significantly increase the rate of strength gain of slag cement concrete to the point that adequate release strength can be achieved in less than 24 h without an increase in total cementitious materials content [8,13].

Figure 9 illustrates the compressive strength of mortar cubes and concrete mixtures at various slag cement replacement levels and ages.

Permeability

It is well documented that the use of slag cement in concrete will result in significant reductions in permeability [14–17]. Reducing permeability is a key factor in improving many aspects of concrete durability, such as corrosion resistance, mitigating sulfate attack, and combating alkali-silica reaction. As permeability is reduced the transport of deleterious substances within the concrete and propagation of deterioration mechanisms are commensurately reduced.

Permeability is decreased principally by the higher volume of calcium-silicate hydrate generated in a slag/portland cement system. The microstructure is changed as pores are partly filled with C-S-H in lieu of calcium hydroxide. Overall pore size is reduced and the interconnectivity of pores becomes more tortuous. Permeability reductions begin generally within the first 28 days of placement, and continue as long as hydration continues. The reduction in permeability beyond 28 days is much more significant in slag cement concrete than in plain portland cement concrete. Additionally, permeability reductions are positively correlated with the percent of slag replacement, through 70 % or greater. Figure 10 shows an example of the reduction in electrical conductivity due to increased slag cement contents–as measured by ASTM Standard Test Method for Electrical Indi-

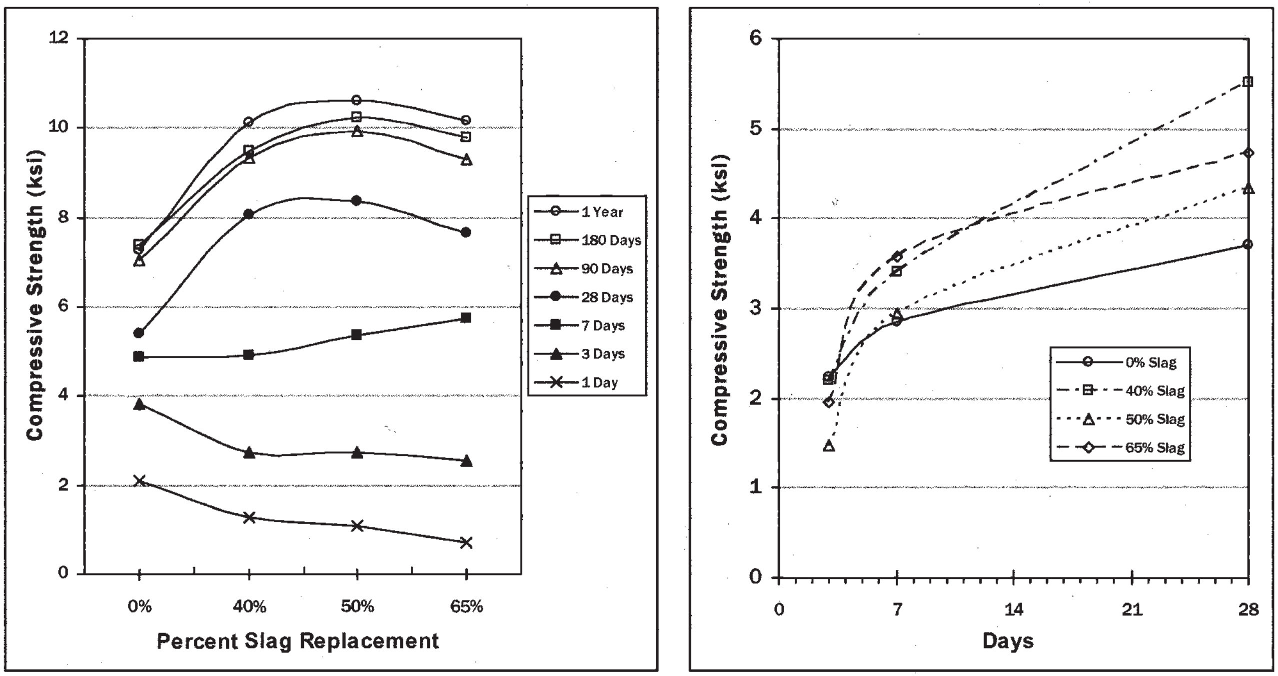

Fig. 9—(a) Mortar cube strength at various slag cement replacement levels, from one day to one year [9]; (b) concrete compressive strength at various slag cement replacement levels, from 3 to 28 days [9].

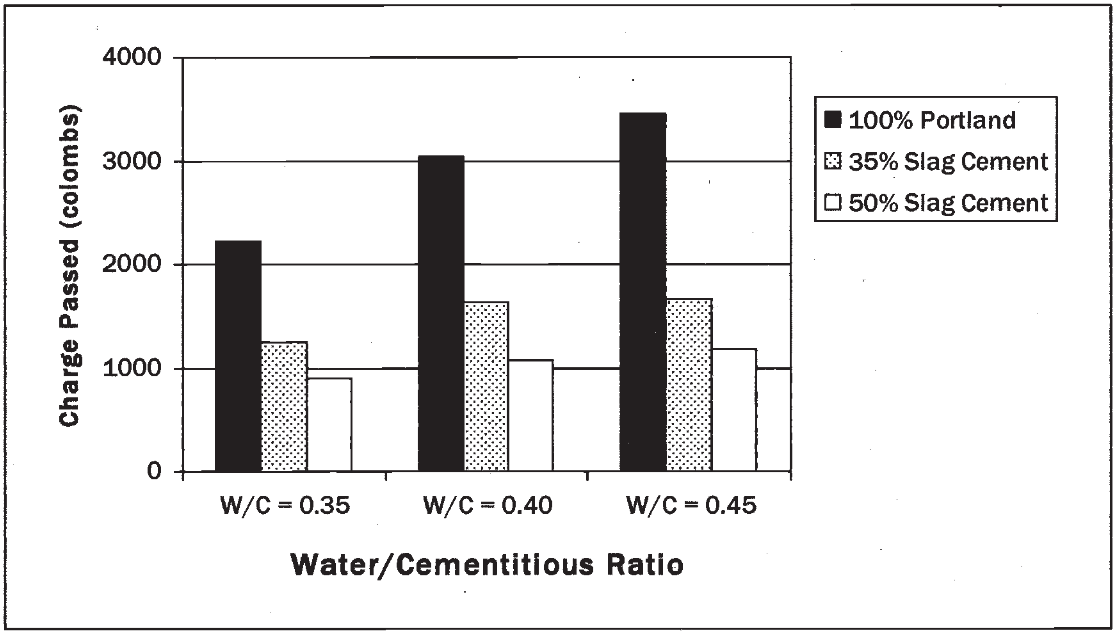

Fig. 10—Permeability of concrete based on the rapid chloride permeability test (ASTM C 1202) [adapted from Ref 14]. Cementitious materials content is 635 lb/cu yd. "Low" permeability is generally defined as less than 2000 coulombs.

cation of Concrete's Ability to Resist Chloride Ion Penetration (C 1202)–at a range of w/cm from 0.35–0.45 [14].

Corrosion Resistance

The use of slag cement in concrete has a positive influence on the corrosion resistance of reinforced concrete, as noted by many researchers [5,9,16–21]. Reduced permeability plays the largest role in this effect, by limiting the transport of chlorides to the reinforcing steel thus maintaining the passivity of the steel. Additionally, the slight reduction of pH in a portland/slag system has been shown to not affect steel reinforcement passivity.

Some research has indicated that even when a critical concentration of chloride ions do reach the reinforcing steel and corrosion is initiated, the rate of corrosion is slowed in concrete containing slag cement [20]. The research showed that increasing slag contents (between 25 and 50 %) resulted in increased concrete electrical resistivity and lower chloride diffusion rates. The reduced diffusion rates are attributed to both reduced permeability from pore structure refinement and greater chloride binding capacity of slag cement concrete. Another study concluded that the corrosion rate in cracked concrete containing as little as 25 % slag cement is also reduced [21]. The researcher suggested that the lower corrosion is due to both continued long-term hydration inducing autogenous healing of cracks, and higher resistivity of the concrete.

Mitigation of Alkali-Silica Reaction

Slag cement can significantly reduce or eliminate the expansion potential of a reactive aggregate, as documented by numerous researchers [9,19,22–24]. Slag cement mitigates ASR in several ways:

- It reduces the total alkalis in the system thus reducing the alkali-silica ratio.
- It consumes alkalis in hydration, making them unavailable for reaction.
- It reduces permeability, therefore slowing the mobility of the alkali.
- It reduces the hydroxyl concentration of pore liquid that reacts with an aggregate.

When using slag cement to reduce ASR, it is important to use an appropriate level of slag cement in the mixture. Several factors will influence the rate and severity of ASR, including aggregate reactivity, portland cement alkali content, presence of water in the concrete, wet/dry cycles, and aggregate size. Therefore testing is recommended to ensure that appropriate expansion reduction can be achieved. The use of ASTM C 1567 can serve to quickly determine (within 16 days) whether specific combinations of portland and slag cements and aggregates will exhibit expansive reactions. If ASTM C 1567 is inconclusive or if greater assurance of nonreactivity is required, ASTM Standard Test Method for Determination of Length Change of Concrete Due to Alkali-Silica Reaction (C 1293) can be used, but this test takes one year to run. Although ASR reduction can be accomplished with as little as 25 % slag cement, often 40 % to as much as 70 % slag cement may be needed to reduce potential expansion to the desired level [3,23,25].

Figure 11 is one example of how slag cement can reduce ASR. The concrete was made with two reactive aggregates in combination with a high-alkali portland cement. Slag cement levels varied from 0 to 50 % and were measured using the concrete prism test ASTM C 1293. To mitigate ASR in the Spratt limestone, a slag cement level of 35 % of cementitious material would be required, while with the Sudsbury greywacke, 25 % would be required [23].

Mitigation of Sulfate Attack

External sulfate attack–where waterborne sulfates react with hydration products of the tri-calcium aluminate phase of portland cement and with calcium hydroxide to form expansive ettringite–can be controlled with appropriate amounts of slag

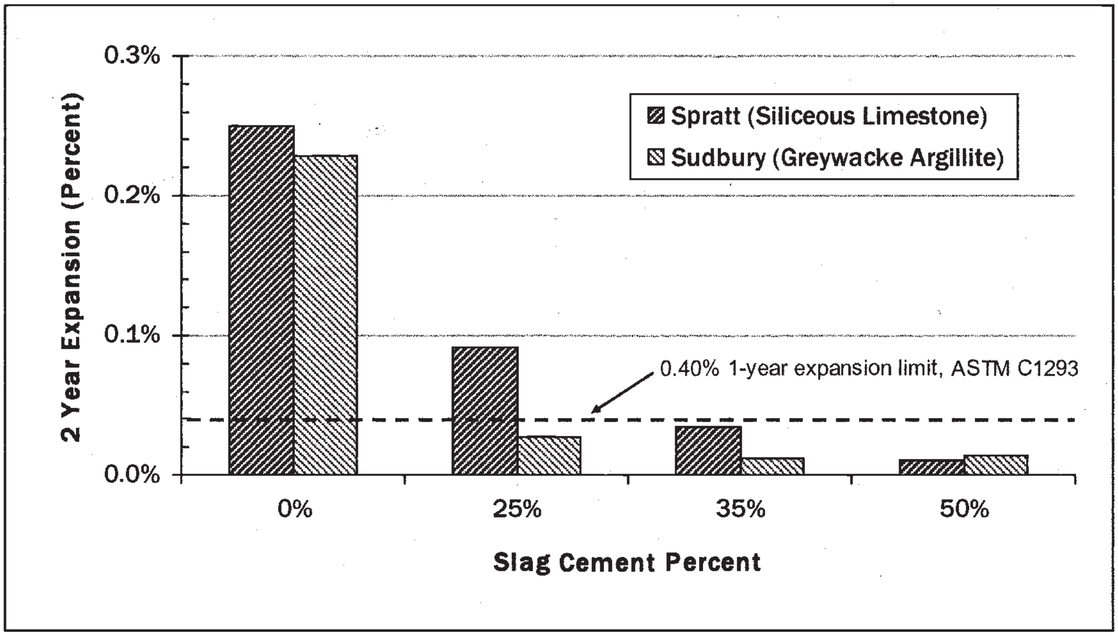

Fig. 11—Mortar bars, tested in accordance with ASTM C 1293, tested for two years at various levels of slag cement substitution, with two different reactive aggregates [adapted from Ref 23]. Using 1-year expansion limits of 0.40 % as a guide (ASTM C 1293 Appendix X1), expansion of the Spratt aggregate is mitigated using 35 % slag cement, while the Sudbury aggregate is mitigated at 25 %.

cement in concrete. Slag cement can reduce sulfate attack through three mechanisms:

- Reduction in concrete permeability, thus reducing transport of sulfates into the concrete.
- Reduction in calcium hydroxide, which is a contributor in the reaction.
- Reduction in the total amount of tri-calcium aluminate through dilution.

Slag cement can be used in conjunction with a Type I or II cement to provide moderate or high levels of sulfate resistance. Very high levels of resistance can be achieved with a combination of Type V and slag cements.

It is important to test the specific materials that will be used in concrete to determine the appropriate amount of slag cement needed to achieve the required level of protection. ASTM C 989 proposes expansion limits, using ASTM Standard Test Method for Length Change of Hydraulic-Cement Mortars Exposed to a Sulfate Solution (C 1012), of 0.10 and 0.05 % at six months to achieve moderate and high levels of sulfate resistance, respectively.

The alumina content of slag cement can influence its ability to mitigate sulfate attack; therefore, it is particularly important to evaluate the sulfate resistance of the portland/slag cement combination if the alumina content of the slag cement exceeds 11 %. This situation may require higher replacement levels of portland with slag cement to achieve an appropriate level of sulfate resistance.

Figure 12 shows the effect of slag cement replacement on sulfate resistance for a slag cement combined with Type I portland cement. For this concrete, a "high" level of sulfate resistance was achieved at 35 % replacement [26]. In many cases, as much as 50 % slag cement may be needed for moderate sulfate resistance, and up to 65 % for high sulfate resistance.

A different type of sulfate attack, delayed ettringite formation (DEF, or sometimes referred to as "internal sulfate attack"), occurs when concrete is subject to high (>160°F) curing temperatures. This is one reason that curing temperatures are limited in precast operations and controlled in mass concrete pours. Although the mechanism of DEF is not completely understood, research has found that the use of slag cement may be effective in controlling DEF expansion at higher temperatures. One researcher found that as little as 17.5 % slag cement substitution was effective in eliminating DEF expansion in temperatures up to 195°F, with no strength penalty even in early strength [27]. Another researcher proposed that at least 35 % slag cement should be used to mitigate DEF in higher-temperature curing (>160°F for most portland cements or >150°F for portland cements with rapid early strength development) [28].

Reduction of Heat in Mass Concrete

The hydration characteristics of slag cement, as well as its increased strength potential, enable it to facilitate the control of heat and mitigation of cracking in mass concrete placements. Typically, when the minimum thickness of a concrete element is large enough to cause concern that the heat generated by cement hydration will result in a high maximum temperature in concrete, as well as a significant differential temperature between the internal concrete and external surface, measures to reduce heat are required. Among other measures that can be taken to control heat (e.g., insulation blankets, cooling pipes, reduced portland cement content), the use of slag cement has been found to be highly effective. Slag cement helps reduce heat by:

- Reducing portland cement content by the percentage of slag used.
- Reducing the rate of early heat generation due to slag cement hydration characteristics.
- Reducing the amount of total cementitious material required to achieve required strength.

Another benefit of using slag cement is the reduced potential for delayed ettringite formation, discussed previously in

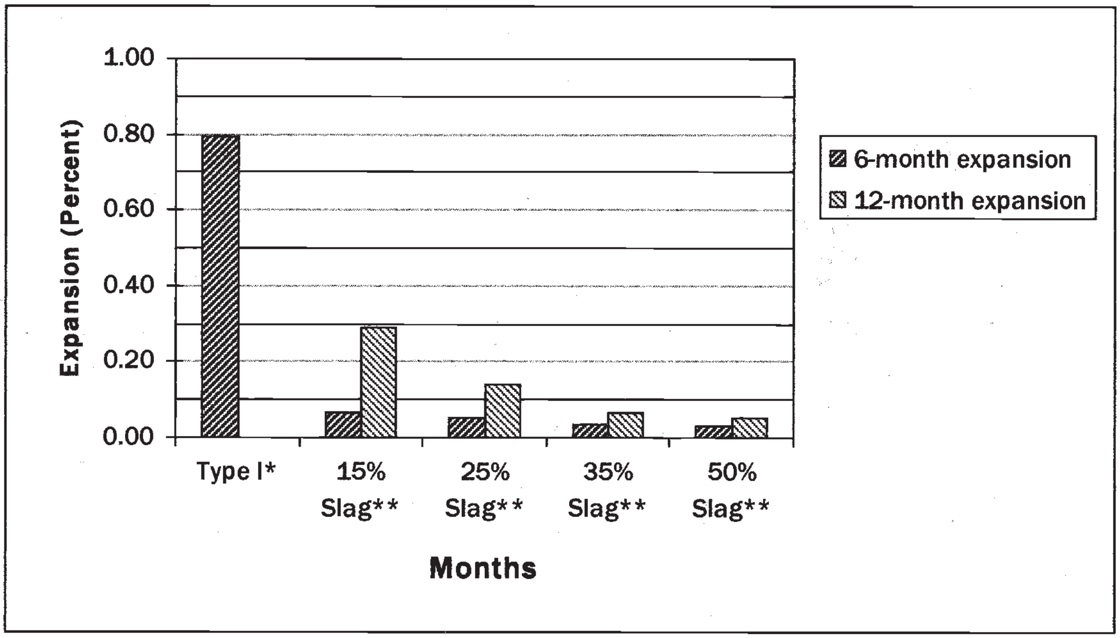

Fig. 12—Testing in accordance with ASTM C 1012 shows the sulfate expansion of a mortar bar made with Type I (non-sulfate resistant) cement and various levels of slag cement substitution. ASTM C 989 proposes that mortar bars that expand less than 0.10 % at 6 months achieve equivalence to a Type II moderate sulfate resistant cement, and 0.05 % at 6 months achieve equivalence to a Type V high sulfate resistant cement. These levels are achieved by 15 and 35 % slag cement, respectively [26].

the section on sulfate attack. One of the reasons for limiting peak temperature in mass concrete is DEF potential.

Figure 13 shows the effect of slag cement on heat of hydration from 0 to 75 % substitution when tested in a semi-adiabatic condition. Not only is the peak specific heat of hydration reduced by nearly 50 % at the 75 % substitution level, but the area under the curve, representing total heat generated, is reduced by an even greater amount. In adiabatic testing, the increase in temperature accelerates the hydration of portland/slag cement systems; therefore, to achieve appropriate heat reduction in mass concrete elements, a slag cement content from 65 to 80 % of cementitious material (by mass) is usually recommended. Figure 14 shows, for the 10-ft thick massive footings on the I-895 Pocahontas Parkway Bridge in Virginia, the temperature rise in the center and surface of a 10-ft thick concrete footing, using 75 % slag cement and 564 lb/cy (334 kg/cm) total cementitious materials. Peak temperature was limited to 130°F and differential did not exceed 28°F, well within specification limits [30].

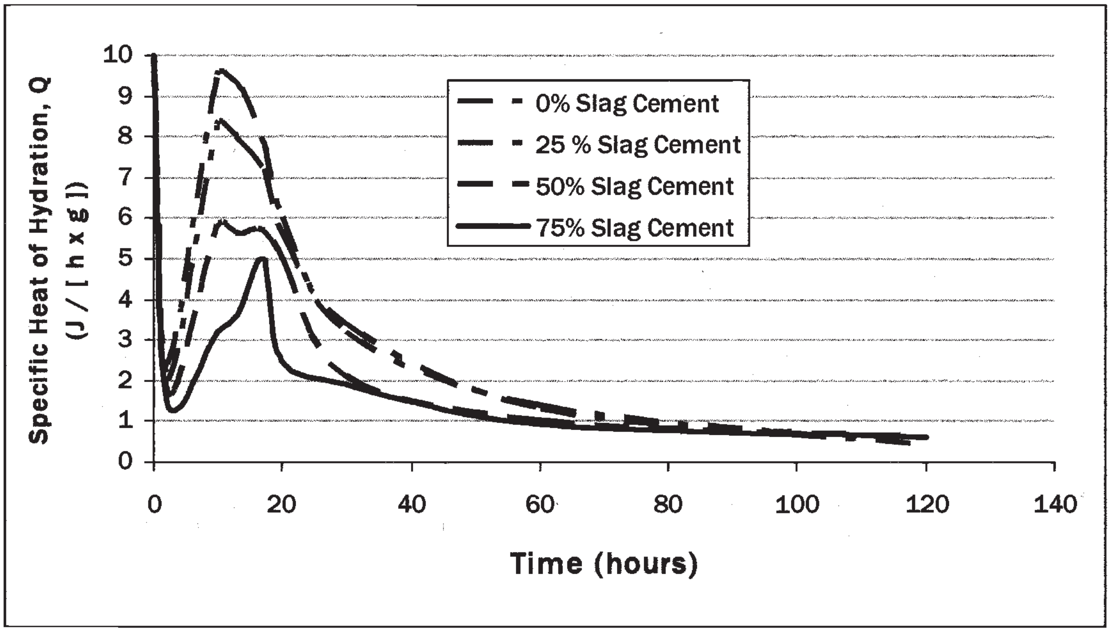

Fig. 13—Specific heat of hydration of plain portland cement and substitution with slag cement up to 75 % [29].

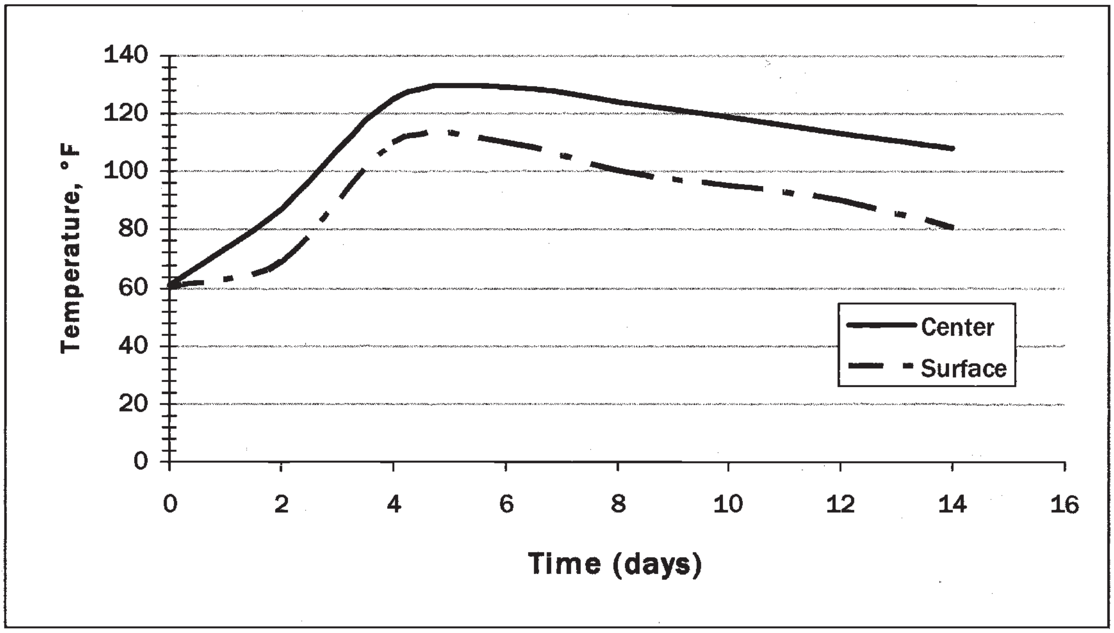

Fig. 14—Temperature of concrete at the center a surface of 10-ft thick footing for I-895 Pocahontas Parkway Bridge in Virginia [30]. A 75 % slag cement concrete mixture with 564 lb/cy of cementitious material was able to not exceed maximum specification requirements for peak and differential temperatures (specification values of 165°F and 35°F, respectively).

Shrinkage

Shrinkage in slag cement concrete is generally not significantly different than shrinkage in plain portland cement concrete. Individual studies have found shrinkage to be less, more, or the same when comparing slag and non-slag concretes. A comprehensive study, which examined 32 references and analyzed data from 62 comparable concrete mixtures, concluded that in unrestrained conditions, the drying shrinkage of concrete containing slag cement was only marginally higher than concrete without slag cement [31]. The average increase in shrinkage was 2.9 %, and when corrected for paste volume (due to slag cement's lower specific gravity than portland), the difference was reduced to 1.5 % (Fig. 15). Also, the amount of slag cement in the mixture, when varied from 20 to 80 %, had no discernable

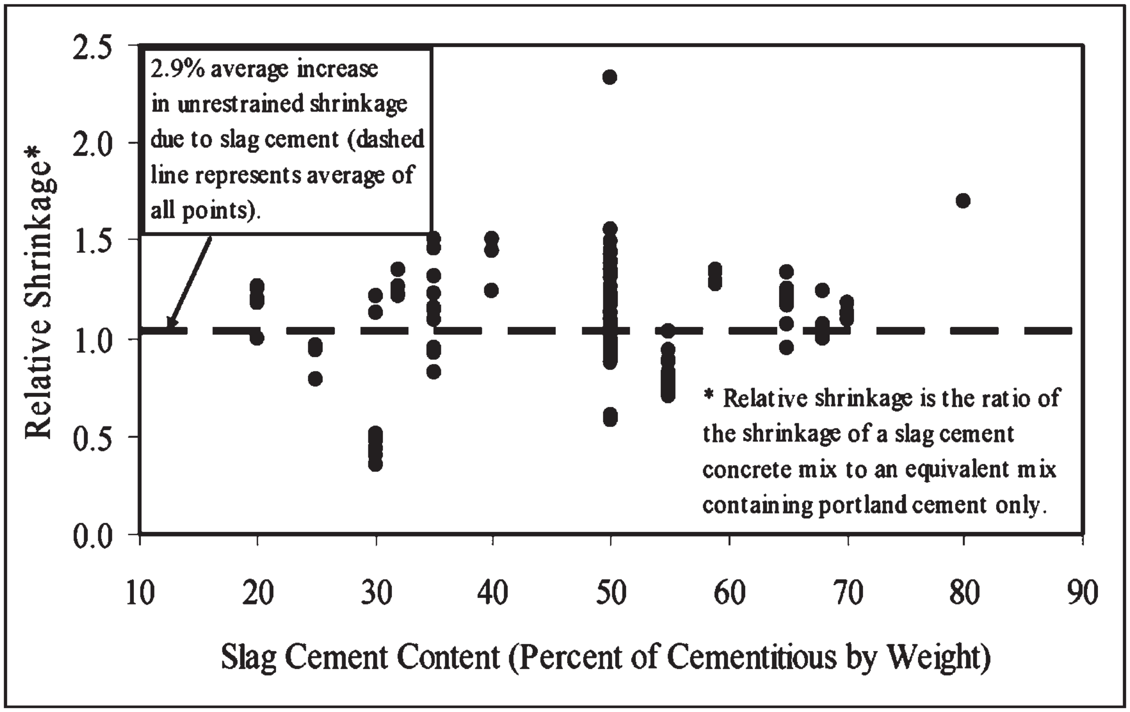

Fig. 15—Relative shrinkage (see note above) of slag cement concrete at various slag substitution levels [31]. Over 62 mixtures, the increase of shrinkage of slag versus non-slag concrete was very small (2.9 %), and no correlation was found between shrinkage and slag substitution level.

effect on the level of shrinkage. Other factors, such as environmental conditions (temperature, humidity), surface-to-volume ratio of the concrete member, curing practices, and material factors other than slag cement (volume and elastic modulus of aggregate, paste content, w/cm of paste) had the most significant effects on shrinkage.

As minimizing paste content (or, conversely, aggregate volume) is critical in limiting shrinkage, mixtures containing slag cement can reduce shrinkage by:

- Adjusting paste volume of the mixture to take into account the lower specific gravity (and higher relative volume) of the slag versus portland cement.
- Where applicable, the higher ultimate strength of slag cement may allow a decrease in total cementitious material, thus reducing total paste volume.

A comprehensive study of creep in slag cement mixtures has not been undertaken, but several individual studies have indicated that long-term creep may be less [32,33]. One particular study of restrained shrinkage cracking of slag cement concrete indicated that cracking was delayed to later ages, and resulted in smaller crack widths [33].

Durability in Freezing and Thawing Environments

Most studies of concrete produced with slag cement under freezing and thawing conditions show that the concrete is durable and compares similarly to ordinary portland cement concrete [5,9,34–38]. As with all concrete, freezing and thawing resistance is closely related to development of an appropriate air void system with adequate air content and proper bubble size and spacing. Slag cement contains no carbon and has relatively consistent particle size, so entrainment of air is generally no more variable than with concrete containing only portland cement. Additionally, adequate strength must be developed in any concrete prior to the first frost cycle, and repeated cycles of freezing and thawing. Because slag cement concrete strength gains are more sensitive to temperature—and because these gains generally develop more slowly at early ages—it is important to ensure that adequate strength is achieved prior to freezing (or, if early freezing is expected, to protect the concrete appropriately).

Any concrete exposed to deicing salts in freezing and thawing environments is potentially susceptible to scaling. When good concreting design and placement practices are used, slag cement concrete—with up to 50 % replacement for portland cement—has provided good scaling durability in the field. However, many laboratory studies using ASTM Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals (C 672) (or similar tests) have indicated that concrete made with slag cement is less resistant to deicer salt scaling, especially at higher replacement levels [39–41]. Other laboratory and field studies have shown similar or even improved scaling resistance with slag cement concrete, compared to non-slag concrete [9,34,37,38]. ACI 318, Building Code Requirements for Structural Concrete, allows up to 50 % slag cement replacement when concrete is exposed to deicing chemicals, assuming that w/cm is 0.45 or less, and concrete compressive strength is at least 4500 psi. Proper finishing and appropriate curing techniques are particularly important for concrete of any type that will be exposed to deicer salts and freezing/thawing conditions. Slag cement concrete, as noted above, may require longer curing times, especially if placement temperatures are low. Finishing practices must take into account potentially longer times of set and differences in bleed water rates and amounts. In a recent survey [41], several Northern U.S. State Departments of Transportation (that specify and utilize slag cement in concrete) have not experienced problems with scaling, nor has the the Ontario Ministry of Transportation [39].

Some researchers feel that ASTM C 672 is a particularly harsh test that does not correlate well with the performance of field concrete made with slag (or fly ash) [42–44]. These researchers suggest that a different testing standard, BNQ NQ 2621-900—the laboratory scaling resistance test procedure adopted by the Quebec Ministry of Transportation—may provide significantly improved correlation between lab and field performance of concretes made with supplementary cementing materials.

Color

Slag cement is a light-colored material when finely ground, generally much lighter than portland cement, fly ash, or silica fume (Fig. 16). The light color of slag cement generally will give concrete a lighter appearance. In exposed architectural concrete this is generally considered a positive attribute and, in some cases (such as the Canadian Embassy in Washington, DC, Fig. 17; or the Rock 'n Roll Hall of Fame in Cleveland, Fig. 18), has been used in lieu of, or in conjunction with, white portland cement. In highways and parking facilities, the light color improves night visibility and may reduce lighting requirements. The Delaware Department of Transportation eliminated the requirement for painting Jersey median barriers when slag cement was used, because of their increased visibility.

In colored concrete, the use of slag may reduce the amount of pigment or coloring agent required for certain colors; however, darker colors may require more pigment. In any case, increased percentages of slag cement will increase the lightness of the concrete.

Slag cement may help lessen the propensity of concrete or mortar to effloresce. The decreased level of calcium hydroxide—a principal contributor to efflorescence staining as it leaches out of concrete or mortar, as well as lowered permeability—helps achieve this reduction.

Another aspect of slag cement is the tendency of hardened concrete containing the material to temporarily turn a dark

Fig. 16—Cementitious materials from top, going clockwise: slag cement, silica fume, Class C fly ash, and portland cement. Fly ash produces lighter colored concrete as slag cement substitution levels increase.

Fig. 17—Canadian embassy in Washington, DC used slag cement in architectural concrete to achieve a very light color.

green or green-blue color. Typically called "greening," this phenomenon is due to a reaction of sulfide sulfur in slag cement with various portland cement compounds. The propensity toward greening depends on the rate of oxidation, the amount of slag in the concrete, curing conditions, and porosity of the concrete surface. Visible greening is a harmless condition, and will normally disappear with a few days' exposure to air and light (this propagates an oxidation reaction that eliminates the greening effect). Concrete where formwork remained for extended periods, or has undergone high levels and long periods of moist curing, may exhibit greening for longer periods of time. Figure 19 illustrates greening in exposed architectural concrete at the Canadian Embassy in Washington, DC shortly after form removal. Because form stripping was delayed several months, greening was initially extensive, but disappeared after several weeks and developed the typical light color of slag cement concrete (Fig. 17) [45]. Often, concrete test cylinders that appear very light on the outside will exhibit greening on the inside once broken. Additionally, old concrete that contains slag and is being demolished may show internal evidence of

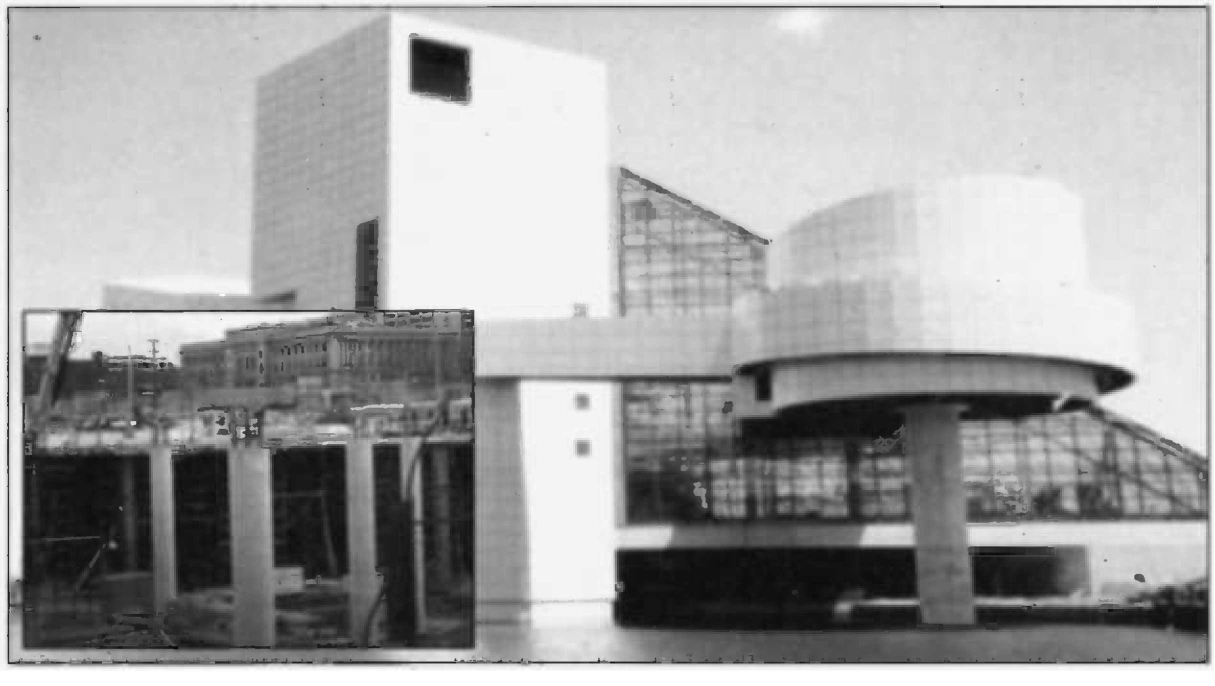

Fig. 18—Rock 'n Roll Hall of Fame in Cleveland used slag cement concrete in the exposed concrete pier foundation (inset) in lieu of white portland cement to closely match the white color of the tile exterior.

Fig. 19—Greening is evident after forms were removed from the Canadian Embassy in Washington, DC. Greening disappeared after several weeks resulting in the desired light concrete appearance of slag cement concrete (see Fig. 17).

greening, once the concrete is cracked and opened. The only time greening might be a concern is in those situations where appearance is important *and* when concrete is not allowed to be exposed to air and/or light (such as a swimming pool). In these cases, the use of slag cement is not recommended.

Compatibility with Other Materials

Aggregates

Slag cement compatibility with aggregates is no less than the compatibility of portland cement with aggregates. When aggregates are susceptible to alkali-silica reaction, slag cement reduces the potential for expansion, as previously discussed. Additionally, slag cement tends to improve the paste-aggregate interface thus producing a stronger bond and a less permeable interface [46].

Chemical Admixtures

When used with chemical admixtures, slag cement has no known limitations, and is compatible in the same way as portland cement. As with any change to a concrete mixture, when slag cement replaces a portion of the portland cement, small adjustments may need to be made to admixture addition rates. For instance, if the slag cement is finer than the portland cement it replaces, air entraining admixture addition may increase slightly. However, once the rate has been established, highly consistent air contents are achievable, as slag is a consistent material that contains no carbon.

Other Supplementary Cementitious Materials

Slag cement is compatible with all supplementary cementitious materials, such as fly ash and silica fume [47]. Using three cementitious materials in a concrete mixture is referred to as a ternary mixture, and is gaining increasing acceptance by specifiers.

Silica fume is sometimes used in high-performance concrete to achieve high levels of strength or low permeability, or both. The use of slag cement in combination with silica fume has the benefits of improving the workability of the portland/silica fume system to decrease stickiness of the mixture and improve finishability. Additionally, research has shown that when both early and long-term strength or permeability reductions, or both, are important, a portland/slag/silica fume system can achieve superior results by enhancing strength and permeability reductions at early ages (principally through the silica fume), and beyond 14 days and throughout the life of the structure (principally through the slag cement) [46]. An early high-strength concrete application for high-rise construction was the Key Tower in Cleveland, which utilized 1050 lb/yd^3 of cementitious material, 65 % portland cement, 27 % slag cement, and 9 % silica fume and which achieved 15 000 psi field strength in 56 days. The use of slag cement was especially helpful in improving mixture workability and reducing stickiness due to the fine particle size of the silica fume.

Both Class C and Class F fly ashes have been used successfully with slag cement to improve strength, durability, and economy, and/or to reduce embodied energy and heat of hydration associated with concrete mixtures. In locations where fly ash is typically used in much of the concrete produced, slag cement can be included to increase the total level of supplementary cementitious materials in a mixture. Fly ash substitution beyond 20 % for a Class F fly ash or 30 % for a Class C fly ash is unusual and can necessitate special testing requirements and admixture use. However, if higher levels of supplementary materials are desired to achieve enhanced strength, permeability reduction, or lower embodied energy and emissions, slag cement can replace an additional percentage of the portland cement. The Iowa Department of Transportation uses ternary mixtures in most of its concrete, including pavements with 35 % slag and 10 % fly ash, 20 % slag and 15 % fly ash, and, in high-performance concrete, 35 % slag and 15 % fly ash [41]. The construction of Houston's Reliant Stadium utilized a high-performance concrete with 30 % slag cement and 20 % fly ash to achieve high-strength (13 000 psi) [48]. Additionally, the use

Fig. 20—Reliant Stadium in Houston, home of the NFL Texans, utilized a high-performance ternary mixture of portland and slag cements and Class C fly ash in the four massive "super columns" that support the retractable roof (inset). The concrete met the requirements of high strength (13 000 psi), low heat (massive placement), and virtually self-consolidating consistency.

of slag cement provided low-heat of hydration and virtually self-consolidating concrete for the four "super columns" that support the retractable roof. The 900 lb/cy mixture typically achieved field strengths of 17 000 psi at 28 days (Fig. 20).

Environmental Benefits

Slag cement, being a by-product of an iron blast furnace, is a recovered industrial material that has low embodied energy and emissions, when compared to portland cement. Using slag cement in concrete has several environmental benefits:

- Use of a recycled material
- Reduced landfill requirements
- Reduced virgin material use
- Reduced energy
- Reduced greenhouse gas emissions

A study comparing the energy to produce a ton of portland cement versus a ton of slag cement showed an 86 % reduction in energy required to produce slag cement [49]. Additionally, the production of slag cement provides a 93 % reduction in virgin materials used (including water) and a 98 % reduction in carbon dioxide emissions. In concrete this is highly significant because, even though portland cement comprises only about 10 to 25 % of the ingredients by weight in concrete, it is responsible for about 90 % of the embodied energy and carbon dioxide emissions contributed from all the ingredients, as well as transportation and concrete plant operations.

The high level of portland cement replacement that is commonly achievable with slag cement enables dramatic reductions in embodied energy, emissions, and virgin material use in concrete. Table 4 shows the percent reductions achievable when 35 and 50 % slag cement are used as a substitute in

TABLE 4—Reductions in Embodied Energy, Carbon Dioxide Emissions, and Virgin Material Use in Concrete Using Slag Cement [49]

Item	3000 psi Ready Mixed Concrete		5000 psi Ready Mixed Concrete		7500 psi Precast Concrete		Concrete Block	
	35 % Slag	50 % Slag	35 % Slag	50 % Slag	35 % Slag	50 % Slag	35 % Slag	50 % Slag
Virgin Material	4.8 %	6.8 %	6.8%	9.8 %	10.3 %	14.6 %	4.3 %	6.3 %
Energy	21.1 %	30.2 %	23.5%	33.7 %	25.7 %	36.5 %	19.9 %	28.6 %
Carbon Dioxide Emissions	30.1 %	42.9 %	31.2%	44.7 %	32.3 %	46.1 %	29.2 %	42.0 %

* Reduction percentages are based on substituting 35 or 50 % slag cement with portland cement in a typical concrete mixture for the specified concrete type. Material, energy, and carbon dioxide values include all operations and transportation required to produce the concrete raw materials, as well as concrete plant operations. Values for all materials are based on average values calculated in the referenced studies.

Fig. 21—Forty-five percent slag cement concrete was used in the structural concrete in The Helena, in Manhattan, to help gain green building certification and a New York State tax incentive for green building construction. Two-day forming cycle was not affected by the use of slag cement, even though much of the construction occurred during the winter.

several different types of concrete (3000 and 5000 psi ready mixed concrete, 7500 psi precast concrete, and concrete block). Virgin material is reduced by up to 15 %, energy by up to 37 %, and carbon dioxide emissions by up to 46 %.

Using recycled materials, and materials with lower embodied energy and emissions is becoming more mainstream with the advent of green building certification systems such as the U.S. Green Building Council's LEED™ rating system. The Helena (Fig. 21) in Manhattan utilized slag cement at a 45 % level in the structural concrete in this 45-story high-rise residential structure to help gain LEED certification, as well as a New York State tax incentive for sustainable construction practices [50].

Conclusions

Slag cement is a widely available supplementary cementing material in most parts of the United States and Canada. Worldwide, it has a long history of accepted use (since the late 19th century). Wide-scale use of slag cement in the United States began in the early 1980s, corresponding with the opening of granulation and grinding facilities at the Sparrows Point, MD blast furnace and the publication of ASTM C 989.

Slag cement substitutes for 20 to 80 % of the portland cement in concrete and provides a wide range of benefits. These benefits result primarily from the increased C-S-H and decreased calcium hydroxide in slag cement concrete, compared with ordinary portland cement concrete. Also, the particle shape, surface density, and smoothness provide benefits in the workability of freshly mixed concrete. Some of the benefits are:

- Freshley mixed concrete–
  - Reduced water demand
  - Improved workability and consolidation
  - Longer working times in warm weather
- Hardened concrete–
  - Higher ultimate strength
  - Lower permeability
  - Greater protection for steel reinforcing against corrosion
  - Mitigation of alkali-silica reaction
  - Mitigation of sulfate attack and delayed ettringite formation
  - Reduction of heat in mass concrete
  - Lighter color
  - Compatibility with other concrete-making materials

Besides these benefits, it should be noted that slag cement concrete is durable in freezing and thawing conditions, is resistant to deicer salt scaling (up to a level of 50 % replacement), and exhibits approximately equivalent shrinkage characteristics to ordinary portland cement concrete.

Placing, finishing, and curing slag cement concrete is similar to plain portland cement concrete, but contractors may experience some differences, including:

- Increased time of set, especially at lower placement temperatures
- Lower strength before about 7–10 days (unless curing temperatures are elevated)
- Slight changes in admixture requirements (but once changed, should remain relatively consistent)
- Differences in bleed water capacity and rate

These differences are normally minor, but may require some adjustments to finishing and curing practices, or even mixture design.

Finally, slag cement contributes to the environmental sustainability of concrete by reducing raw material use, lowering emissions, and decreasing energy.

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PART VI
Specialized Concretes

45

Ready-Mixed Concrete

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Preface

THIS CHAPTER ON READY-MIXED CONCRETE HAS been a part of *ASTM STP 169* since it was first published in 1955. Colin Lobo is the current secretary of ASTM Subcommittee C09.40 on Ready-Mixed Concrete and Richard Gaynor has been a member of the subcommittee since the early 1960s.

This chapter follows the organization of ASTM Specification C 94/C 94M on Ready-Mixed Concrete and provides the authors' perspective of the intent of the specification requirements, developments in technology that are changing the ready-mixed concrete industry and how it operates, and the potential impact on these specifications. This discussion pertains to ASTM C 94/C 94M-04a, the current version of the specification at the time of this printing.

Introduction

ASTM International has two specifications covering ready-mixed concrete: ASTM Specification for Ready-Mixed Concrete (C 94/C 94M) and ASTM Specification for Concrete Made by Volumetric Batching and Continuous Mixing (C 685/C 685M). This paper will cover both specifications, but the emphasis will be on ASTM C 94/C 94M because of its much greater use.

ASTM C 94/C 94M and C 685/C 685M are specifications for concrete, as manufactured and delivered to a purchaser in a freshly mixed and unhardened state. They form the basis for a contract between a manufacturer and a purchaser [1,2].

History of the Industry

The first concrete mixed off-site and delivered to the job may have been furnished in 1913, but it was not a recognizable industry in the United States until the late 1920s when the first revolving drum truck mixers were developed [3]. ASTM C 94/C 94M was first published in 1933. The volumetric batching with continuous mixing specification, ASTM C 685/C 685M, was published in 1971. In 1933, the ready-mixed concrete industry consumed about 5 % of the portland cement produced in the United States. In the early days, the competition was with site-mixed concrete. However, as measured by the amount of cement used within the United States, the industry's use grew from about one-third to two-thirds in the period from 1950 to 1975. In 2003 the industry used approximately 75 % of the total cement consumption [4]. At that time, the estimated U.S. ready-mixed concrete production was 405 million cubic yards (310 million cubic meters) [5].

The 1994 edition of this chapter on ready-mixed concrete [6] indicated a change in trend of the ready-mixed concrete industry whereby acquisitions and consolidation had resulted in larger companies that operated across several states. This trend has continued with larger multinational corporations establishing vertically integrated company structures that include cementitious materials, aggregates, concrete and other building products, and asphalt paving operations. Trends in industry statistics are better quantified with reporting by public companies and information on the Internet. However, there still exists a large segment of the industry, quantified by the number of companies that still operate smaller local family-owned businesses. There also exist a significant number of large privately held companies that produce in the range of 2 to 5 million cubic yards (1.5 to 3.8 million cubic meters). The estimated number of companies has changed from about 5000 in 1978 to 3700 in 1994 to about 2400 in 2003 [7,8]. The historical pattern where 10 % of the companies produced 50 % of the concrete has changed, and now 6 or 7 % of companies produce 50 % of the concrete. The "average" ready-mixed concrete company produces about 700 000 cubic yards (535 000 cubic meters), and operates about 12 plants and 125 mixer trucks. The average quantity of concrete produced per plant is in the range of 60 000 cubic yards (46 000 cubic meters) annually in the United States [8]. These average numbers are quite skewed due to a wide distribution of company size and structure. At the present time, small companies operating less than 15 trucks account for more than half the number of companies and produce less than 10 % of the concrete.

The changing demographics of companies operating in the industry also fosters greater technical sophistication and greater use of performance specifications. Increasingly, the ready-mixed concrete producer is the concrete technology expert. He is now relied on as an expert on concrete material technology and innovative products by highway departments and other specifiers. His involvement in standards development, research, and opportunities for technology transfer through the American Concrete Institute, ASTM International, Transportation Research Board, and other technical organizations have helped greatly.

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Specifications

ASTM C 94/C 94M and C 685/C 685M are specifications for ready-mixed concrete. As such, the specifications address the separate and joint responsibilities of the various parties in a typical job—the owner, specifying agency, contractor, concrete producer, and testing agency.

The ASTM C 94/C 94M and C 685/C 685M specifications are used in a number of rather different situations.

In major public and private construction, they are incorporated by reference in the job specifications. Here, the basic contract is between an owner and his representative (the specifier) and a contractor. The concrete producer is a material supplier who agrees in his contract with the job contractor to comply with ASTM C 94/C 94M or C 685/C 685M.

At the other end of the scale, in small jobs, these ASTM specifications form the basis for an agreement to furnish material for incidental construction, perhaps to a homeowner.

A third and important use is the protection of the public interest by incorporation by reference in public building codes. In this instance, the requirements, which are binding on the owner, are then passed down to the contractor and finally to the concrete producer through a purchase order from the contractor. It is, however, the purchaser's responsibility to invoke the pertinent code requirements in the construction specification and the purchase order for concrete.

Inevitably, circumstances often dictate that job specification requirements differ from those in ASTM C 94/C 94M and C 685/C 685M. Therefore, both specifications include the statement that if there are differences, the purchaser's specifications shall govern.

Since the ready-mixed concrete producer functions as a supplier of materials and often has no binding contract with the owner, there is a need for close cooperation between the contractor and the concrete producer. In 1965, the Associated General Contractors and the National Ready-Mixed Concrete Association adopted a "Joint Statement of Responsibilities" that has been found useful over the years and has been reviewed by both associations periodically and republished without change [9]. The statement addresses the separate and joint responsibilities of each party and is helpful in defining the traditional roles of each. More recently a partnership between the American Society for Concrete Contractors and the National Ready-Mixed Concrete Association have produced checklists for concrete preconstruction conferences and ordering concrete that provide guidelines to establish responsibilities and identify responsible individuals to address specific situations and make decisions during the course of the job [10].

Basis of Purchase

An early section in both ASTM C 94/C 94M and C 685/C 685M defines the cubic yard (or cubic meter) of concrete as the basis of purchases and then describes the method of testing and calculation of the volume of fresh concrete. The volume of concrete is defined as the total weight of the batch divided by the concrete density (unit weight) as determined by ASTM Test Method for Density (Unit Weight), Yield, and Air Content (Gravimetric) of Concrete (C 138). The unit weight used is the average of the results of three tests made on separate samples from different loads. Although the density method, ASTM C 138, permits tests in 0.2 ft^3 (6 L) air meter bases, ASTM C 94/C 94M requires tests in standard $^1/_2$ ft^3 (14 L) unit weight buckets for improved accuracy. There has been some consideration of deferring to the smaller density measure sizes permitted in C 138 based on coarse aggregate size, because in real practice the larger density measures are cumbersome to handle and are seldom seen on the job site. This might be a revision in future versions of C 94/C 94M and C 685/C 685M.

Yield or Volume In-Place

A note explains that the volume of concrete delivered may be less than expected due to waste, spillage, over-excavation, spreading of forms, settlement in forms, or some loss of air. Further, the volume of hardened concrete is about 2 % less than its volume in the plastic state. Because the quantity of concrete actually used on a job will be greater than that calculated from plan dimensions, yield tests must be made early in the job and appropriate adjustments made in batch proportions if deficiencies are found.

When yield is confirmed by the density test, another possible source of error is in the weights of materials batched. Scale accuracy should be checked at three- to six-month intervals and certified calibrations should be conducted at least annually. If these checks are made, the concrete discharged from the truck should be within 1 or 2 % of that determined by the standard yield test. Due to different tolerances on weighing materials, air content, and other factors that affect the yield determination, a tolerance on the yield from the quantity ordered is difficult to establish. Seldom do the contractors' initial estimates agree that closely with the amount delivered by the concrete producer. The practice of ordering full loads by many contractors practically ensures that 2 to 3 % more will be ordered than is needed. Estimates should be revised towards the end of every pour and communicated to the concrete producer to avoid returning unused concrete or ordering short loads. A 6-in. (150-mm) slab that is 3/8 in. (9 mm) too thick will require 6 % more concrete, and deflection of bar joist construction, which is not shored, can produce slabs that average 1 in. (25 mm) too thick [11,12].

Under ASTM C 685/C 685M, for concrete batched volumetrically using mobile mixers, a check on the yield is made by weighing the concrete discharged in a given number of revolutions of the cement feeder and then determining the density of that concrete by ASTM C 138. The volume of concrete discharged is then the weight of concrete discharged per revolution times the number of revolutions divided by the density.

Truck Mixer Hold Back

Generally, it is realized that when concrete is batched into a clean, washed-out truck mixer, it will take 1 to 2 % of a capacity batch to coat the drum and blades. This material is principally mortar with a negligible amount of coarse aggregate. Since mixers are washed out and wash water and solids discharged only at the end of the day, this does not significantly affect the volume of concrete delivered if the truck delivers four or five batches each day.

However, when a 1 yd^3 (0.76 m^3) batch is mixed in a clean 10 yd^3 (7.6 m^3) capacity truck mixer, the effect on concrete proportions can be dramatic. Compared to the batched proportions, if the amount of mortar retained is equal to 2 % of the weight of a capacity batch, the cement content of the concrete discharged will drop from 600 to 470 lb/yd^3 (356 to 280 kg/m^3), and the sand as a percentage of the total aggregate will decrease from 36 to 26 %. The batch will be harsh to handle and will have low cement content and low strength.

The solution is to increase cement, sand, and water weights up to about 40 % in such small truck-mixed batches, referred to as short loads. If a clean tilting central mixer is used, the effect on proportions is much more dramatic since the gross volume of the drum is about 25 % larger than a truck mixer, the surface to be coated is much greater, and the hold back perhaps 50 % greater.

Ordering Information

Both ASTM specifications include the fundamental elements of prescription and performance specifications and list the basic information needed by both parties. However, the purchaser often includes additional requirements. Sometimes this is an attempt to ensure quality, perhaps because of past bad experiences or in an effort to provide characteristics that were not readily available in standard mixes. The growing number of types of chemical admixtures and supplementary cementitious materials permits the concrete producer to produce concrete with properties quite different from the "standard mixes" in common use a few years ago. Some of the newer applications are flowing concrete, self-consolidating concrete, anti-wash-out concrete, low-permeability corrosion-resistant concrete, concrete with reduced shrinkage characteristics, fiber-reinforced concrete, controlled low-strength materials, pervious concrete, and high-performance concrete. Concrete with three or even five admixtures is becoming more common. Control of such mixtures can be a significant challenge for concrete producers, especially when a variety of concrete types are batched during a particular period.

The Ordering Information section requires the purchaser to specify the size of coarse aggregate, slump, air content, unit weight of structural lightweight concrete, and one of three options: A, B, or C. The options are as follows:

Option A is the performance format in which the purchaser specifies strength and the producer selects proportions of ingredients.

Option B is the prescription format in which the purchaser specifies cement content, maximum water content, and admixtures.

Option C is a mixed format in which the purchaser specifies minimum cement content, required strength, and admixtures, if required.

At least at this point, the performance format of ordering concrete in C 94/C 94M is based on strength. A broader performance-based method that addresses other characteristics to include durability is most likely covered in job contract documents, which would apply. The producer should ensure that the basic information required for the mixture is provided with the order, although C 94/C 94M indicates that it is the purchaser's responsibility. The situations can vary but in many cases the producer may have more expertise or knowledge than the purchaser on acceptable local practice [13].

Water-Cementitious Materials Ratio in Specifications

The A, B, and C options do not include a requirement for a maximum water-cementitious materials (w/cm) ratio even though Option B does include both cement and water content. The principal reason that it is not included is the difficulty of actually measuring the w/cm ratio in practice. From the user's perspective, a w/cm ratio is needed to ensure durability, that is, resistance to freezing and thawing, deicer salts and associated scaling, sulfate exposure, or intrusion of chlorides in reinforced concrete. The maximum w/cm ratios usually cited are those required in the "Building Code Requirements for Structural Concrete" (ACI 318) [14] or "Specifications for Structural Concrete" (ACI 301) [15], which range from 0.50 to 0.40. In many high-performance concretes, it is possible to produce concretes with w/cm ratios as low as 0.30 or lower and values as low as 0.35 are being specified in parking structures for resistance to intrusion of chlorides.

The concrete producer has difficulty conforming to a maximum w/cm ratio since the amount of water necessary to produce a given slump will vary with local ingredient materials and delivery time, which depends on delays related to traffic or on the job site. Additionally, the moisture content of the aggregates will vary from batch to batch making accurate measurement and/or adjustments difficult. Another difficulty is that the procedures used by specifiers to determine compliance with maximum w/cm ratio specifications are rarely defined; and if every batch is to be checked, the production and placement schedule will be disrupted.

In practice, specifiers use a number of systems of enforcing maximum w/cm ratio specifications. One of the simplest is to require submission of laboratory trial batch or previous field mixture proportions with accompanying test data demonstrating compliance with the specified maximum w/cm ratio and then relying on routine strength tests to determine compliance. This system breaks down if the specified strength is not reasonably consistent with the strength obtained at the desired w/cm ratio. Increasingly, large agencies are requiring 2 to 4 yd^3 (1.5 to 3 m^3) trial batches instead of laboratory trial batches to improve the accuracy of the mix approval process.

For all of the above reasons, if the w/cm ratio is to be strictly enforced in the field, then compliance with a specified maximum w/cm ratio for field acceptance is inappropriate without an understood statistical tolerance, not unlike that used in ACI 318 or in ASTM C 94/C 94M for strength.

On well-controlled jobs, the standard deviation of the w/cm ratio ranges from 0.02 to 0.03. A concrete producer would have to furnish concrete with an average w/cm ratio of as low as 0.35 or even lower to avoid batches that have calculated w/cm ratios greater than 0.40!

For some time AASHTO has had a test procedure, AASHTO T 318, for determining the water content of concrete by rapidly drying a small sample of concrete in a microwave oven [16]. Currently, ASTM subcommittee on Fresh Concrete Tests, C09.60, has started work on standardizing a similar procedure. The idea is that the measured water content in the test can be used with the recorded quantity of cementitious material batched to calculate a w/cm ratio. Several State DOTs have tried the method and although they have thought it was useful it has not been widely used in their specifications. The Port Authority of New York and New Jersey have used it in specifications that include payment adjustments in airport construction and feel that it has helped greatly to improve construction quality [17].

The writers' opinions on w/cm ratio specifications is that they should not be used and do not, as a single mixture parameter, provide better assurance of "durability" than other much more reliable, more accurately measured performance characteristics. At a minimum, the strength level specified for the concrete should be consistent with the specified w/cm ratio, so that the purchaser is at least assured that concrete being delivered is of the desired quality. This issue is one that has been under considerable discussion for several years [18,19].

Approval of Mixtures

Under all three of the options of ordering concrete in ASTM C 94/C 94M and C 685/C 685M, if the purchaser requests it, the manufacturer is required to furnish proportions of ingredients that will be used. This information is related to that required to be furnished on the delivery ticket.

The system of requiring submittal and "approval" of concrete proportions is firmly embedded in specifications and codes in the United States. This process started prior to about 1940 when virtually all concrete was furnished as prescription or cement content mixes–not strength or for other performance-based requirements. C 94/C 94M does not address mixture submittals. These are addressed in ACI 318 for mixtures and in ACI 301 for mixtures and construction means and methods.

Today, most mixtures have specified design strengths. Once mixture proportions are identified in a submittal, it is difficult to reduce cement content without generating opposition or suspicion from specifiers or contractors. The net result is that there is little incentive for the concrete producer to implement a quality control program that is designed to control strength with a high degree of uniformity at the target level that complies with the specified strength, and the concrete is furnished with higher cement content and much higher strength than is actually needed.

There has been some question as to the usefulness of a submittal of mixture proportions in the case of a strength-based specification. The design professional or specifier rarely "approves" the submittal, and the strength acceptance criteria of strength test results during the course of the job govern in any event. Ideally, for a performance-based specification, the submittal should furnish mixture pre-qualification test data indicating compliance with specification requirements. The composition of the concrete mixture that describes the details of the ingredients and proportions are really irrelevant. Selected job site acceptance tests can then be used to demonstrate compliance with the job specification and the pertinent submittal.

Another aspect of adhering to submitted mixture proportions is that adjustments to concrete mixtures are necessary on a real time basis to accommodate variations in ingredient materials and seasonal factors. Oftentimes a required resubmittal can delay construction schedules. If the producer was free to vary proportions to produce the required performance, the incentive would exist for him to invest in sophisticated quality control processes that facilitate the production, batching, delivery, and testing of consistent and uniform concrete batches. Adoption of performance-based requirements and elimination of prescriptive mixture proportions are necessary before concrete suppliers will have the incentive to adopt more formal and advanced quality control systems.

There is, in the American Concrete Institute (ACI) Building Code ACI 318, a provision that permits a producer to recalculate the required over-design when he has accumulated 15 tests and to make adjustments in proportions to conform to a lower required average strength level compared to what he would furnish if he did not have a strength test record. This is at least a beginning for performance specifications.

Materials

This section includes references to the commonly used ASTM specifications for cement, aggregates, admixtures, and water. A number of materials such as fibers, expansive cement, corrosion inhibitors, and other ingredients are not included, either because there is no ASTM specification or because there is a feeling that the use of the material requires different batching, mixing, delivering, or testing procedures than those for "normal" concretes.

Water Quality

In 2004 a specification for mixing water for use in concrete, ASTM C 1602/C 1602M, was approved and the requirements for water in C 94/C 94M make reference to this specification. ASTM C 1602/C 1602M defines water sources as *potable, non-potable,* and *water from concrete production operations*. It recognizes that one or more of these sources may be combined to make up the total mixing water used in concrete. Potable water is permitted to be used without qualification testing. Qualification requirements and associated testing frequencies are established for other sources of water. The qualification of water sources requires testing for effects of the water source on strength and setting time when compared to control concrete made with potable water. The qualification requirements indicate that the strength should not be less than 90 % of the control and setting time should not be retarded by more than 90 min or accelerated by more than 60 min. The 90 % limit for strength is intended to allow equivalence to acceptable water with a statistical allowance for testing variability. The qualification criteria apply to the total mixing water with the intent that when a concrete producer qualifies a certain critical composition of water, those compositions of lesser impact on concrete properties can be used without qualification testing. For instance, if a producer conducts qualification tests for a combination of wash water and potable water at a solids content of 50 000 ppm, mixing water with lower concentrations of solids are thereby qualified for use.

Considerable pressure from evolving environmental regulations is forcing the ready-mixed concrete industry to reuse more of its process water. These regulations control, and in many cases prohibit, the disposal of wash water, yard runoff, and returned concrete. As a result producers are increasingly reusing wash water as mixing water and are considering incorporation of returned concrete or the partially hydrated cement from returned concrete in freshly mixed concrete.

Mixer Wash Water

ASTM C 1602/C 1602M permits the use of water from mixer wash-out operations as mixing water in concrete if the water meets the qualification tests for strength and set time mentioned above. The qualification criteria apply to the total mixing water. The specification requires the density of wash water to be measured daily and establishes testing frequency for strength and setting time based on the density of the total water that the producer intends to use. This recognizes that more monitoring and control is needed when wash water with a higher solids content is used.

Optional limits can be invoked by the purchaser on the amount of chlorides, sulfates, alkalies, and total solids in the water. The specification provides for a testing frequency for these tests. As currently written, ASTM C 1602/C 1602M has an optional limit of 50 000 ppm of solids in total mixing water. Typically, this permits up to about 15 lb/yd^3 (9 kg/m^3) of dry solids.

It should be noted that the amount of air-entraining admixture required to produce the required air content may increase, and even double, if these admixtures are added to high pH wash water before the water is batched into the mixer. The air-entraining admixtures should be added either with the sand or with an increment of clean mixing water. Although it does not appear that similar problems are experienced when other commonly used accelerators, retarders, or water reducers are batched into or at the same time as wash water, this possibility should not be ignored with the increasingly sophisticated admixture systems that are being developed and used. Research and industry experience with the use of wash water at relatively low solids content has been that effects on product quality are not significant or important [20–23]. These studies represent the use of wash water that has been clarified through a settling pond system.

Increasingly the industry is moving towards zero-discharge production facilities. Returned concrete and mixer wash water is processed through concrete recycling or reclaiming units that wash out and separate the aggregates. The cement and sand fines are maintained in a slurry suspension in agitation tanks. This slurry is then used as a part or whole of the mixing water in concrete. The percentage used will depend on the density of the wash water slurry, which is related to the solids content. This operational process eliminates steps of removal of fines from process or storm water settling basins, drying, and disposal. Water slurries used at higher solids content, composed primarily of partially hydrated cement, cause an increase in the mixing water requirement for target slump and can result in lower strength. Hydrated cement also causes acceleration of the setting time of concrete [24–27]. Hydration stabilizing admixtures (HSA), which suspend the hydration of cement, have been used very effectively to overcome some of the negative effects of using recycled water at higher solids content. The use of these admixtures facilitates the use of a larger quantity of wash water slurries with higher solids content without adverse affects on concrete properties. This allows the producer to maintain a production mass balance whereby the generated wash water is reused. Complete recycling systems include accurate water density gages, separate tanks that feed admixture-treated water at constant solids content, and computerized systems that automate the process [25,28]. Effective admixture dosages can be established by determining the loss on ignition (LOI) of the dried solids from the water slurry. A low LOI value represents a low degree of hydration of cement indicating that the HSA is functioning for the storage duration of the slurry [25]. A simpler qualitative quality control tool is to monitor the rate of settlement of solids in a column of water. Unhydrated cement will settle at a faster rate indicating the effectiveness of the admixture dosage used [28].

ASTM C 1603/C 1603M establishes procedures for measuring the density and solids content of wash water slurries reclaimed from returned concrete. It establishes methods of estimating the solids content of water from the measured density, a property that is easier to measure in production situations. ASTM C 1603/C 1603M also provides guidance on blending two sources of water to stay within a predetermined or specified target solids content or density.

Returned Concrete

ASTM C 94/C 94M is silent on the reuse of returned concrete but there are efforts currently to address it in the specification. In this scenario the concrete remaining in a truck mixer is tempered with water at the job-site and fresh materials are batched on top of it at the plant. Sufficient caution and control should be exercised to ensure that the resulting concrete mixture meets the purchaser's requirements. Factors that affect the properties of the resulting concrete include the quantity and age of the returned concrete used, cement factor, and temperature history. Generally, the resulting concrete will be of lower strength due to a higher water content and will set faster. These effects are exacerbated when a larger quantity of returned concrete is used and when the returned concrete has reached a higher degree of hydration [29,30]. Hydration stabilizing admixtures (HSA) were originally developed to treat and reuse returned concrete. Returned concrete is stabilized for reuse on the same day or even the next day. Laboratory studies have demonstrated that returned concrete can be successfully treated with HSA without adversely affecting concrete properties [31–33]. While several concrete producers have installed systems that allow for the controlled and quantified use of HSA-treated returned concrete, the operational challenges of tracking a variety of factors have limited its widespread use.

Slump and Air Content

The section on slump in ASTM C 94/C 94M contains tolerances in two different formats: a "maximum" or "not to exceed" value, and a target value. The tolerance varies by level of slump. It also establishes a 30-min period after arrival on the job during which the producer is responsible for the slump. The job-site addition of high-range water reducers (HRWR) to produce flowing concrete has created a number of field control problems when the slump of the flowing concrete is subject to strict slump control and testing. If job-site adjustment is preferred, a more practical procedure is to specify the slump of the concrete before addition of the high-range water reducer and to accept the fact that the slump of the flowing concrete will vary. New admixture chemistries for high-range water reducers have reduced the problems associated with slump loss with time and improved the linear relationship of dosage to concrete slump level. Slump retention has also improved with these newer generation HRWR admixtures. It is more common for high-range water reducers to be added at the plant. Requiring a slump level prior to adding the admixture at the plant is not practical. With increased use of self-consolidating concrete, alternative measures of concrete consistency, such as slump flow and other empirical measures to quantify the rheology of concrete will be necessary. Such methods are in the process of being standardized.

The section on air content contains a table of recommended air contents taken from ACI Committee 211 [34]. The delivery tolerance is ±1.5 % of the specified air content. When a preliminary sample is taken prior to discharge and has a low air content, C 94/C 94M allows the addition of air entraining admixture followed by 30 revolutions of mixing.

There have been a number of problems that have surfaced recently in the control and measurement of air content. Some years ago, Meininger [35] and Helmuth [36] described tests indicating that some fly ashes containing carbon adsorb air-entraining agent. This caused a higher dosage requirement of the admixture for the required air content and a reduction of concrete air content during delivery. Ozyildirim [37] and Hover [38] have made studies to determine if the pressure meter accurately measures the air content of plastic concrete and whether the pressure method provides a reasonable estimate of the amount to be expected in hardened concrete. These issues are the subject of other chapters in this book. However, the answer generally appears to be that ASTM Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231) does provide the desired measurement and that the air content of hardened concrete is often either lower or higher for a multitude of reasons. Another aspect is that newer technologies for air-entraining admixtures create more stable air void systems but need additional mixing energy to develop the required air void system and air content. It has been reported that the air void systems generated from these air entraining admixtures are of smaller size and could possibly function for freezing and thawing resistance at lower total air contents. The primary intent with entrained air is to obtain a proper air void system in concrete characterized by a proper spacing factor and bubble size, indicated by the specific surface. These parameters have been typically measured in hardened concrete specimens using ASTM C 457. More recently, there have been attempts to standardize the Air Void Analyzer that measures the distribution of the size of air voids in plastic concrete [39,40].

Another issue is the observation that much of the initial air can be lost during pumping, particularly with the newer long boom pumps with 5-in. (125-mm) lines. The authors believe that this occurs when a section of the boom is essentially vertical and concrete slides down from its weight and develops a vacuum in the pump line. The air bubbles then expand and fail to reform when the concrete drops out the end of the pump line or impacts an elbow in the boom [41]. The solution has been to insert some resistance in the line. This can be as simple as inserting a loop in the flexible hose or laying a length of hose on the deck [42]. Under normal conditions, the loss of air in pumping should not exceed 1 or 1.5 %. Air can also be lost when concrete is dropped or discharged from a belt conveyor. Hover has demonstrated that even though air content is reduced through a variety of placement methods that cause concrete to fall through a certain distance, the volume of air lost is in the larger size bubbles and the remaining air content is adequate to provide resistance to cycles of freezing and thawing [43].

C 94/C 94M establishes that the point of sampling for acceptance testing is at the discharge point from the transportation unit. Some specifications require testing the concrete at the point of placement in the job, which makes the concrete producer, the contractor, and the pumping contractor responsible for the final results for acceptance of the tests for air content and strength. The concrete producer prefers that tests be made on discharge from the truck. If he agrees to provide 8 % air because 3 % will be lost during placing, it should be recognized that strength tests will be reduced 15 to 20 % if cylinders are made from samples obtained at the truck discharge. Likewise, if the test samples are obtained after pumping with the pump located near the truck with the boom near vertical, the air content will be reduced. However, if this concrete is placed at a higher level with a less critical boom configuration, the air will not be lost and the strength in-place may be significantly lower. The problem is exacerbated if the pump location is such that placement occurs both close to the pump with a critical boom configuration and also a long distance away where the boom will be in a horizontal position. The contractor, pumper, specifier, and producer need to work closely together to determine if loss of air is likely to be a problem and what procedures will be used. Naturally these are issues that must be resolved in a pre-job conference or earlier.

Another problem is the development of surface delaminations and blisters when air-entrained concrete is steel troweled. This occurs in industrial floors with mechanical and vibrating screeds that are mechanically troweled. Although a number of factors are involved, the solution is to avoid the use of entrained air in industrial floors or, at least, to keep the air content below 3 or 4 %.

Batching and Measuring Materials by ASTM C 94/C 94M

ASTM C 94/C 94M requirements for measuring materials recognize both individual scales and hoppers for weighing a single material and cumulative scales and an associated hopper for weighing more than one material. This system, and the terminology that is used, developed from the use of dial scales where the material weights were accumulated as materials were weighed. With the increasing use of computerized batching equipment, it has now become possible to have the computer do the subtraction and print the weights of the individual materials in a cumulative batcher, including a recognition of the zero reading or tare. A note is needed in ASTM C 94/C 94M to explain the fact that cumulative tolerances apply to cumulative batchers irrespective of the format used to report the batch weights. This means that the tolerance of any individual weighing is based on a tolerance percentage of the intended cumulative weight.

Cementitious Materials

Cement, slag, fly ash, and other pozzolans may be weighed cumulatively on a single scale provided cement is weighed first. The weighing tolerance is ±1 % of the required cumulative weight. For small batches where the cumulative weight is less than 30 % of scale capacity, the tolerance is from −0 to +4 %. The primary reason for weighing cement first is related to the flow characteristics of fly ash that could cause it to be overweighed and result in a lower batched quantity of cement. This requirement tends to ensure the correct cement weight and, if anything, an excess of mineral admixtures. Cumulative weighing of cement and supplementary cementitious materials (also referred to as mineral admixtures) also has the advantage that the batcher tends to “rathole” when it discharges into the mixer and the cementitious materials blend as they are loaded. There have been a few instances when individual separate batchers were used for cement and fly ash and the fly ash wound up in one part of the batch, separate from most of the cement. When separate batchers are used, which is rare, the batcher discharge must blend the two materials.

Batching silica fume is not addressed in the current version of C 94/C 94M; however, this is currently being considered since it is a small amount and if it is weighed cumulatively as the last ingredient it is possible to meet current batching tolerances by batching 60 % of the desired amount if 5 % by weight of cementitious materials is desired. With the load cells and automation now available it is possible to treat the silica fume as a separate material and batch it with an accuracy of 0.3 % of scale capacity. Normally this would be within ±10 % for 5 % of silica fume and correspondingly less for 10 or 15 % of silica fume by weight of cementitious materials. Another alternative might be to weigh the silica fume to 100.3 % of the amount desired with a tolerance of ±0.3 % of the capacity of the scale. This will slow down the batching process. Producers still using dial scales and 5- or 10-year-old automation are likely to use silica fume in preweighed bags. Likewise even those with the newer systems should not attempt to weigh silica fume for batches less than about half the batching capacity of the cumulative system. Naturally if the plant uses a separate individual silica fume weigh batcher the tolerance would be ±2 % of the desired amount. This would speed up batching and provide some flexibility in controlling the batching process when silica fume is used.

Although it is not addressed in ASTM C 94/C 94M, many concrete plants contain several silos for cementitious materials, and there is always the possibility that material can be placed in the wrong silo. Producers generally use different colored fill pipes, different sized connections, signs, and distinctively colored bills of lading to distinguish between materials. In some cases, fill pipes can be at different locations in the plant. Some producers use keys and locks on fly ash pipes and control access to the keys. Because fly ash tends to flow freely through cracks, some specifications [44,45] do not permit common walls between multi-compartment cement and fly ash silos. The space between these double-walled bins needs to be free-draining with access provided for inspection.

Aggregates

Aggregates can be weighed either in cumulative or individual weigh batchers. The basic batching tolerance in cumulative batches is 1 % of the cumulative weight and 2 % of the required amount in individual batchers. For cumulative weights less than 30 % of scale capacity, the tolerance is ±0.3 % of scale capacity or ±3 % of the required cumulative weight, whichever is less. This means that at less than 10 % of scale capacity, the 3 % tolerance will govern.

Mixing Water

ASTM C 94/C 94M defines mixing water as any water added to the batch plus surface moisture on aggregates, ice, or liquid admixtures. The tolerance on batching added water is ±1 % of the total water content, not a percentage of that being batched. The tolerance on the total water is ±3 % of the total water content. This does mean that if a water meter is accurate to about 10 lb (4.5 kg) or 1 gal (3.78 L), and the nominal total water content is 280 lb/yd^3 (166 kg/m^3), then the minimum batch size that can be batched is about 3 yd^3 (2.3 m^3). The NRMCA Checklist [46] establishes a tolerance of ±1.5 % on added water and is considered more realistic. When water is weighed, the present ASTM C 94/C 94M tolerances are more easily achieved, but the ultimate importance to concrete quality is questionable.

The total mixing water consists of that measured in the plant plus that from free moisture on aggregates, or an adjustment for that absorbed by less than SSD aggregate; that from wash water measured through the batch plant; and that from certain water-based admixtures that are used in significant quantities, such as high dosages of high range water reducing admixtures, corrosion inhibitors, or shrinkage reducing admixtures. Other sources of mixing water include that which is in the mixer during a job site wash out, which is generally quantifiable through the measuring device on the truck water tank (site gage or water meter), and some minimal quantities that could enter the mixer at the wash-down rack before the vehicle departs the plant. Additionally, water may be added from the truck water tank on arrival at the job site that is within the limit permitted by the specification or that which controls the strength of the concrete. Often the delivery ticket will indicate the maximum additional water permissible. The truck driver is normally advised by the company to record the amount of job-site-added water on the delivery ticket and obtain the signature of the person requesting this addition.

Mixers need to be cleaned at the job site before returning to the plant. This will require about 40 gal of water from the truck water tank to wash down the truck chute, interior of the drum, and fins. When facilities are not provided at the job site to discharge this water, it is carried back to the plant and either discharged or used with the next batch with the appropriate corrections to the plant batch water. The accuracy of the quantity in the mixer truck prior to batching is possibly questionable as this is generally verbally communicated by the driver to the batch man.

Chemical Admixtures

Chemical admixtures are rarely batched by weight, except when they are added in prepackaged fixed amounts. Volumetric dispenser systems are quite sophisticated and are integrated into plant automation. Admixtures are required to be batched to within ±3 % of the desired amount or plus or minus the amount or dosage required per 100 lb (50 kg) of cement, whichever is greater.

It should be noted that on small batches of lean concrete the admixture batching accuracy may be as large as ±25 % of the amount batched. Even in a 10-yd^3 (7.6-m^3) capacity batch, if the dosage rate is 1/2 oz/100 lb (0.3 mL/kg) and the dispenser is accurate to ±1 oz (30 mL), the overall batching accuracy will be ±5 %. However, variations of this size would not change the air content, setting time, or concrete strength. In a 2-yd^3 (1.5-m^3) load, the effects will be significant.

Batching Plant

The requirements of ASTM C 94/C 94M for the batching plant are relatively basic and straightforward. Scales are required to be accurate to ±0.2 % of the capacity of the scale when tested with standard weights. C 94/C 94M requires testing the scale at its quarter points. The scale accuracy requirements of the NRMCA Check List [46] are the same as in C 94/C 94M, and those of the Concrete Plant Manufacturer's Bureau (CPMB) [45] for new equipment are ±0.1 % of capacity. The scale accuracy and calibration requirements are similar to *NIST Handbook 44* [47] with some minor differences that were clarified by a revision to C 94/C 94M in 1992.

Scales in concrete plants generally consist of a container supported at the four corners and transmit reduced loads through a system of levers to a load cell.

Calibrated beam or springless dial-indicating devices are now beginning to disappear. Use of load cells to support the batchers has the potential to simplify a scale by eliminating much of the lever system and dial scale that has typically been used. Earlier load cells were not widely accepted since the dial scale provides a mechanical backup in case of a failure of the electronic load cell system in the middle of a placement.

ASTM C 94/C 94M does not define or require different types of batching controls. The CPMB Standards [45] provide a consistent terminology for plant control systems.

A manual control is one that is operated manually and is dependent on the operator's visual observation of the scale or meter. A semiautomatic control is one that, when started, stops automatically when the required weight has been reached. A semiautomatic interlocked control is similar but contains provisions to prevent discharge of the device until the material is within tolerances.

An automated control starts and stops automatically and includes interlocks to:

(a) prevent charging until the scale returns to zero,
(b) prevent charging if the discharge gate is open,
(c) prevent discharging if the charging gate is open, and
(d) prevent discharge until the material is within tolerances.

These definitions refer to the capability of a control as designed but overrides are in place to allow for manual control of batching.

A batching system consists of the required combination of individual batchers. The CPMB Standards define the following types:

1. Manual—a combination of manual batchers except that water or admixture batchers may be semiautomatic or automatic.
2. Partially automatic—includes at least one automatic or semiautomatic batcher. Interlocks are optional.
3. Semiautomatic batching—a system of semiautomatic interlocked or automatic batchers and volumetric devices in which the interlocks, other than those required for individual batchers, are optional.
4. Automatic batching—requires a combination of automatic devices:
   (a) that must start with a single starting mechanism, except water or admixture not batched at the same time, and may have separate starting devices;
   (b) where each batcher must return to zero within tolerance and reset to start; and
   (c) where discharge of any ingredient may not start until individual batchers have returned to zero and all weighed ingredients have been batched within tolerance.

The concrete producer is constantly trying to reduce the time required for batching and moving truck mixers through the plant. Several innovations in batching efficiency and plant configurations have been developed to facilitate this. There are batching systems that do not exactly follow the definition of automatic systems above by which the batching of the next batch is started before the previous batch is completely charged into a mixer. This sequence allows a higher number of trucks to be moved through the plant and increases the production rating of the plant.

The development of computerized batching systems and control panels has made great strides, and the cost of such systems continues to decline. These have greatly simplified the process of recording batch weights and printing delivery tickets.

Recorders

The CPMB Standards define both digital and graphical recorders. Graphical recorders were once popular for large jobs where one mix design was produced for extended periods. This made it easy to identify mistakes and malfunctions. More recently, digital recorders have become the norm. They print delivery tickets, provide information for billing and inventory control, and provide a record for the producer to determine what the scale did for every batch shipped. The CPMB Standards define three types: “Digital Recorders,” “Digital Batch Documentation Recorders,” and “Digital Concrete Certification Recorders.” All require that if target weights, simulated weights, or other than actual batch weights are recorded, they must be clearly identified. Digital recorders must reproduce the scale reading within 0.1 % of scale capacity or within one increment on volumetric batching devices. The differences between the types are the degree to which they record and print tickets, identify the material batched, give an indication of whether all materials are within batching tolerances, sand moisture content, and whether they record and print basic information such as plant, truck, destination, and space for the driver and purchaser to sign.

Mixing Concrete

There are three types of mixing operations defined in ASTM C 94/C 94M:

a. Central mixing where concrete is mixed in a central plant mixer and delivered to the job in a revolving drum truck, an agitator, or nonagitating unit.
b. Shrink mixing where concrete materials are blended in a central plant mixer with the mixing completed in a revolving drum truck mixer.
c. Truck mixing where ingredients are loaded into a revolving drum truck mixer for mixing and delivery.

The Standards of Plant Mixer Manufacturers Division (PMMD) of the Concrete Plant Manufacturers Bureau [45] define four principal types of concrete plant mixers:

1. Tilting mixers—revolving drum mixers that discharge by tilting. This is the type used in most ready-mixed concrete plants. Standard sizes range from 2 to 15 yd^3 (1.5 to 11.5 m^3). The rated capacity ranges from 30 to 50 % of gross drum volume.
2. Vertical shaft mixers—these mixers have an annular mixing compartment with rotating blades or paddles. The mixing compartment or pan may rotate or not. Generally, they discharge through a door or hatch in the bottom of the “pan.” They were popular several years ago because of their low overall height and rapid mixing, but because of rapid wear are little used today.
3. Non-tilting drum mixers—these are revolving drum horizontal axis non-tilting mixers. These are similar to a truck mixer in operation. There has been some renewed interest in these mixers recently with demonstrated shorter mixing cycles, reduced wear, and less noise.
4. Horizontal shaft mixer—these have a horizontal cylindrical mixing compartment with blades or paddles rotating about the horizontal axis. A number of new designs have been introduced recently although the general concept is not new.

In addition to the recognized PMMD types, there has been considerable interest in several other high-energy mixing designs that are reported to provide more efficient mixing at higher energy levels, presumably shear rates that result in more efficient dispersion of cement and produce significant cement savings [48]. One of the most popular is a horizontal shaft planetary mixer that is used in precast concrete plants. Another type is a slurry mixer that mixes the cementitious materials and water prior to charging this mixed slurry into a truck mixer. It is generally an addition to a truck mixing operation.

Central Mixing

The choice between central or truck mixing depends on a large number of factors. The technical advantage of central mixing is that it provides centralized control of the mixing process and requires a less-skilled truck mixer operator. However, the CPMB study of economic factors [49,50] shows that the decision will depend on a number of other factors, including the market area, market volume, blade life in trucks, and truck utilization. The percentage of the ready-mixed concrete produced by central mixing is in the range of 20 to 25 % of total plants. This contrasts with Europe and Japan where close to 100 % of ready-mixed concrete is produced in central mixing plants.

The principal advantages of truck mixing are lower capital investment, lower plant heights, lower electrical costs, and somewhat greater flexibility when long deliveries are required in rural areas. With special loading sequences in truck mixing plants designed to keep cement essentially dry until the concrete is mixed at the job site, loss of slump and use of retempering water can be avoided [51,52].

In the mid-1960s, the Federal Highway Administration (FHWA) [52,53] conducted mixing efficiency tests of tilting drum central mixers. Generally, they demonstrated that 45 to 90 s mixing times were feasible if care was taken to blend or ribbon-load all ingredients as they entered the mixer. The importance of the loading sequences is likely much less if, as in most ready-mixed concrete operations, the concrete will be transported in a revolving drum truck mixer. As will be noted later, loading sequences to ensure uniform mixing in truck mixers must not attempt to achieve uniform ribbon loading or blending of ingredients.

Although most State Departments of Transportation (DOTs) permit 60 or 90 s mixing time in central mixers, ASTM C 94/C 94M still requires a minimum of 1 min mixing for the first cubic yard and an additional 15 s for each additional cubic yard. This minimum can be reduced if mixing uniformity criteria are met after shorter periods.

Shrink Mixing

Early in the development of the ready-mixed concrete industry, shrink mixing was designed to permit hauling a larger batch in a truck mixer. The idea was to partially mix and shrink the volume of concrete before it was placed in a truck mixer for final mixing. Because of weight laws only a small percentage of producers are able to ship a mixer loaded to 80 % of gross drum volume. A large number of central mix plants use shrink mixing to optimize production cycle times by using a shorter period of 30 to 60 s of mixing in the central mixer and complete the process by additional mixing at mixing speed in the truck mixer. This also reduces wear in the truck mixers.

Truck Mixing

Two general types of inclined axis truck mixers are in use today: the traditional rear discharge unit, and the front discharge unit. Because the front discharge unit requires a special truck chassis, it tends to be a somewhat more expensive unit than a rear discharge unit. With a rear discharge unit, the mixer can be more easily positioned on the truck chassis to comply with the truck weight laws of the various states.

Many contractors prefer front discharge units because the truck driver can drive into the job with little direction from contractor personnel, control chute movement, and discharge from within the truck cab.

The requirements of ASTM C 94/C 94M for truck mixers are that the volume of mixed concrete not exceed 63 % of the gross drum volume for truck-mixed or shrink-mixed concrete or 80 % when used as an agitator for central mixed concrete.

The extra carrying capacity of central mixed concrete is much less of a consideration with present weight laws and today's 9 to 11 yd^3 (7 to 8.5 m^3) mixers. When units were only 5 or 6 yd^3 (4 to 4.5 m^3) capacity, the angle of inclination of the drum was greater and mixers would hold 80 % of the gross drum capacity, even in hilly areas. With the larger units used today the angle of inclination of the drum to the horizontal is less and few, if any, can carry 80 % of the gross drum volume without spilling, especially in hilly areas. The large discharge openings used on truck mixers designed to discharge low slump concrete for slip formed and paving applications are especially sensitive to spilling concrete when they are used to deliver concrete at a slump higher than about 4 in. (100 mm).

Front discharge units tend to have much larger gross drum volumes than rear discharge units to allow for the cylindrical section to clear over and past the truck cab and to avoid spilling concrete. The result is that the manufacturer's rated mixing capacity may be less than half the gross drum volume. The Standards of the Truck Mixer Manufacturers Bureau (TMMB) [54] establishes minimum and maximum gross volume requirements for standard rated sizes of rear discharge truck mixers. The same minimum limits apply to front discharge units but the maximum limits are not applicable. All truck mixers that have a TMMB Rating Plate conform to C 94/C 94M required maximum of 63 % of gross drum volume for concrete mixing [54].

ASTM C 94/C 94M requires revolution counters on truck mixers and that the mixer be capable of mixing concrete in 70 to 100 revolutions.

Uniformity of mixing in truck mixers depends greatly on the batching sequence used to load cement aggregates and water into the drum for mixing.

1. Ribbon Loading. Attempting to ribbon all materials simultaneously should not be done, but with several modifications it is preferred and most often used. The charging of solids into a mixer should be started with the coarse aggregate to ensure that about 10 % of the required weight enters into the head of the drum. Then the coarse aggregate and sand should be ribbon loaded together with about 10 % coarse aggregate loaded last to clean off the blades in the discharge end. Starting the charging sequence with coarse aggregate helps avoid sand packing in the head of the drum. The cementitious materials should be ribboned with fine and coarse aggregate starting after the sand starts, extending through the charging procedure but ending before the coarse aggregate to reduce dust. The charging sequence of the water is important. About 25 % of the added water is batched as the last ingredient either from the plant or the rear nozzle from the truck water tank. The other 75 % of the added water is incorporated prior to the solids and portions ribboned with the solids. The final water batched washes all the solids into the mixer and ensures uniform slump and strength after adequate mixing.
2. Cement-last loadings are typically used for special cements or when charging cement from a remote bin. This loading follows procedures similar to ribbon loadings for aggregate except adding a higher percentage of coarse aggregate up front. The charging procedure is sensitive to the quantity of water added at the tail end and this quantity may need some adjustments.
3. Slurry Mixing. Today there are two types of slurry mixing, one where water is batched into the truck mixer first followed by the cementitious materials which are then mixed for a minute or so and then the aggregates are added. Although there is often insufficient water to make a true slurry, this procedure will *always* eliminate cement balls. This procedure slows down the loading process and tends to produce cement dust, but in mixer drums without large buildup of hardened concrete and worn blades it produces uniformly mixed batches of concrete. The other type of slurry mixing is a process where a slurry is mixed in a separate high energy mixer in the plant with the slurry discharged into the truck mixer as the aggregates are being loaded. The specific loading and mixing procedures vary with the particular slurry mixer used.

"Cement balls" and "sand streaks" are sometimes a problem in truck mixers with the traditional ribbon and cement-last loadings. Generally what happens is that the sand charging is started too early, packs into the head end of the drum and does not get mixed until some of the concrete has been discharged. Cement balls consist of cement, sand, and coarse aggregate. They can usually be ground up by mixing concrete with a slump of 3 in. (75 mm) or less and then adjusting the slump to the desired level. Concretes with slumps over 6 in. with high cement factors are more prone to cement balls but usually a loading procedure can be found to avoid them without going to slurry mixing.

Another sign of improper mixing is the observation of streaks of sand during discharge and could be related to the internal condition of the mixer.

Mixing Uniformity Testing

The limits set for mixing uniformity include tests for:

1. air-free unit weight of concrete,
2. air-free unit weight of mortar,
3. air content,
4. slump,
5. coarse aggregate content, and
6. 7-day compressive strength.

Acceptable performance requires compliance with five of the six tests. Two distinct samples are taken after discharging approximately 15 and 85 % of the load. For central mixers, the samples can be taken during discharge or directly from the mixer at points approximately equidistant from the front and rear portions of the load.

Slump tests of samples taken after the discharge of 15 and 85 % of the load can be made as a quick test of the probable degree of mixing uniformity. C 94/C 94M also requires mixers to be examined or weighed routinely to detect accumulations of hardened concrete. The NRMCA Plant Certification program has a process in place for an annual inspection of trucks that is a requirement for obtaining a Certification of Conformance for the production facility [46].

Generally, it has been found that when blade wear or accumulations of hardened concrete have become significant enough to affect mixing uniformity, discharge performance will also have deteriorated enough to be noticeable, particularly with moderately low slump concrete.

Control of the Addition of Water

ASTM C 94/C 94M recognizes that concrete loses slump with the passage of time and that either water will have to be added to restore slump or the slump on initial mixing will have to be higher than that required at the job. The rate at which slump is lost depends on a great number of factors, including concrete temperature, properties of the cementitious materials, and the admixtures used. The literature on this issue is extensive [51,55–58].

Under ASTM C 94/C 94M, within the limits set by the maximum w/cm ratio, water can be added only once on arrival at the job. Because of the difficulty of measuring aggregate moisture and inevitable traffic or job site delays, concrete is generally shipped from the plant at a slump less than the specified maximum with some water held back to allow for a job site adjustment. Repeated tempering or retempering, especially after discharge of a significant quantity, should not be permitted. However, initial tempering to obtain the desired slump is routinely necessary. The "one" addition of water permitted in ASTM C 94/C 94M should not be taken too strictly and the driver should be permitted to adjust the amount added over a period of perhaps 3 to 5 min on arrival at the job site.

The specification requires 30 revolutions at mixing speed to ensure incorporation of the water. Tests by NRMCA confirm that the 30 revolutions are necessary if mixing is at less than about 15 rpm. However, if mixing is at 22 to 25 rpm as few as 5 or 10 revolutions will be sufficient [58]. Many modern day mixers are not equipped to mix at speeds higher than about 20 rpm.

The specifications further require that discharge be completed within 90 min after the cement is wetted or before the drum has completed 300 revolutions, whichever comes first. Both can be waived by the purchaser if the concrete can be placed without the addition of water. The 90-min time limit has been one of the more controversial requirements in the specification. Some time ago an attempt was made to allow 90 min to the start of discharge with the provision that the concrete could be used as long as the slump was acceptable for placement and no water was added. The proposal was not accepted by the ASTM Subcommittee. However, in the authors' opinion, it should have been. Both field and laboratory data demonstrate that concrete strengths tend to improve with time, but *only* when water is not added [51,55].

Clearly, the 90-min time limit is too conservative when the concrete temperature is less than about 70°F (21°C) [59]. At higher temperatures, the time limit can only be justified by a concern that the prohibition against water addition, after the adjustment on arrival at the job site, cannot or will not be enforced.

The 300 revolution limit can be a problem with longer haul distances and job site adjustments that require subsequent mixing. In the past, keeping within the 300 revolution limit was a problem with addition of high range water reducing admixture at the job site. This was done because the older generation of HRWR admixtures resulted in a high rate of slump loss. With the carboxylate-based HRWR admixtures, slump loss is less of a problem and the admixture is added at the plant to a higher slump such that the desired slump is in the required range at the job site. Concrete producers also prefer to incorporate the admixture at the plant, and if slump loss is a problem a small water addition at the job site will be sufficient. It is important to note and recognize that the accuracy of the slump test deteriorates at these high slump levels.

Except for a very few soft aggregates, the 300 revolution limit is of no practical consequence. With soft aggregates that are subject to grinding, the effect will be to decrease slump, resulting in an increase of the mixing water requirement. Sand is more subject to grinding than coarse aggregate because of its large surface area. The 300 revolution limit was developed many years ago when mixers were powered by separate mixer engines and had only one basic drum speed, about 6 rpm. The limit on revolutions tended to control delivery time.

C 94/C 94M provides for means to make job-site adjustments for water and air by determining slump and/or air content on a preliminary sample before a significant quantity is discharged. At least 30 revolutions are required at mixing speed to incorporate these additions into the concrete mixture. These adjustments can add up towards the limits of time and total number of revolutions.

The ASTM specifications recognize that many of the requirements such as the 90-min time limit can be waived by the specifier. This is the type of issue that should be discussed at a pre-job conference [10].

C 94/C 94M covers concrete furnished in both cold and hot weather. In cold weather a minimum concrete temperature requirement is imposed based on the section size and this is repeated from ACI 306R [60]. C 94/C 94M does not specify a maximum concrete temperature in hot weather but indicates in a note that problems may be encountered as concrete temperatures approach 90°F. Local specifications and practices typically address temperature criteria for concrete.

Volumetric Batching and Continuous Mixing

Volumetric concrete mixers have been available since the 1960s, and are used to produce concrete in accordance with ASTM C 685/C 685M. The equipment is truck- or trailer-mounted and consists of individual bins for sand, coarse aggregate, cement, and water. The aggregates are delivered through calibrated gates that control the amount of material being delivered. Water and admixtures are controlled with valves and flow meters. Production rate is inversely proportional to cement content. The materials are fed into an inclined chute that contains a mixing auger. The auger mixes and elevates the concrete for discharge. The units are available in sizes from 4 to 12 yd^3 (3 to 9 m^3). The mixing auger holds about 2 ft^3 (0.06 m^3) of concrete and mixing time is 15 or 20 s. Slump is readily adjustable and the unit can be started and stopped as concrete is needed. ASTM C 685/C 685M requires calibration at six-month intervals.

The unit is versatile and has been used in a variety of work, both where the amount of concrete is relatively limited, and for specialty applications. In somewhat larger work where requirements are less than about 40 yd^3 (30 m^3) per hour, it has been used as a job-site mixing plant.

The advantage of the unit is that it produces freshly mixed concrete. Therefore, strengths and other properties are improved. The units may encounter problems with false setting cements because of the very short mixing time.

The Volumetric Mixer Manufacturers Bureau (VMMB), an organization of equipment manufacturers, has established standards for volumetric concrete mixers that establish minimum bin capacities for rated capacity of concrete that can be produced. The standard reflects many of the requirements of C 685/C 685M and requires the manufacturers to conduct a mixing uniformity evaluation on units of a similar design. Mixers that meet the requirements of these standards are eligible for VMMB rating plates [61].

Sampling and Testing

Testing Laboratories

Both ASTM C 94/C 94M and C 685/C 685M contain requirements that the individual who samples and tests the concrete be qualified and knowledgeable in the proper conduct of the test procedures required. This requires an ACI Concrete Field Testing Technician, Grade I certification, or an equivalent process. The concept of requiring demonstrated knowledge of the test procedures has grown rapidly. As of 2004 there were about 57 000 technicians currently certified under the ACI Grade I Field Testing Technician program. More recently, C 94/C 94M has included requirements for certification of laboratory technicians who are involved in concrete acceptance testing. Certification requirements for laboratory technicians are also a requirement for laboratories that conform to ASTM C 1077. In 2004 there were about 3000 laboratory technicians certified through one of the ACI Laboratory Technician Certification programs. The ACI and the National Institute for Certification in Engineering Technologies (NICET) personnel certification programs are becoming increasingly popular.

Requirements for accreditation of laboratory facilities that conduct acceptance testing are also increasing. Some of the organizations that sponsor accreditation programs for laboratories that conduct testing of construction materials include:

1. AASHTO–American Association of State and Highway Transportation Officials in Washington, District of Columbia.
2. A2LA–American Association of Laboratory Accreditation, in Gaithersburg, Maryland.
3. NVLAP–National Voluntary Laboratory Accreditation Program, in Gaithersburg, Maryland.

The AASHTO Accreditation program is probably the most recognized by state highway agencies and is generally a requirement for acceptance testing on transportation construction projects. There are about 900 accredited laboratories under the AASHTO program and all 50 state central laboratories hold the AASHTO accreditation. Another popular regional laboratory accreditation program is that of the Concrete Materials Engineering Council (CMEC), in Orlando, Florida. The program is required by the Florida Building Code and is supported by the concrete industry, engineering testing laboratories, and the Florida Department of Transportation. In 2004 about 110 laboratories in Florida were certified and accredited by CMEC to comply with ASTM C 1077.

Sampling

Under ASTM C 94/C 94M, concrete samples, except those for uniformity testing, are required to be taken in accordance with ASTM Practice for Sampling Freshly Mixed Concrete (C 172). Generally, this means the sample should be taken at two or more regularly spaced intervals "during discharge of the middle portion of the batch." In practice, this is seldom done and the sample is taken as a single increment near the start of discharge. Preliminary tests of air content and slump are permitted if the samples are obtained after at least $^1/_4$ yd^3 ($^1/_4$ m^3) has been discharged. The procedure in ASTM C 685/C 685M permits sampling any time after at least 2 ft^3 (0.06 m^3) has been discharged.

Sampling at a single point during discharge should be permitted in ASTM C 94/C 94M and C 172. The risk is that when cement is the last ingredient loaded in the mixer and the concrete is not well mixed, the first concrete discharged can have high strength and will not be representative of the majority of the batch. More often, the first portion of concrete discharged is of a more fluid consistency than the bulk of the load and samples from this portion are more likely to have lower measured strengths.

Compressive Strength Testing

ASTM C 94/C 94M requires air, slump, and density tests when strength specimens are made. If either falls outside the specifications, a retest of a new sample is required before the concrete is considered to have failed. Additionally, if the air or slump is low, additional air-entraining admixture or water can be added. The density test is considered useful for yield calculations and additional information in the case of problems with test results. This section of C 94/C 94M allows density tests to be conducted in accordance with C 138/C 138M, which will allow the measurement with the base of a pressure air meter.

Both ASTM C 94/C 94M and C 685/C 685M require that strength specimens be made in accordance with ASTM Test Methods of Making and Curing Concrete Test Specimens in the Field (C 31), cured under standard moist curing procedures and tested by ASTM Test Method for Compressive Strength of Cylindrical Concrete Specimens (C 39). A test is defined as the average of results from two cylinders made from the same sample and tested at the same age. ASTM C 31 indicates that 6 by 12 in. (150 by 300 mm) is the standard size, but allows 4 by 8 in. (100 by 200 mm) specimens when required by the project specifications. Increasingly, 4 by 8 in. (100 by 200 mm) cylinders are being used for concretes with specified strengths greater than about 8000 psi (55 MPa). This is because few compression testing machines are available with load capacities greater than 300 000 lb (1335 kN), that is, 10 600 psi (73 MPa) on a 6 by 12 in. (100 by 300 mm) cylinder.

Several years ago, an unsuccessful attempt was made to modify the section of ASTM C 94/C 94M that required discarding the result of a test of a single cylinder if it "shows definite evidence, *other than low strength,* of improper sampling, handling, curing, or testing." A proposal was made to set a limit on the range of pairs of cylinders that are averaged for a test and permit discarding the low value.

The average within-test coefficient of variation of 6 by 12 in. (150 by 300 mm) cylinder tests, for strength levels less than about 8000 psi (55 MPa), is about 2.9 %, as indicated in the precision statement of ASTM C39, or even a little better [62]. Data has shown that the multilab coefficient of variation of strength test results, developed from several labs testing specimens made from the same batch, is in the range of 5 % [63]. Within-test coefficient of variation of 4 by 8 in. (100 by 200 mm) cylinders is in the range of 3.0 % [64].

If the range between two cylinders exceeds 8 or 9 % of their average more than 1 time in 20, the test should be considered suspect. Here is some general guidance that is the opinion of the authors:

1. If the higher of the two values is more like the other tests of this concrete, and the 7-day result is normal, discard the lower result. If neither cylinder is unusually high or low, average the two and accept the result.
2. And whether or not the lower or higher test is disregarded, consider the possibility that the sampling and testing may be poor (see Table 1 [62]). Testing with a within-test coefficient of variation of 4 % will have one range in three exceeding 5.5 %! Improve testing.

Table 1 can be used to evaluate cylinder testing data, but recognize that whether a single low test is sufficiently unusual to discard the result depends on "normal" quality of the testing on that job. What this means is that when 15, 30, or 60 pairs of cylinders have been tested, calculate the ranges and convert the average range to the estimated coefficient of variation.

TABLE 1—Standards for Evaluating Performance of Testing Using Within Test Coefficient of Variation [62]

Excellent	Below 1.5 %
Very good	1.5 to 2.0 %
Good	2.0 to 3.0 %
Fair	3.0 to 4.0 %
Poor	Above 4 %

$$C_v = \frac{F \times \overline{R}}{\overline{X}}$$

where

C_v = coefficient of variation, %;
$\overline{R}$ = average range, psi (MPa);
F = factor used to estimate standard deviation from average range: 0.8865 for two cylinders; 0.5907 for three cylinders; and
$\overline{X}$ = average strength.

If the coefficient of variation is much over 2.9 %, realize that many, if not most, jobs (laboratories) can do better. Note also that if you have less than 60 pairs of results, the frequency of large ranges can be larger than indicated in Table 1. With only ten results, the ranges in Table 1 would be about 10 % larger [65].

There is some concern that the within-test coefficient of variation may be larger for very high strength concretes; but in the writers' opinion, this is the result of problems of capping, curing, and testing these concretes. With careful attention, it should be possible to obtain a within-test coefficient of variation of 2.9 % or less. However, sulfur mortar caps must be less than 3/32 in. (2.4 mm) thick and probably should not be permitted on concrete with strength greater than about 10 000 psi (70 MPa). Neat cement caps are an option and often ground ends are not sufficiently flat to give optimum results. Use of unbonded caps, C 1231, is now permitted for concrete strengths up to 12 000 psi (80 MPa).

In 2005 ASTM C 94/C 94M and C 685/C 685M will be revised to conform to the two acceptance criteria used in the ACI 318 building code:

1. The average of any three consecutive strength tests should be equal to or greater than the specified strength, $f'c$.
2. No individual strength test should be more than 500 psi (3.4 MPa) below the specified strengths up to 5000 psi [35 MPa] or less than 0.90 f'_c for specified strengths equal to or greater that value.

A table provides advice on the "over-design" needed to meet these requirements, depending on the standard deviation expected. The values given have been calculated from the equations given in ACI 318–02 [14], with correction factors for instances where the standard deviation is calculated from between 15 and 30 tests.

Failure to Meet Strength Requirements

A section in both specifications requires that, if the concrete was properly tested, the manufacturer and purchaser confer to see if they can agree on what adjustments, if any, should be made. If they do not agree, then a decision is to be made by a panel of three engineers. An arbitration process is preferred and recommended as the first course of action.

NRMCA *Publication 133* [66] outlines an orderly and deliberate process for determining the strength of the concrete in the structure and for developing information on the assignment of responsibility for deficiencies, if any. The practice suggests that if the cause of the low strength was improper testing, the party responsible should bear the cost of the investigation.

Closure

A statement in the 1994 version of this chapter recognized that if everyone would accept responsibility for quality, not just the quality control department, things will change greatly. The quality focus has been emphasized from the federal and state agencies through initiatives like the National Partnership for Highway Quality and the FHWA "Highways for LIFE" Program. The ready-mixed industry has made great strides towards the goal of higher level of quality and this commitment is clear at the highest levels of companies. There is a clear recognition that an effective means of promoting the increased use of concrete is to make certain that the quality assurance process is in place, that the producer is confident that the product will perform, and that the customer is assured of the producer's reliability. Towards that end the industry has increased its investment in its people by requiring education and industry certifications of drivers, plant operators, sales force, quality control personnel, and middle and upper management.

There are new challenges on the regulatory front that changes the way the concrete industry can function. Many of these have an impact on the technical aspects of the product. The industry has evolved to highly automated solutions of environmental management that facilitates the processing and reuse of returned concrete and wash water. To the extent that innovations have been developed, the specifications have not kept pace. A significant aspect in the future is the focus on sustainable and "green" construction. This will offer new challenges relative to the ingredients we use to make concrete. Sustainability not only includes recycling but also conservation of energy and resources and prolonged service life.

Self-consolidating concrete is likely to be the norm for most structural concrete in the future and this will require the development and revision of existing standards to allow for its acceptance and use. Construction defect litigation has developed a high level of caution and conservatism within the construction industry and this stifles innovation. This has also resulted in a reluctance by some entities to accept responsibility for certain aspects of projects.

There is a strong desire of the more technically competent concrete producers and contractors, both small and large, to evolve construction specifications to achieve a higher emphasis on performance while eliminating prescriptive limits, especially when they create conflicts in specifications or cause problems with the placement or service life of the structure. This desire to attain more control of the product they produce will also require a higher level of technical competence and acceptance of responsibilities. The future of concrete construction will only get better with these initiatives in place.

In 2005, ASTM International and NRMCA published a more detailed discussion of ASTM C 94/C 94M that analyzes the requirements and intent of every clause in the specification supported by figures and numerical examples [67]. This reference will be of interest to readers of this chapter.

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Lightweight Concrete and Aggregates

Thomas A. Holm[1] *and John P. Ries*[1]

Preface

THE GENERAL PRESENTATION OF THIS EDITION IS similar to previous editions, with additional information on structural properties of lightweight aggregates, as well as strength making, durability, and placement characteristics of lightweight concrete to reflect the current state of the art. This edition also includes new discussions relative to the moisture dynamics and the enhanced hydration of cementitious materials brought about by internal curing when using water-entraining lightweight aggregates. Specified density concrete, concrete with densities between traditional lightweight and normal-weight concretes, is also discussed. In *ASTM STP 169* this chapter was authored by R. E. Davis and J. W. Kelly. *ASTM 169A* and *ASTM 169B* were authored by D. W. Lewis. *ASTM 169C* was authored by T. A. Holm.

Classification of Lightweight Aggregates and Lightweight Aggregate Concretes

Lightweight aggregate concretes are broadly divided into three groups based upon their use and physical properties: structural, structural/insulating, and insulating. Bulk density, thermal conductivity, and compressive strength ranges normally associated with each class of concrete are summarized in Table 1.

This chapter primarily addresses structural concretes where weight reduction is achieved through the use of lightweight aggregates. Cellular concrete is covered in a separate chapter in this volume, where lighter weight is developed primarily by inclusion of large amounts of air or gas through the use of foaming-type agents.

Structural

Structural lightweight concretes generally contain aggregates made from pyroprocessed shales, clays, slates, expanded slags, expanded fly ash, and those mined from natural porous volcanic sources. Structural lightweight concrete is normally classified by a minimum compressive strength that was jointly established by the ASTM Specification for Lightweight Aggregates (C 330) and the Standard Building Code for Reinforced Concrete (ACI 318) [1]. The 318 code definition is structural concrete made with lightweight aggregate; the equilibrium density as determined by ASTM Test Method for Determining Density of Structural Lightweight Concrete (C 567) not exceeding 115 lb/ft^3 and the compressive strength is more than 17.2 MPa (2500 psi). This is a definition, not a specification and project requirements may permit higher equilibrium densities. Although structural concrete with equilibrium densities from 1450 to 1920 kg/m^3 (90 to 120 lb/ft^3) are often used, most lightweight aggregate concrete used in structures have equilibrium densities between 1760 to 1840 kg/m^3 (110 and 115 lb/ft^3).

Virtually all manufactured structural lightweight aggregates are produced from raw materials including suitable shales, clays, slates, fly ashes, or blast furnace slags. Naturally occurring lightweight aggregates are mined from volcanic deposits that include pumice and scoria types. Pyroprocessing methods include the rotary kiln process (a long, slowly rotating, nearly horizontal cylinder lined with refractory materials similar to cement kilns); the sintering process wherein a bed of raw materials including fuel is carried by a traveling grate under ignition hoods; and the rapid agitation of molten slag with controlled amounts of air or water. No single description of raw material processing is all-inclusive and the reader is urged to consult the lightweight aggregate manufacturer for physical and mechanical properties of the aggregates and the concretes made with them.

Structural lightweight aggregates can be manufactured from raw materials such as, for example, soft shales and clays that have limited structural applications in their natural state. This is an environmentally sound practice as it minimizes demands on finite resources of quality natural sands, stones, and gravels.

Structural/Insulating

Industrial applications that call for "fill" concretes often require compressive strengths and densities in the intermediate between structural and insulating concretes. These concretes may be produced with high air contents and include structural lightweight aggregate, or sanded insulating lightweight aggregate mixtures, or they may incorporate both structural and insulating lightweight aggregates. Compressive strengths from 3.4 to 17 MPa (500 to 2500 psi) are common with thermal resistance ranging between insulating and structural concrete.

Insulating

Insulating concretes are very light nonstructural concretes, employed primarily for high thermal resistance, that incorporate low-density low-strength aggregates such as vermiculite and perlite. With low densities, seldom exceeding 800 kg/m^3 (50 lb/ft^3), thermal resistance is high. These concretes are not intended to be exposed to weather and generally have a compressive strength range from about 0.69 to 3.4 MPa (100 to 500 psi).

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TABLE 1—Lightweight Aggregate Concrete Classified According to Use and Physical Properties[a]

Class of Lightweight Aggregate Concrete	Type of Lightweight Aggregate Used in Concrete	Typical Range of Lightweight Concrete Density	Typical Range of Compressive Strength	Typical Range of Thermal Conductivities
Structural	Structural-grade C 330	1440 to 1840 (90 to 115) at equilibrium	$>$ 17 ($>$ 2500)	not specified in C 330
Structural/Insulating	Either structural C 330 or insulating C 332 or a combination of C 330 and C 332	800 to 1440 (50 to 90) at equilibrium	3.4 to 17 (500 to 2500)	C 332 from (0.22) (1.50) to (0.43) (3.00) oven dry
Insulating	Insulating-grade C 332	240 to 800 (15 to 50) oven dry	0.7 to 3.4 (100 to 500)	C 332 from (0.065) (0.45) to (0.22) (1.50) oven dry

[a] Densities are in kg/m^3 (lb/ft^3), compressive strengths in MPa (psi), and thermal conductivity in W/m · °K (Btu · in./h · ft^2 · °F).

Properties of Lightweight Aggregate

Internal Structure of Lightweight Aggregates

Lightweight aggregates have a low particle density because of the cellular structure. The cellular structure within the particles is normally developed by heating certain raw materials to incipient fusion, at which temperature gases are evolved within the pyroplastic mass causing expansion that is retained upon cooling. Strong, durable, lightweight aggregates contain a uniformly distributed system of pores that have a size range of approximately 5 to 300 μm (0.000040 in.) and which are developed in a relatively crack-free, high-strength vitreous matrix (Fig. 1).

Particle Shape and Surface Texture

Depending on the source and the method of production, lightweight aggregates exhibit considerable differences in particle shape and texture. Shapes may be cubical, rounded, angular, or irregular. Textures may range from fine pore, relatively smooth skins to highly irregular surfaces with large exposed pores. Particle shape and surface texture directly influence workability, coarse-to-fine aggregate ratio, cement content requirements, and water demand in concrete mixtures, as well as other physical properties.

Relative Density

The relative density of an aggregate is the ratio between the mass of the material and the volume occupied by the individual particles contained in that sample. This volume includes the pores within the particles but does not include the voids between the particles. Relative density of individual particles depends both on the relative density of the poreless vitreous material and the pore volume within the particles, and generally increases when particle size decreases. The relative density of the pore-free vitreous material may be determined by pulverizing the lightweight aggregate in a jar mill and then following procedures used for determination of the relative density of cement.

Bulk Density

Aggregate bulk density is defined as the ratio of the mass of a given quantity of material and the total volume occupied by it. This volume includes the voids between, as well as the pores within, the particles. Bulk density is a function of particle shape, density, size, gradings, and moisture content, as well as the method of packing the material (loose, vibrated, rodded), and varies not only for different materials, but for different sizes and gradations of a particular material. Table 2 summarizes the maximum bulk density for lightweight aggregates listed in ASTM (C 330) and ASTM Specification for Lightweight Aggregates for Concrete Masonry Units (C 331). ASTM Standard Specification for Lightweight Aggregates for Insulating Concrete (C 332) provides minimum density requirements for perlite and vermiculite to limit over-expanded, weak particles that would break down in mixing. The relationship between the particle relative density and the bulk density of a sample is illustrated in Fig. 2 for a hypothetical lightweight aggregate.

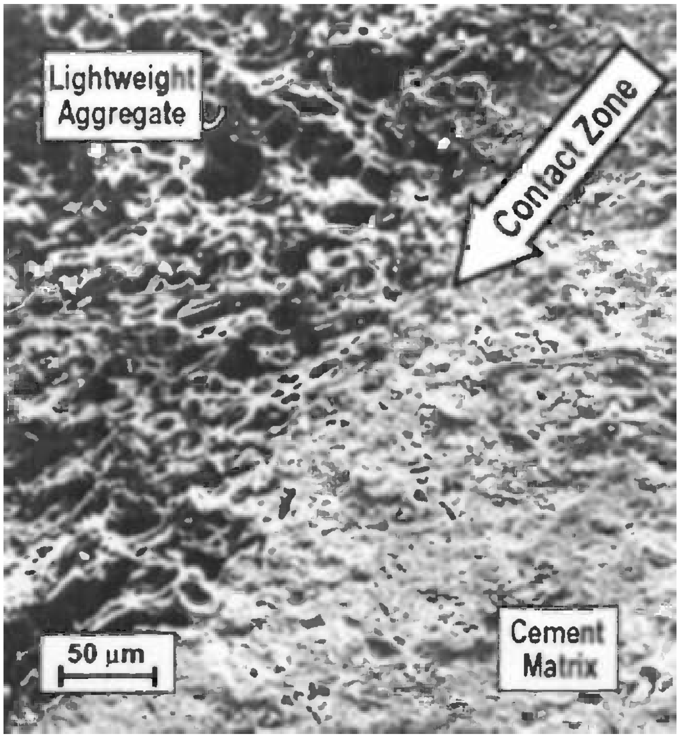


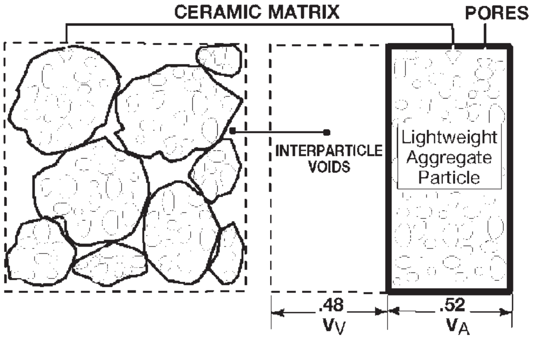
Fig. 1—Contact zone—structural lightweight concrete from 30-year-old bridge deck, W. P. Lane Memorial Bridge over the Chesapeake Bay, Annapolis, Maryland: compression strength 24 MPa (3500 psi); density 1680 kg/m^3 (105 lb/ft^3).

TABLE 2—Requirements of ASTM C 330, C 331, and C 332 for Dry Loose Bulk Density of Lightweight Aggregates

Aggregate Size and Group	Maximum Dry Loose Bulk Density kg/m³ (lb/ft³)		Minimum Dry Loose Bulk Density kg/m³ (lb/ft³)	
ASTM C 330 and C 331				
fine aggregate	1120	(70)	...	
coarse aggregate	880	(55)	...	
combined fine and coarse aggregate	1040	(65)	...	
ASTM C 332				
Group 1				
Perlite	196	(12)	120	(7.5)
Vermiculite	160	(10)	88	(5.5)
Group 2				
fine aggregate	1120	(70)	...	
coarse aggregate	880	(55)	...	
combined fine and coarse aggregate	1040	(65)	...	

Grading

Grading requirements are generally similar to those provided for normal-weight aggregate with the exception that lightweight aggregate particle size distribution permits a higher weight through smaller sieves. This modification recognizes the increase in relative density typical for the smaller particles of most lightweight aggregates, and that while standards are established by weights passing each sieve size, ideal formulations are developed through volumetric considerations.



The following calculations are based on a hypothetical lightweight aggregate sample (illustrated above) that has a bulk loose dry density of 44.6 lb/ft³ (714 kg/m³) and a relative density (SSD pychnometer) of 1.52 after a 24-hour soak resulting in a moisture content of 10.5% by weight. The relative density of the ceramic matrix was measured to be 2.60.

$$RD_D\left[\begin{array}{c}\text{Relative}\\\text{Density,}\\\text{Dry}\end{array}\right]=\frac{RD_{24}}{(1+M)}\left[\frac{\begin{array}{c}\text{Pychnometer Relative}\\\text{Density after 24-Hour Soak}\end{array}}{\begin{array}{c}\text{Moisture Content by}\\\text{Weight after 24-hour Soak}\end{array}}\right]=\frac{1.52}{1+.105}=1.38\ (1380)$$

$$V_A\left[\begin{array}{c}\text{Fractional Part of Bulk}\\\text{Volume Occupied by}\\\text{Aggregate Particles}\end{array}\right]=\frac{D_B}{RD_D}\left[\frac{\begin{array}{c}\text{Measured Bulk Dry}\\\text{Loose Density}\end{array}}{\begin{array}{c}\text{Relative Density}\\\text{of Dry Particle}\end{array}}\right]=\frac{714}{1380}=0.52$$

$$V_V\left[\begin{array}{c}\text{Fractional Part of Bulk}\\\text{Volume Occupied by Voids}\\\text{between Particles}\end{array}\right]=1.00-0.52=0.48$$

Fig. 2—Schematic representation of lightweight aggregate bulk volume, interparticle voids, and internal particle pores.

Structural lightweight aggregate producers normally stock materials in several standard sizes that include coarse, intermediate, and fine gradings. By combining size fractions or by replacing some or all of the fine fraction with a normal-weight sand, a wide range of concrete densities may be obtained. The aggregate producer is the best source of information for the proper aggregate combinations to meet fresh concrete density specifications and equilibrium density for dead load design considerations.

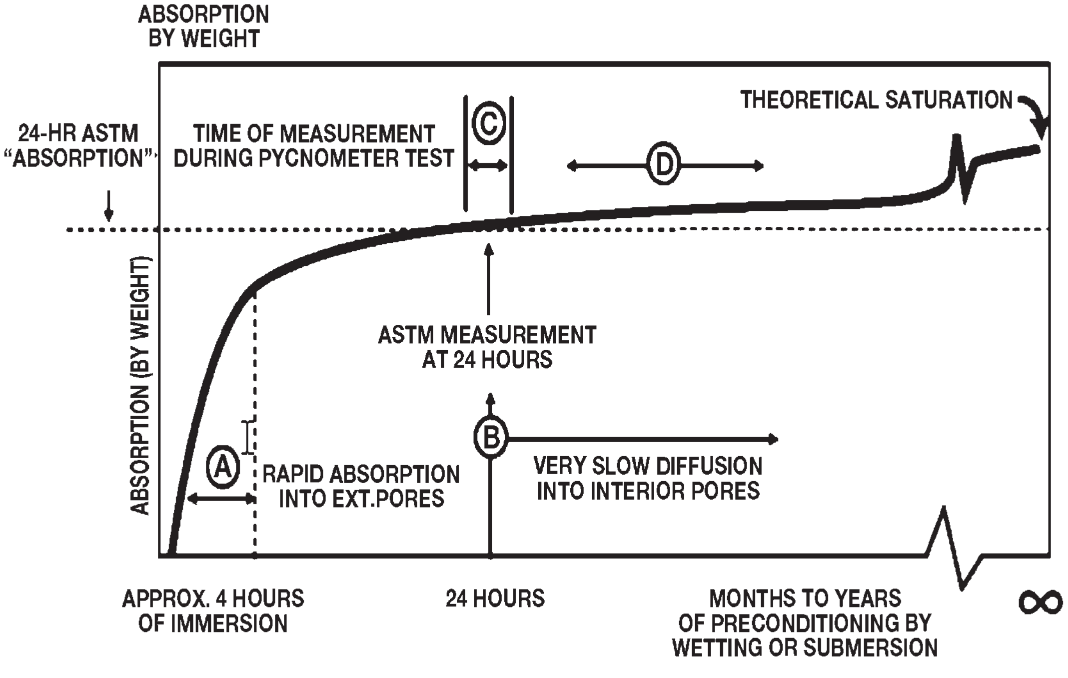
Absorption Characteristics

Due to their porous structure, lightweight aggregates absorb more water than their normal-weight aggregate counterparts. Based upon a 24-h absorption test, conducted in accordance with the procedures of ASTM Test Method Specific Gravity and Absorption of Coarse Aggregate (C 127) and ASTM Test Method Density, Relative Density and Absorption of Fine Aggregate (C 128), lightweight aggregates will absorb from 5 to more than 25 % by weight of dry aggregate. By contrast, normal-weight aggregates generally absorb less than 2 %. The important difference in measurements of stockpile moisture contents is that with lightweight aggregates the moisture is largely absorbed into the interior of the particles whereas in normal-weight aggregates it is primarily adsorbed, surface moisture. Recognition of this essential difference is important in mixture proportioning, batching, and field control. Rate of absorption of lightweight aggregates is dependent on the characteristics of pore size, connection, and distribution, particularly those close to the surface. Pores close to the surface are readily permeable and fill within the first few hours of exposure to moisture. Interior pores, however, fill slowly, with many months of submersion necessary. A fraction of the interior pores are essentially non-interconnected and remain unfilled after years of immersion.

Internally absorbed water within the particle is not immediately available for chemical interaction with cement and mixing water, but is extremely beneficial in maintaining longer periods of curing essential to improvements in the hydration of cement and the aggregate/matrix contact zone.

ASTM C 127 procedures prescribe measuring the "saturated" particle density in a pycnometer and then determining the absorbed moisture content on the sample that had been immersed in water for 24 h. With lightweight aggregate it is more accurate to report partially saturated after a 24-h soak because the particle is not fully saturated yet. After a 24-h immersion in water, the rate of moisture absorption into the lightweight aggregate will be so low that the partially saturated particle density will be essentially unchanged during the time necessary to take weight measurements in the pycnometer. After the moisture content is known, the over-dry particle density may be directly computed. As can be seen in Fig. 3, the rate of absorbtion can be divided into several regions.

Following the prescribed procedures, the degree of saturation, that is, the fractional part of the pore volume occupied by water, will generally be in the range of approximately 25 to 35 % of the theoretical total saturation of all pores for



Region A. Rapid entry of water by capillary absorption by close to surface pores within the first few hours.
Region B. Very slow diffusion into interior pores.
Region C. When the moisture content is approximately equal to that obtained by ASTM procedure (24 hour immersion), then the slope of the line reflecting further absorption represents the very slow process of diffusion. This is the basis for providing accurate relative density values during the relatively short time used to conduct pycnometer tests at 24 hours.
Region D. Absorption developed over an extended period of time used to mix, transport, place, and prior to initial set (6-8 hours ±) will be very small, and consequently the W/Cm ratio will be decreased by an equivalent small amount.

Fig. 3—Absorption vs. time for typical structural grade expanded shale, clay, or slate (ESCS).

structural lightweight aggregates. The use of the ASTM expression "saturated surface dry" for lightweight aggregates is theoretically inaccurate, analytically misleading, and, therefore, inappropriate.

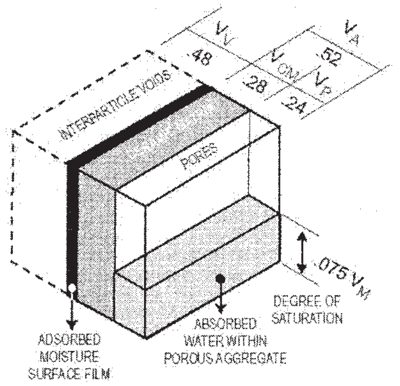
From a practical perspective and considering the fact that most lightweight concrete is placed by pumping, the usual practice is to batch the lightweight aggregate at a moisture condition greater than the "Absorption Value" defined by ASTM procedures (24-h immersion). In this condition the absorbed (internal) moisture content will be in excess of the arbitrarily defined ASTM 24-hour "absorption" value. The degree of saturation necessary for adequate pumping is determined by practical field experience for each aggregate and may be obtained from the lightweight aggregate supplier.

Due to pre-wetting, there will invariably be a film of surface water on the lightweight aggregate. As with normal-weight concrete it is essential to evaluate this quantity of surface water for an accurate determination of the "net" mixing water to achieve the desired workability and to determine the effective water-to-cementitious materials ratio.

To accurately determine the amount of absorbed water and the amount of surface water it is necessary to run the usual moisture test as follows. Measure the weight of the wet, ready-to-batch surface moist sample. After towel drying, measure the weight of the surface dry sample. Conduct the drying test to calculate the moisture content absorbed within the sample. The surface water (adsorbed) on the lightweight aggregate is then determined by the difference between the as-received and the absorbed moisture contents. (Fig. 4)

Proportioning Lightweight Concrete

Proportioning procedures used for ordinary concrete mixes apply to lightweight concrete with added attention given to



$$\text{Fractional Part of Lightweight Aggregate Paticle } (V_A) \text{ occupied by the solid ceramic Matrix} = \frac{RD_D}{RD_{CM}} \left[\frac{\text{Relative Dry Density}}{\text{Relative Density of the Solid Ceramic Matrix}}\right] = \frac{1.38}{2.60} = 0.53$$

$$\text{Fractional Part of Lightweight Aggregate Particle Occupied by Pores} = 1.00 - 0.53 = 0.47$$

$$V_{CM}\left[\begin{array}{c}\text{Fractional Part of}\\ \text{Bulk Volume occupied}\\ \text{by the Solid Ceramic}\\ \text{Matrix}\end{array}\right] = V_A \times \left[\begin{array}{c}\text{Fractional Part of}\\ \text{Aggregate Particle}\\ (V_A) \text{ occupied by}\\ \text{the Solid Ceramic}\\ \text{Matrix}\end{array}\right] = 0.52 \times 0.53 = 0.28$$

$$\therefore\ V_P\left[\begin{array}{l}\text{Fractional Part of}\\ \text{Bulk Volume}\\ \text{Occupied by Pores}\\ \text{in Aggregate}\end{array}\right] = 0.52 - 0.28 = 0.24$$

$$V_M\left[\begin{array}{c}\text{Fractional Volume}\\ \text{of Bulk Loose}\\ \text{Sample Occupied}\\ \text{by Moisture}\end{array}\right] = \frac{\left[\begin{array}{c}\text{Moisture Content}\\ \text{by Weight}\end{array}\right]\left[\begin{array}{c}\text{Bulk Loose Dry}\\ \text{Density of Sample}\end{array}\right]}{1000\left[\text{Density of Water}\right]} = \frac{0.105 \times 714}{1000} = .075$$

$$DS\left[\begin{array}{c}\text{Degree of Saturation}\\ \text{of the Pores Occupied}\\ \text{by Moisture}\end{array}\right] = \frac{0.075}{0.24} = 0.31\ ^{*}$$

* "Saturated Surface Day" after 24-hour submersion for this illustrative sample represents water filling only 31% of the available pore space.

Fig. 4—Schematic representation of volumes occupied by the ceramic matrix, the internal pores, and the degree of saturation of absorbed water (see Fig. 2).

concrete density and the water absorption characteristics of the lightweight aggregate, Standard Practice for Selecting Proportions for Structural Lightweight Concrete, ACI 211.2. Structural lightweight concretes are generally proportioned by absolute volume methods in which the fresh concrete produced is considered equal to the sum of the absolute volumes of cement, aggregates, net water, and entrained air. Proportioning by this method requires the determination of absorbed and surface moisture contents and the aggregate's relative density factor.

When lightweight aggregates have been preconditioned to levels of absorbed moisture greater than that developed after a one-day immersion, the rate of further absorption will be very low and the water-to-cementitious materials ratios can be established with precision. Thus, lightweight concrete can meet water-to-cementitious materials specification requirements with the same facility as normal-weight concrete. Water absorbed within the lightweight aggregate prior to mixing is not used for calculating the water-to-cementitious materials ratio at the time of setting. This absorbed water is available, however, for internal curing and the continued cement hydration after external curing has ended.

Air Content

As with normal-weight concrete, air-entrained lightweight concrete significantly improves durability and resistance to scaling, reduces density, and improves workability. With 4 to 6 % air contents, bleeding and segregation are reduced and mixing water requirements are lowered while maintaining optimum workability. Because of the elastic compatibility of the lightweight aggregate and mortar matrix, strength reduction penalties due to high air contents will be lower for structural lightweight concrete than for normal-weight concretes.

Air content of lightweight aggregate concretes is determined in accordance with the procedures of ASTM Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173). Volumetric measurements assure reliable results while the pressure method, Standard Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method (ASTM C 231), will provide erratic data due to the influence of aggregate porosity.

Air contents higher than required for durability considerations are frequently developed for high thermal resistance, or for lowering density of semi-structural "fill" concrete, with reduced compressive strength as a natural consequence.

Admixtures

Use of water reducers, retarders, and superplasticizers will result in improved lightweight concrete characteristics in a manner similar to that of normal-weight concretes; however, superplasticizers, while effective, will slightly increase the density of lightweight concrete.

Mixing, Placing, and Curing

Properly proportioned structural lightweight concrete can be mixed, delivered, and placed with the same equipment as normal-weight concretes. The most important consideration in handling any type of concrete is to avoid segregation of coarse aggregate from the mortar fraction. The basic principles required to secure a well-placed concrete also apply to lightweight concrete:

(a) well-proportioned, workable mixes that use a minimum amount of mixing water;
(b) equipment capable of expeditiously moving the concrete;
(c) proper consolidation in the forms; and
(d) quality workmanship in finishing.

Well-proportioned structural lightweight concretes can be placed and screeded with less physical effort than that required for ordinary concrete. Excessive vibration should be avoided, as this practice serves to drive the heavier mortar fraction down from the surface where it is required for finishing. On completion of final finishing, curing operations similar to normal-weight concrete should begin as soon as possible. Lightweight concretes batched with pre-wet aggregates carry their own internal water supply for curing and as a result are more forgiving to unfavorable ambient conditions and poor curing practices.

Prewetting

Lightweight aggregates may absorb part of the mixing water when exposed to increased pumping pressures. To avoid loss of workability, it is essential to properly prewet the lightweight aggregates prior to pumping. Prewetting is often done at the aggregate production plant and continued at the concrete plant or can be done entirely at the concrete plant by wetting stockpile with sprinkler systems. It is essential to consult the aggregate supplier on methods and duration of prewetting.

Prewetting will significantly reduce the lightweight aggregates' rate of absorption, minimizing water transfer from the mortar fraction that, in turn, causes slump loss during pumping. Prewetting will result in an increased relative aggregate density factor that, in turn, develops higher fresh concrete density. The higher water content due to prewetting will eventually diffuse out of the concrete, developing a longer period of internal curing as well as a larger differential between fresh and equilibrium density than that associated with normal-weight concretes. Aggregate suppliers should be consulted for mixture proportion recommendations necessary for consistent pumpability.

Internal Curing

Lightweight aggregate batched at a high degree of absorbed water may be substituted for normal-weight aggregates to provide "internal curing" in concrete containing a high volume of cementitious materials. High cementitious concretes are vulnerable to self-desiccation and early-age cracking, and benefit significantly from the slowly released internal moisture. Field experience has shown that high strength concrete is not necessarily high performance concrete and that high performance concrete need not necessarily be high strength. A frequent, unintended consequence of high strength concrete is early-age cracking. Blending lightweight aggregate containing absorbed water is significantly helpful for concretes made with a low ratio of water-to-cementitious material or concretes containing high volumes of supplementary cementitious materials that are sensitive to curing procedures. This process is often referred to as water entrainment.

Time-dependent improvement in the quality of concrete containing prewet lightweight aggregate is greater than that of concrete containing normal-weight aggregate. The reason is better hydration of the cementitious materials provided by moisture available from the slowly released reservoir of absorbed water within the pores of the lightweight aggregate. The fact that absorbed moisture in the lightweight aggregate is available for internal curing has been known for more than four decades. The first documentation of improved long term strength gains made possible by the use of saturated *normal-weight* aggregates was reported in 1957 by Paul Klieger [2], who, in addition, commented in detail on the role of absorbed water in lightweight aggregates for extended internal curing.

In his 1965 report, “Concrete Strength Measurement—Cores vs. Cylinders,” presented to the National Sand and Gravel Association and the National Ready Mixed Concrete Association, Bloem [3] states, “Measured strength for lightweight concrete cylinders was not reduced by simulated field curing methods employed. This would tend to support the suggestion that the high absorption of lightweight aggregate may have the beneficial effect of supplying curing water internally.” This was confirmed by Campbell and Tobin [4] in their comprehensive program which compared strengths of cores taken from field-cured exposed slabs with test results obtained from laboratory specimens cured strictly in accordance with ASTM C 31. Their tests confirmed that availability of absorbed moisture in lightweight aggregate produced a more forgiving concrete that was less sensitive to poor field curing conditions. Addressing the long-term service performance of lightweight concrete, Holm [5] cited the improved integrity of the contact zone between the lightweight aggregate and the matrix. The improved quality was attributed to internal curing, and better cement hydration and pozzolanic activity at the interface, and reduction in stress concentrations resulting from elastic compatibility of the concrete constituents.

The benefits of internal curing go far beyond any improvements in long-term strength gain, which from some combinations of materials may be minimal or nonexistent. The principal contribution of internal curing results in the reduction of permeability that develops from a significant extension in the time of curing. Powers [6] showed that extending the time of curing increased the volume of cementitious products formed which caused the capillaries to become segmented and discontinuous.

It appears that in 1991, Philleo [7] was the first to recognize the potential benefits to high performance normal-weight concrete possible with the addition of lightweight aggregate containing high volumes of absorbed moisture. Reduced sensitivity to poor curing conditions in concretes containing an adequate volume of prewet lightweight aggregate has also been reported [8]. Since 1995 a large number of papers addressing the role of water entrainment’s influence on internal curing and autogenous shrinkage have been published, of which Bentz et al. is typical [9].

The benefits of internal curing are increasingly important when supplementary cementitious materials (silica fume, fly ash, metokaolin, calcined shales, clays and slates, as well as the fines of lightweight aggregate) are included in the mixture. It is well known that the pozzolanic reaction of finely divided alumina-silicates with calcium hydroxide liberated as cement hydrates is contingent upon the availability of moisture. Additionally, internal curing provided by absorbed water minimizes the “plastic” (early) shrinkage due to rapid drying of concretes exposed to unfavorable drying conditions [10].

Sampling and Field Adjustments

Changes in lightweight aggregate moisture content, grading, or relative density, as well as usual job site variation in entrained air, suggest frequent checks of the fresh concrete to facilitate adjustments necessary for consistent concrete characteristics. Standardized field tests for slump, ASTM C 143; fresh density, ASTM C 138; and entrained-air content, ASTM C 173 are used to verify conformance of field concretes with the project specification. Sampling should be conducted in accordance with ASTM Practice Sampling Freshly Mixed Concrete (C 172). ASTM Test Method for Density of Structural Lightweight Concrete (C 567) describes methods for calculating the in-service, equilibrium density of structural lightweight concrete. When variations in fresh density exceed ± 4 lb/ft^3, an adjustment in batch weights may be required to restore specified concrete properties. To avoid adverse effects on durability, strength, and workability, air content should not vary more than ± 1.5 % from target values.

Properties of Lightweight Concrete

Comprehensive information detailing the properties of lightweight concretes and lightweight aggregates has been published by Shideler [11], Holm [12], Carlson [13], and Valore [14]. The first two deal with structural-grade concretes, Carlson reported on lightweight aggregate for concrete masonry units, and Valore covered both structural and insulating concretes. In most instances, test procedures for measuring properties of lightweight concretes were the same as commonly used for normal-weight concretes. In limited cases, different procedures particularly suited to measure lightweight concrete characteristics were developed.

Density

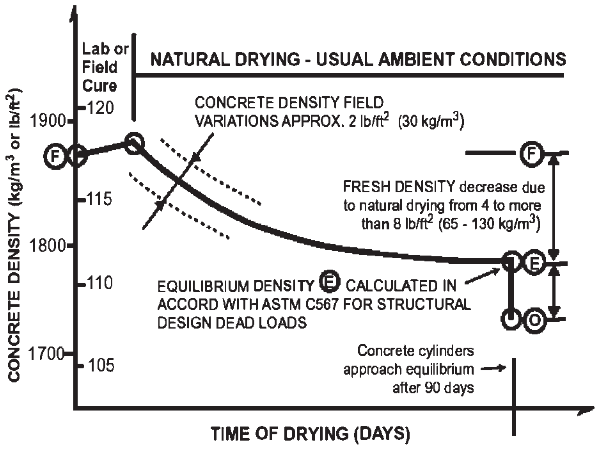
Although there are numerous structural applications for all lightweight concretes (coarse and fine lightweight aggregate), usual commercial practice in North America is to design lightweight concretes where part or all of the fine aggregate used is natural sand. Long-span bridges using concretes with three-way blends (coarse and fine lightweight aggregates and small supplemental natural sand volumes) have provided long-term durability and structural efficiency by increasing the density/strength ratios [15]. Normal-weight sand replacement will typically increase unit weight from about 80 to more than 160 kg/m^3 (5 to 10 lb/ft^3). Using increasing amounts of cement to obtain high strengths above 35 MPa (5000 psi), concrete will increase equilibrium density, ASTM C 567, from 32 to 96 kg/m^3 (2 to 6 lb/ft^3).

The fresh density of lightweight aggregate concretes is a function of mixture proportions, air contents, water demand, and the relative density and moisture content of the lightweight aggregate. Decrease in density of exposed concrete is due to moisture loss that, in turn, is a function of ambient conditions and surface area/volume ratio of the concrete element. Design professionals should specify a maximum fresh density for lightweight concrete, as limits for acceptability should be controlled at time of placement.

Unless otherwise specified, the dead loads used for design should be based upon the calculated equilibrium density that, for most conditions and structural members, may be assumed to be reached after 90 days. Extensive tests reported in ACI 213, Structural Lightweight Concrete, conducted during North American durability studies, demonstrated that despite wide initial variations of aggregate moisture content, equilibrium density was found to be about 50 kg/m^3 (3.1 lb/ft^3) above oven-dry density (Fig. 5). When weights and moisture contents of all the constituents of the batch of concrete are known, an approximate calculated equilibrium density may be determined.

Specified Density Concrete

The use of specified density concrete is based on engineers’ decisions to improve structural efficiency by optimizing concrete



F - FRESH DENSITY: Specified for field control (unit weight bucket). Measurements on 6' x 12' (150 x 300 mm) cylinders will average 2.5 lb/ft^3 (40 kg/m^3) higher than filed measurements on 0.5 ft^3 (.014 m^3) unit weight bucket.

E - EQUILIBRIUM DENSITY: Typically 3 lb/ft^3 (50 kg/m^3) greater than OVEN DRY DENSITY - O

Fig. 5—Concrete density versus time of drying for structural lightweight concrete.

density. Specified density concrete is defined as concrete with a range of density less than what is generally associated with normal-weight concrete, 2320–2480 kg/m^3 (145–155 lb/ft^3), and greater than the code-defined maximum density for lightweight concrete, 1840 kg/m^3 (115 lb/ft^3). Specified density concrete is achieved by replacing part of the ordinary normal-weight aggregate (Relative Density >2.60) with either coarse or fine lightweight aggregate (Relative Density generally < 1.60). Specified density concrete has been used on bridges, marine structures, precast elements, and consumer products in North America, Europe, and several other parts of the world [16].

The concept of specified density concrete is not new. For more than 20 years precast concrete manufacturers have evaluated trade-offs between the concrete density and transportation costs. Shown in Table 3 are the physical properties of concrete in which 25, 50, 75, and 100 % of the normal-weight limestone coarse aggregate was replaced by an equal absolute volume of lightweight aggregate [10]. This resulted in 5, 11, 15, and 21 % reductions in density, respectively.

By adjusting the density of the concrete, precasters are able to maximize the number of concrete elements on a truck without exceeding highway load limits. This reduces the number of truck loads which lowers transportation and project cost, as well as reducing the environmental consequences of trucking products, especially into central cities. Opportunities for increased trucking efficiency also apply when transporting smaller concrete products (hollow core plank, wallboard, precast steps, and other consumer products). Specified density concrete has the added benefit of enhanced cement hydration. See section on “Internal Curing” for more detail.

Compressive Strength

While most structural lightweight aggregates are capable of producing concretes with compressive strengths in excess of 35 MPa (5000 psi), a limited number of lightweight aggregates can be used in concretes that develop cylinder strengths from 48 to >69 MPa (7000 to >10 000 psi) [17].

While compressive strengths of 21 to 35 MPa (3000 to 5000 psi) are common for cast-in-place lightweight concretes, higher strengths are presently being specified for precast bridge mem-

TABLE 3—Physical Properties of Concrete Mixtures

Limestone Coarse Aggregate replaced by varying percentages of structural Lightweight Aggregate.
Concrete manufactured and tested at Prestressed Plant to optimize structural efficiency and reduce transportation costs.

Mixture Number Coarse Aggregate Target Equilibrium Density kg/m^3 (lb/ft^3)		1 Limestone 2300 (143)	2 .75S,.25L 2160 (135)	3 .5S 5L 2050 (128)	4 .25S,.75L 1920 (112)	5 LWA 1800 (112)	M NONE 2000 (125)
Physical Properties @ 18–24 h							
Compressive Strength	MPA (ksi)	24 (3.50)	26 (3.75)	29 (4.27)	28 (4.10)	26 (3.80)	34 (4.88)
Elastic Modulus (Test)	GPa (ksi × 10^3)	24 (3.42)	23 (3.30)	23 (3.27)	20 (2.97)	18 (2.67)	23 (3.38)
Elastic Modulus (Calc. ACI 318)	GPA(ksi × 10^3)	26 (3.70)	24 (3.49)	20 (2.89)	17 (2.42)	15 (2.17)	17 (2.48)
E (Test) / E (Calc. ACI 318)		1.08	1.06	0.61	0.81	0.81	0.73
Physical Properties @ 29 Days							
Compressive Strength	MPA (ksi)	39 (5.60)	41 (5.89)	41 (5.91)	42 (6.12)	42 (6.12)	47 (6.85)
Elastic Modulus (Test)	GPa (ksi × 10^3)	30 (4.28)	28 (4.09)	26 (3.81)	24 (3.25)	22 (3.25)	27 (3.96)
Elastic Modulus (Calc. ACI 318)	GPA(ksi × 10^3)	31 (4.49)	28 (4.10)	29 (4.17)	20 (2.92)	20 (2.92)	31 (4.50)
E (Test) / E (Calc. ACI 318)		1.05	1.00	1.09	0.89	0.9	1.14
Tensile Split Strength @ 29 Days	MPa (psi)	4.0 (580)	4.2 (615)	3.7 (531)	3.4 (492)	3.4 (498)	3.5 (504)

Note: 1. All concrete mixtures contain 446 kg/m^3 (752 pcy) cement, 708 kg/m^3 (1190 pcy) Natural Sand.
2. All concrete mixtures, Air 3.5 ± 0.5 %, Slump 100 mm (4 in.)
3. Mortar Mixture “M” contains 716 kg/m^3 (1208 pcy) Cement, 1050 kg/m^3 (1770 pcy) Natural Sand, Air 5.5 %, Slump 140 mm (5.5 in.)
4. All strength and modulus tests conducted on 152 × 304 mm (6 in. × 12 in.) cylinders.

bers and offshore applications. Lightweight aggregate concrete will demonstrate a strength "ceiling" where further additions of cementitious materials will not significantly raise the maximum attainable strength. Strength ceilings differ for each lightweight aggregate source and are the result of pore size and distribution as well as the strength characteristics of the vitreous material surrounding the pores. The strength ceiling of a particular lightweight aggregate may be increased by reduction of the top size in a particular grading formulation.

Tensile Strength

Shear, torsion, development length, bond strengths, and crack resistance are related to tensile strength, which is, in turn, dependent upon tensile strength of the coarse aggregate particle and the mortar and the degree to which the two phases are securely bonded. Traditionally, tensile strength has been defined as a function of compressive strength, but this is known to be only a first approximation that does not reflect aggregate particle strength, surface characteristics, nor the concrete's moisture content and distribution. The splitting tensile strength, as determined by ASTM Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens (C 496), is used throughout North America as a simple, practical design criterion that is known to be a more reliable indicator of tensile-related properties than beam flexural tests. Splitting tests are conducted by applying diametrically opposite compressive line loads to a concrete cylinder laid horizontally in a testing machine. A minimum tensile splitting strength of 2.0 MPa (290 psi) is a requirement for structural lightweight aggregates conforming to the requirements of ASTM C 330. As tensile splitting results vary for different combinations of materials, the specifier should consult with the aggregate suppliers for laboratory-developed splitting strength data. Tensile splitting strength test data should be developed prior to the start of special projects where development of early-age tensile-related forces occur, as in handling precast or tilt-up members.

Tensile splitting strength tests on structural lightweight concrete specimens that are allowed to dry correlate better with the behavior of concrete in actual structures. Moisture loss progressing slowly into the interior of concrete members will result in the development of outer envelope tensile stresses that balance the compressive stresses in the still-moist interior zones. ASTM C 496 requires a 7-day moist and 21-day laboratory air drying at 23°C (73.4°F) and 50 % relative humidity prior to conducting splitting tests. Structural lightweight concrete splitting-tensile strengths vary from approximately 75 to 100 % of normal-weight concretes of equal compressive strength. Replacing lightweight fine aggregate with normal-weight fine aggregate will normally increase tensile strength.

Modulus of Elasticity

The modulus of elasticity of concrete is a function of the modulus of each constituent (mortar, lightweight, and normal-weight aggregates) and their relative mixture proportion. The modulus of normal-weight concretes is higher because the moduli of the natural aggregate particles are greater than those of lightweight aggregate particles. For practical design conditions, the modulus of elasticity of concretes with densities between 1400 to 2500 kg/m^3 (90 to 155 lb/ft^3) and within normal strength ranges may be assumed to follow the ACI 318 formula

$$E = 33 w_c^{1,5} \sqrt{f_c'} \qquad E = 0.04 \sqrt{w_c^3 f_c'}$$

where

E = the secant modulus in MPa (psi),
w_c = the density in kg/m^3 (lb/ft^3), and
f_c = the compressive strength in MPa (psi) of a 150 by 300 mm (6 by 12 in.) cylinder.

This or any other formula should be considered as only a first approximation, as the modulus is significantly affected (±25 %) by binder characteristics, moisture, aggregate type, and other variables. The formula generally overestimates the modulus for high-strength lightweight concretes. When design conditions require accurate elastic modulus data, laboratory tests should be conducted on specific concretes proposed for the project according to the procedures of ASTM Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression (C 469).

Shrinkage

As with ordinary concretes, shrinkage of structural lightweight concretes is principally determined by

(a) shrinkage characteristics of the cement paste fraction,
(b) internal restraint provided by the aggregate fraction,
(c) the relative absolute volume fractions occupied by the shrinkage medium (cement paste fraction) and the restraining skeletal structure (aggregate fraction), and
(d) humidity and temperature environments.

Aggregate characteristics influence the quantity of cementitious materials (the shrinking fraction) necessary to produce a required strength at a given water content. Particle strength, shape, and grading influence water demand and directly determine the fractional volume and quality of the cement paste necessary to meet specified strength levels. When structural lightweight aggregate concretes are proportioned with cementitious material quantities similar to that required for normal aggregate concretes, the shrinkage of lightweight concrete is generally, but not always, slightly greater than that of normal-weight concrete due to the lower aggregate stiffness. The time rate of shrinkage strain development in structural lightweight concrete is slower, and the time required to reach a plateau of equilibrium is longer when the as-batched, lightweight-aggregate absorbed moisture is high [10].

Creep

Time-related increases in concrete strain due to sustained stress can be measured according to the procedures of ASTM Test Method for Creep of Concrete in Compression (C 512). Creep and shrinkage characteristics of any concrete type are principally influenced by water and cementitious materials (paste volume fraction), aggregate characteristics, age at time of loading, type of curing, and applied stress/strength ratio. As creep and shrinkage strains will cause an increase in long-time deflections, loss of prestress, a reduction in stress concentration, and changes in camber, it is essential for design engineers to have an accurate assessment of these time-related characteristics as a necessary design input. ACI 213 [10] demonstrates wide envelopes of one-year specific creep values for all lightweight, normally cured concretes. Test results for higher-strength, steam-cured concretes with a blend of lightweight and normal-weight aggregates have a range of values that narrows significantly and closely envelopes the performance of the normal-weight "reference" concrete. These values are principally based upon the results of the comprehensive testing program of Shideler [12]. Long-term investigations by Troxell [18] on normal-weight concretes report similarly wide envelopes of results for differing natural aggregate types, so comparisons with "reference" concretes should be based upon data specific to the concretes considered.

Durability

Numerous accelerated freezing and thawing testing programs conducted on structural lightweight concrete studying the influence of air-void system, cement content, aggregate moisture content, specimen drying times, and testing environment have arrived at similar conclusions: air-entrained lightweight concretes proportioned with proper air-void systems provide good durability results. Observations of the resistance to deterioration in the presence of deicing salts on mature bridges indicate similar performance between structural lightweight and normal-weight concretes [19]. Several comprehensive investigations into the long-term weathering performance of bridge decks [20] and marine structures [21] exposed for many years to severe environments support the findings of laboratory investigations and demonstrate that properly proportioned and placed lightweight concretes perform equal to or better than normal-weight concretes.

Core samples taken from hulls of 70-year-old lightweight concrete ships as well as 40-year-old lightweight concrete bridges have demonstrated concretes with a high integrity contact zone between aggregate/matrix with low levels of microcracking [5]. This proven record of high resistance to weathering and corrosion is due to physical and chemical mechanisms that include: (a) a higher resistance to macrocracking; (b) superior aggregate/matrix adhesion; and (c) the reduction of internal stresses due to elastic matching of coarse aggregate and the cementitious matrix. Micro-cracking is mitigated by the high ultimate strain capacity provided when concretes have a high strength/modulus ratio. The ratio at which the disruptive dilation of concrete starts is higher for lightweight concrete than for equal strength normal-weight concrete. Near-surface pores provided by the lightweight fine aggregates have been shown to accommodate etringite [15].

Long-term pozzolanic action developed at the surface of the pyroprocessed silica/alumina-rich lightweight aggregate will combine with the calcium hydroxide liberated during cement hydration. This will reduce overall concrete permeability, minimize leaching of soluble compounds, and further improve the integrity of the contact zone.

It is widely recognized that while ASTM Test Method for Resistance of Concrete to Rapid Freezing and Thawing (C 666) provides a useful comparative testing procedure, there remains an inadequate correlation between the results of this accelerated laboratory test and the observed behavior of mature concretes exposed to natural freezing and thawing. When lightweight concrete is tested, ASTM C 330 "Standard Specification for Lightweight Aggregates for Structural Concrete" requires modification of the procedures of ASTM C 666 to provide 14 days of drying in laboratory air after 14 days of moist curing. Durability of any concrete, both normal-weight and lightweight, is significantly influenced by the resistance to macro and micro-cracking. It is imperative that permeability and strain capacity characteristics of the concrete be sufficient to protect reinforcing steel from corrosion, which is clearly the dominant form of structural deterioration observed in current construction.

The matrix protective quality of insulating type concretes proportioned for thermal resistance by using high volumes of entrained air and low cement contents will be significantly reduced. Very low density, non-structural concretes will not provide adequate resistance to the intrusion of chlorides and carbonation, etc. [21].

Field Tests

For more than 25 years, field exposure testing programs have been conducted by the Canadian Department of Minerals, Energy and Technology (CANMET) on various types of concretes exposed to the extremely harsh marine environment at the Treat Island Severe Weather Exposure Station maintained by the U.S. Army Corps of Engineers at Eastport, Maine. Concrete specimens placed on a mid-tide wharf experience alternating conditions of seawater immersion followed by cold air exposure at low tide. In typical winters, the specimens experience over 100 cycles of freezing and thawing. Starting in 1980, five sets of specimens incorporating lightweight aggregate have been placed at this site. Kondratova listed several websites that could be used to examine the performance of these specimens, and reported that the deterioration rate was similar for the lightweight and normal-weight concretes. After more than 25 years of exposure to a severe marine environment, properly proportioned concretes of both types were providing durable performance [22].

Contact Zone

The expression "contact zone" includes two distinctively different phenomena: (1) the mechanical adhesion of the cementitious matrix to the surface of the aggregate, and (2) the variation of physical and chemical characteristics of the transition layer of the cementitious matrix close to the aggregate particle. Collapse of the structural integrity of the concrete conglomerate may come from the failure of either the aggregate or cementitious matrix, or from a breakdown in the contact zone causing a separation of the still-intact phases. The various mechanisms that act to maintain continuity, or that cause separation, have not received the same attention as has the air void system necessary to protect the matrix. Aggregates are frequently dismissed as being inert fillers and, as a result, they and the associated contact zone have not received adequate attention.

In order that concrete performs satisfactorily in severe exposure conditions, it is essential that a good bond develop and be maintained between the aggregate and the enveloping mortar matrix. A high incidence of interfacial cracking or aggregate debonding will have a serious effect on durability if these cracks fill with water and subsequently freeze. Deterioration will result, with pieces of apparently sound mortar separating from the bottom of the aggregate, usually with some of the mortar remaining firmly attached to the top side of the aggregate. An equally serious consequence of microcracking is the easy path provided for the ingress for aggressive agents into the mass of the concrete, rendering ineffective the protective layer of concrete over the reinforcing steel. The morphology and distribution of chemical elements at the transition layer in a number of mature structures that have withstood severe exposure were examined and reported by Bremner et al. [23].

The contact zone of lightweight aggregate concrete has been demonstrated to be significantly superior to that of normal-weight concretes that do not contain supplementary cementitious meterial [19,24]. This profound improvement in the quality, integrity, and microstructure stems from a number of characteristics unique to lightweight concrete that includes:

- The pozzolanic alumina/silicate surface of the fired ceramic aggregate combines with $CaOH_2$ liberated by hydration of the portland cement.
- Reduced micro-cracking in the contact zone because of the elastic similarity of the aggregate and the surrounding cementitious matrix [25].
- Hygral equilibrium between two porous materials (lightweight aggregate and porous cementitious matrix) as opposed to the usual condition with normal-weight aggregate, where bleed-water lenses form around essentially nonabsorbent coarse natural aggregates. These lenses have water-to-cementitious materials ratios significantly higher than in the rest of the matrix. When supplementary cementitious materials are added, the high-quality microstructure of the contact zone around lightweight aggregate is moderately enhanced. However, when supplementary cementitious materials are used in concretes containing normal-weight aggregate, this zone of weakness is profoundly improved.

While the reduction in compressive and tensile strength due to poor contact zone is important, the significance of increasing permeability is even greater. Increasing permeability inevitably leads to penetration of aggressive agents that accelerate corrosion of embedded reinforcement. The permeability of concrete is greater than the permeability of its constituents. This increase in permeability results from interfacial flaws at the aggregate surface linking up with micro-cracking in the transition layer.

The phenomenon of bleed water collecting and being entrapped under coarse particles of lightweight aggregate is mitigated if not eliminated. This has been verified in practice by the examination of the contact zone of lightweight concrete tensile splitting cylinders, as well as by visual examination of sandblasted vertical surfaces of building structures. This observation should not be surprising because, with structural lightweight concrete, the aggregate/matrix interface is a boundary between two porous media, while with normal-weight concrete there is an abrupt transition at the dense aggregate/porous cementitious matrix interface.

Implication of Contact Zone on Failure Mechanisms

Exposed concrete must endure the superposition of dynamic forces including variable live loads, rapid temperature changes, moisture gradients, and dilation due to chemical changes. These factors cause a predominantly tensile-related failure. Yet, the uniaxial compressive strength is traditionally considered the preeminent single index of quality, despite the fact that inadequate concrete performance is seldom related to this parameter. The simplicity and ease of compression testing has diverted our focus from life-cycle performance and the development of appropriate measurement techniques that quantify durable concrete characteristics.

In general, weakest link mechanisms are undetected in uniaxial compression tests due to concrete's forgiving load-sharing characteristics in compression, because of localized yielding and the closure of temperature and volume-change cracks. Weakest link mechanisms, however, are decisive in tensile failures in both dynamic and durability exposure conditions. In most concretes the limiting factor in the long-term performance is the integrity of contact zone.

Additionally, a full comprehension has not been developed regarding the accommodation mechanism by which the pores closest to the aggregate/matrix interface provide an accessible space for products that cause deleterious expansion. While research has identified ettringite, alkali-silica gel, marine salts, and corrosion products in these near-surface pores, it is still not fully understood how these products impact service life.

Resistance to Alkali-Aggregate Deleterious Expansion

ACI 201 "Guide to Durable Concrete" reports no documented instance of in-service distress caused by alkali reactions with lightweight aggregate [26]. No evidence of alkali-lightweight aggregate distress was observed in tests conducted on samples from a 70-year-old marine structure and several lightweight concrete bridge decks that were more than 30 years old [27]. The pyroprocessing of the aggregates tends to activate the particles' surfaces such that they act as a source of silica to react with the alkalis from the cement at an early age to counteract any potential long-term disruptive expansion. As maintained earlier, another factor that enables lightweight aggregate to reduce disruptive expansion is the availability of space within the expanded aggregate for reactive material to precipitate in a benign manner. Precipitation of alkali-rich material in the pores of an expanded aggregate was observed in concrete made with a well-known reactive normal-weight coarse aggregate in which some of the nonreactive fine aggregates were replaced with lightweight fine aggregates [28].

Though laboratory studies and field experience have indicated no deleterious expansion resulting from the reaction between the alkalis in the cement and the lightweight aggregates, the natural aggregate portion of a sand-lightweight concrete mixture should be evaluated in accordance with applicable ASTM standards.

Many lightweight concrete mixtures designed for an equilibrium density in the range of 110 lb/ft^3 (1760 kg/m^3) and above are produced using either natural sand or a naturally occurring coarse aggregate. In either case, these natural aggregates should be considered a potential source to develop alkali-aggregate reaction until they have been demonstrated by an appropriate ASTM test procedure or by having an established service history to be of negligible effect.

Abrasion Resistance

Abrasion resistance of concrete depends on strength, hardness, and toughness characteristics of the cement matrix and the aggregates, as well as on the bond between these two phases. Most lightweight aggregates suitable for structural concretes are composed of solidified vitreous ceramic comparable to quartz on the Mohs Scale of Hardness. Structural lightweight concrete bridge decks that have been subjected to more than 100 million vehicle crossings, including truck traffic, show wearing performance similar to that of normal-weight concretes. Hoff [29] reported that specially developed testing procedures that measured ice abrasion of concrete exposed to arctic conditions demonstrated essentially similar performance for lightweight and normal-weight concretes.

Fire Resistance

When tested according to the procedures of ASTM Method for Fire Tests of Building Construction and Materials (E 119), structural lightweight aggregate concrete slabs, walls, and beams have demonstrated greater fire endurance periods than equivalent-thickness members made with normal-weight aggregates (Fig. 6). Superior performance is due to a combination of lower thermal conductivity (lower temperature rise on unexposed surfaces), lower coefficient of thermal expansion (lower forces developed under restraint), and the inherent thermal stability developed by aggregates that have been already exposed to temperatures greater than 1093°C (2000°F) during pyroprocessing.

Specifications

Specifications for structural lightweight concrete usually require minimum values for compressive and tensile splitting strength, maximum limitations on slump, specified range of air content, and a limitation on maximum fresh density. The density of lightweight concrete depends primarily on the relative density factor of the lightweight aggregates, and it is also influenced to a lesser degree by cementitious materials, water, air contents, and proportions of coarse-to-fine aggregate.

Conclusion

Structural lightweight concrete is a unique construction material that should be specified, designed, and constructed in a manner that recognizes and takes advantage of its unique physical and mechanical properties (ASTM C 330, C 496, C 567).

Acknowledgments

The principal sources of information for this chapter include the *Guide for Structural Lightweight Aggregate Concrete* (ACI 213) [14], ACI 318 Building Code Requirements for Reinforced Concrete [1], and the "State-of-the-Art Report on High-Strength High-Durability, Structural Low Density Concrete for Applications in Severe Marine Environments" [15].

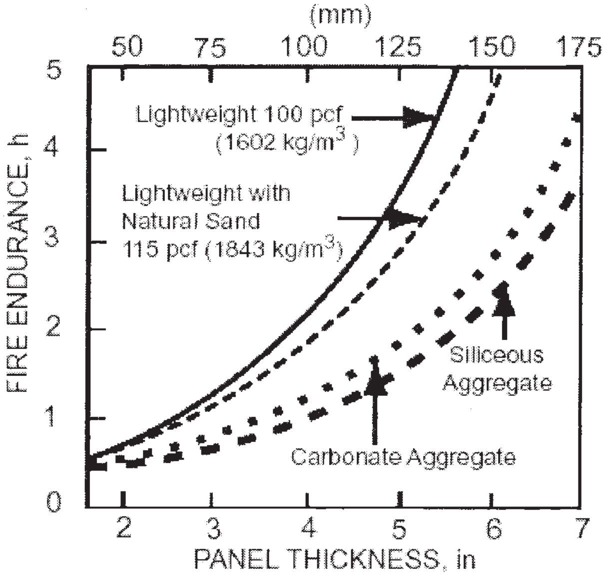


Fig. 6—Fire endurance (heat transmission) of concrete slabs as a function of thickness for naturally dried specimens [10].

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Cellular Concrete

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Preface

THE CHAPTER ON CELLULAR CONCRETE WAS FIRST presented in *ASTM STP 169B* in 1978 and was authored by Professor Leo M. Legatski [1]. In *ASTM STP 169C* in 1994, the chapter was authored by his son Leo A. Legatski [2] who updated various sections and stressed new applications of the material. The author acknowledges both father and son for their earlier contributions as their work provides the background for this edition. In the interest of consistency, some of the fundamental aspects of the earlier publications are retained in this chapter. The current edition updates the topics, introduces new information on the material properties and applications of cellular concrete, and includes up-to-date references.

Introduction

Cellular concrete is a low-density material having a homogeneous void or cell structure formed by the addition of a prepared foam or by the generation of gas within the fresh cementitious mixture. It is usually cast in densities ranging from 320 to 1920 kg/m^3 (20–120 lb/ft^3). Density control is achieved by adding a calculated amount of air as a preformed foam to a cementitious slurry with or without the addition of sand or other materials. The air cells created by the preformed foam may account for up to 80 % of the total volume.

Reducing the mixture density by the addition of preformed foam is accompanied by a reduction in strength properties and thermal conductivity. Normally, it is possible to select a reduced concrete density to satisfy strength requirements and provide increased insulating value.

As a result of the reduced strength properties, typical applications for cellular concrete are usually concrete applications that are required to resist modest loads over relatively small spans. Examples of nonstructural cellular concrete applications include: floor fills, sloping roof screeds, filling voids to support slabs and roadways, and reducing lateral loads on wall structures.

The purpose of this chapter is to provide an overview of the production, physical properties, and common applications of cellular concrete.

Background

Cellular concrete is a lightweight concrete, consisting of a system of macroscopic air cells uniformly distributed in either a cement slurry or a cement grout (containing aggregate). The cell size varies approximately from 0.10 to 1 mm (0.004–0.04 in.). The air cells must be tough and sufficiently stable in order to withstand the rigors of mixing and placing as the air cells are separated, coated with cement paste, and the concrete is pumped or otherwise transported to the point of placement.

In general, there are two basic methods for introducing the air cells (bubbles) into the plastic mixture: through blending foam (or a foaming agent) in the mix or by generating a gas in the fresh mix by a chemical reaction [3,4]. Hence, the terms "foamed" and "gas concrete," respectively, which are sometimes used in referring to the process used to generate the void system. In the foaming process, a stable preformed foam is added to the cementitious slurry during mixing in an ordinary mixer. It is also possible to form air cells in the slurry by vigorous mixing of the slurry with a foaming additive in a high-speed mixer. In the high-speed mixing method, the volume of air cells produced depends on the amount and properties of the foam concentrate, the mixing time, and the temperature of the water and other materials. Consequently, batch density control of this process is difficult at best.

The other method for forming air cells is through a chemical reaction, which generates gas in the slurry (mortar). The bubbles maintain their shape through the setting stage and become discrete air cells in the cementitious matrix. Typically, small amounts of finely divided aluminum powder or aluminum paste are added to the mix, which react with the soluble alkalis in the cement slurry liberating hydrogen gas and forming bubbles. This method of producing air cells is best suited for factory production in the manufacture of autoclaved aerated concrete (AAC) structural elements. High-pressure steam curing is employed in this case, which provides improved strength and dimensional stability.

The American Concrete Institute (ACI) published three reports of its Committee 523 on Cellular Concrete [5–7]. Two of the reports address cast-in-place cellular concrete, and the third report is devoted to precast cellular concrete. These reports address cellular concretes of various densities and provide in depth information on the properties, mix design, and use of the material. Additional information on cellular concrete has also been presented in special publications by ACI and the Portland Cement Association [8,9]. This chapter does not address AAC, which is factory-produced using a chemical reaction process for air cell generation. It deals only with air cells produced using preformed foam, which is the process most commonly used for cast-in-place construction. Detailed technical information on AAC is available in the published literature [10–15].

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Classification of Cellular Concretes

Cellular concrete weighs substantially less than normal weight concrete. It covers a broad spectrum of concretes ranging in density from 320 to 1920 kg/m^3 (20–120 lb/ft^3). In densities not exceeding 800 kg/m^3 (50 lb/ft^3), cellular concrete, often referred to as insulating concrete or low-density concrete, has excellent insulation properties and is used primarily in nonstructural applications for thermal and sound insulation, roof decks, fill for slab-on-grade subbases, firewalls, and underground thermal conduit linings. It is typically made of neat cement mixtures with preformed foam, and the 28-day compressive strength is generally between 0.7 and 7 MPa (100 and 1000 psi).

In densities ranging from 800 to 900 kg/m^3 (50–120 lb/ft^3), cellular concrete is considered in a semistructural range, and applications vary according to density and strength. Typically, sand or lightweight aggregates are added to improve strength or impart special desired properties to the mix. For this group, the 28-day compressive strength ranges approximately from 3.4 to 17 MPa (500–2500 psi). Applications of the material range widely from nonstructural fill for thermal and sound insulation of floors and roofs at the lower densities to cast-in-place walls, floors, and roofs at the higher densities.

Based on density range and the components of the mixture, cellular concrete may be classified as follows:

Neat-Cement Cellular Concrete

Neat-cement cellular concrete consists of portland cement, water, and preformed foam. It contains no aggregates. Neat-cement cellular concrete is usually limited to the low-density range with an upper limit on cast density of about 800 kg/m^3 (50 lb/ft^3). This class of cellular concrete is referred to as low-density or insulating concrete. Substitution of pozzolanic materials for a portion of the cement lowers the actual cement content and permits lower densities of the neat-cement mixtures, while reducing the heat of hydration.

Sanded Cellular Concrete

Sanded cellular concrete is cellular concrete that contains fine aggregate (sand) in addition to cement, water, and preformed foam. These concretes are usually produced in cast densities of 800–1920 kg/m^3 (50–120 lb/ft^3). The mixture properties are primarily dependent on the cement content, the water-cement ratio, and the specific characteristics of the sand.

Lightweight Aggregate Cellular Concrete

Lightweight aggregate cellular concrete is similar in density to sanded cellular concrete but with low-density lightweight aggregate (such as perlite or vermiculite) replacing all or part of the sand. The lightweight aggregates may be of structural grade in order to increase the strength-density ratio [16]. The inclusion of such material is recommended if it is locally available as its procurement from afar often results in a higher cost of the final product.

Materials

In its basic form, cellular concrete is made by blending preformed foam into a cement slurry, and it may or may not contain aggregate materials. A brief description of the basic ingredients as well as other materials that may be used in cellular concrete is given herein.

Cement

Portland cement, portland blast furnace slag cement, or portland pozzolan cement conforming to the requirements of ASTM Standard Specification for Portland Cement (C 150), ASTM Standard Specification for Blended Hydraulic Cements (C 595), or ASTM Standard Performance Specification for Hydraulic Cement (C 1157) are used in cellular concrete. Blended cements may result in slower rates of strength development during the first 3 to 5 days. Higher rates of early strength development may be achieved by using high-early-strength portland cement (Type III).

Fine Aggregate

Natural or manufactured sand meeting the requirements of ASTM Standard Specification for Concrete Aggregates (C 33), ASTM Standard Specification for Aggregate for Masonry Mortar (C 144), ASTM Standard Specification for Lightweight Aggregates for Structural Concrete (C 330), or ASTM Standard Specification for Lightweight Aggregates for Concrete Masonry Units (C 331) may be used in cellular concrete. Sand of other gradation not conforming to these standards have also been used in some cases, provided tests or service records have shown that it produced cellular concrete of the desired quality.

Lightweight Coarse Aggregate

For cellular concrete incorporating lightweight coarse aggregates, ASTM Standard Specification for Lightweight Aggregate for Insulating Concrete (C 332) specifies two groups of lightweight aggregates. The classification into two groups is based on the expected concrete unit weight and thermal conductivity. Group I aggregates, which are prepared by heat processing to expand products such as perlite and vermiculite, are more commonly used in insulating cellular concrete as they will produce concrete with lower unit weight and thermal conductivity [17]. Group II aggregates, which are prepared by expanding, calcining, or sintering various natural or artificial materials such as slag, clay, shale, pumice, scoria, or tuff, are relatively heavier in weight and may be used in higher density cellular concrete where improved strength is desired.

Water

Water for mixing and curing cellular concrete should be potable and free of deleterious amounts of oils, acids, alkalis, salts, and organic materials that would adversely affect the set time, strength, or performance of the concrete.

Preformed Foam

Preformed foam is produced by blending a foam concentrate, water, and compressed air in predetermined proportions in a foam generator. The liquid expands in volume up to about 30 times, resulting in preformed foam with density approximately in the range of 32–56 kg/m^3 (2.0–3.5 lb/ft^3). Foam concentrates are typically based on hydrolyzed protein or synthetic detergents. The foam concentrate must have a chemical composition capable of producing stable air cells that remain intact during handling (mixing, pumping, and placing) and until the concrete hardens. ASTM Standard Method for Testing Foaming Agents for Use in Producing Cellular Concrete Using Preformed Foam (C 796) provides test procedures for evaluating foam concentrates in a standard cellular concrete mix for density, water absorption, and compressive strength. Performance specifications for evaluating foam concentrates in cellular concrete mixtures are given by ASTM Standard Specification for

Foaming Agents Used in Making Preformed Foam for Cellular Concrete (C 869).

Chemical Admixtures

For cellular concrete with cast density of 1440 kg/m^3 (90 lb/ft^3) or above, water reducing admixtures may be used to reduce the water to cement ratio in the mix and, hence, increase the compressive strength of the concrete. Accelerating admixtures may be used if early strength development is required. Chemical admixtures should conform to ASTM Standard Specification for Chemical Admixtures for Concrete (C 494) and should be used in accordance with manufacturer recommendations. Guidance for use of chemical admixtures is provided in this volume chapter on Chemical Admixtures. Compatibility of the chemical admixtures with the foaming agent or other ingredients in the mix should be established by trial batches.

Supplementary Cementitious Materials

Fly ash or natural pozzolans conforming to ASTM Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete (C 618) may be used in cellular concrete to the advantage. The supplementary cementitious material may be added as a filler or partial replacement for the portland cement. Substituting fly ash for a portion of the cement usually has reasonable cost savings without adversely affecting significant properties of the cellular concrete. Its pozzolonic properties improve flowability, increase compressive strength, reduce the heat of hydration, and reduce water permeability. Guidance for use of supplementary cementitious materials is provided in this volume chapter on Supplementary Cementitious Materials. Supplementary cementitious materials must be compatible with the foam concentrate and other ingredients in the mix, which can be established by trial test batches.

Fiber Reinforcement

Fiber reinforcement may be used in cellular concrete to help control shrinkage cracking. Various fibers used include steel, glass, polypropylene, polyester, nylon, or other special composites. The fiber quantity is a compromise based on the required workability of the concrete, the fiber's efficiency, and its cost. The fiber reinforcement may also improve the energy absorption and spall resistance properties of the cellular concrete. Research using monofilament polypropylene fibers in cellular concrete mixtures, with densities 560–624 kg/m^3 (35–39 lb/ft^3), demonstrated a ductile elasto-plastic load-deflection behavior when tested to failure in bending, compression, tension, and shear [18]. In this study, loading and unloading also remained elasto-plastic over a wide range of deformation and crack width.

A volume of fiber equal to about 0.5 % of the absolute volume of cement is often the starting point for trial mixtures. The upper limit depends upon the projected use of the concrete, its required workability, and cost. The proposed fiber length ranges from about 13 to 38 mm (0.50–1.5 in.). A lower fiber length limit is based on the fiber's ability to bond with the cement paste. The upper length limit depends on the increasing difficulty of dispersing fibers in the mixture as the length is increased. A length of 19 mm (0.75 in.) has been found to be very efficient for cellular concrete.

Polymers

Polymers such as latexes and acrylics may be used as additives in cellular concrete to improve certain strength characteristics or reduce permeability and absorptivity. Guidance on the use of polymers is provided in this volume chapter on Polymer-Modified Concrete and Mortar. Depending on its chemical structure, latex can set up an additional matrix within the normal cement structure and impart specialized properties to the material.

Mixture Proportioning

Density is a critical parameter in the mix proportioning of cellular concrete as it is a direct function of the compressive strength and thermal conductivity of the material. The first step in mix proportioning is the selection of the wet density of the cellular concrete, the cement content, and the water-cement ratio. The choice of these parameters is usually based on the strength and thermal conductivity requirements. Slurry or grout mixtures are proportioned using the absolute (solid) volume method. According to this approach, the sum of absolute volumes of cement, water, and aggregate (if used) for a unit volume of concrete (cubic meter or cubic yard) subtracted from the unit volume gives the volume of air required per unit volume of concrete [1,6,19].

Three foam gun calibration factors are necessary for mix proportioning. These factors, which can be obtained from a calibration test, are the foam/air volume ratio (usually about 1.05 to 1.07), the foam density (32–56 kg/m^3 (2.0–3.5 lb/ft^3)), and the foam volume output per unit of time. It should be pointed out that the weight of water calculated from the cement content and water-cement ratio should be reduced by the weight of the foam volume (which is considered as water).

Slurry Mixtures

Slurry mixtures are low-density mixtures without aggregates. Proportioning a slurry mixture involves selecting a density and then selecting a water-cement ratio that is compatible for both that density and the specific application. The water-cement ratio can be within a reasonable range for a selected density.

If the water-cement ratio is too low, the cement will seek water from the preformed foam resulting in a loss of volume (yield) and an increase in density. Too high a water-cement ratio results in excessive water bleeding and a lower compressive strength concrete mixture.

Grout Mixtures

Grout mixtures are slurry mixtures with aggregates. Their proportioning involves selecting a cement content and a water-cement ratio for a specific density. The selection of these two variables will result in a cellular concrete of a given strength based on the characteristics of the ingredients (cement and aggregate). If different properties such as higher strength are required, the density, cement content, and water-cement ratio may be varied according to the designer's experience. An example of the proportioning of grout mixtures is given in ACI 523.3R [6].

Batching, Mixing, and Application Techniques

Accurate batching of ingredients is more critical with cellular concrete mixtures than with regular concrete. Because a significant portion of the material volume (25–80 %) is air, the other materials must be batched accurately. For these mixtures, the cement and sand (or other aggregate) are usually weighed into the batch. The mixture water is metered while the preformed foam is time injected into the mixture through a calibrated nozzle.

The mixing technique should be compatible with the type of mixture, its ingredients, the job requirements, and the method of placement. Mixing should efficiently mix the cement and water and properly blend them with the other ingredients including the preformed foam. Paddle, high shear, continuous, and rotary drum mixers may all be acceptable for specific applications depending on the quality requirements of the final product.

Cellular concrete mixtures are typically job-site produced and placed. If ready-mix trucks are used for sanded mixtures (floor-fill applications), the grout is delivered to the job site and the preformed foam is added just prior to placement. This maintains the quality and freshness of the material. The rotary drum action of a ready-mix truck is acceptable for sanded mixtures with densities greater than 800 kg/m^3 (50 lb/ft^3).

For low-density applications (roof deck and engineered fill) with neat-cement slurries at densities less than 800 kg/m^3 (50 lb/ft^3), rotary drum mixing action is not ideal. Instead, paddle type or shear mixers are common methods for both batch and continuous mixing procedures. After the cement/water slurry is produced in these mixers, the preformed foam is added and blended prior to or during placement with a positive displacement pump. As the mixture is pumped, density is measured at the point of placement for quality control. Mix adjustments can then be made to account for pumping distances and other special application conditions.

Pumping is the most common method of placement, but other methods can be used. Positive displacement pumps such as Moyno or peristaltic pumps are used for low-density mixtures. Although piston pumps are efficient for grout mixtures at densities greater than 1440 kg/m^3 (90 lb/ft^3), they do not efficiently pump low-density mixtures.

Casting techniques are different for each type of cellular concrete application. The thinner floor-fill mixtures utilize a rolling screed to provide a constant thickness. Since roof deck applications are cast slope-to-drain, string lines provide guides for casting and darby finishing the material by experienced tradesmen. Screed rails may also be used. Finishing operations, in general, should be kept to a minimum; smoothing with a darby or bullfloat is usually sufficient [20].

Geotechnical fill applications have the greatest variation in casting techniques. Generally, these fills are several meters (feet) thick so that they are cast in lifts of up to 1.0 m (3.3 ft) thick based on the available area to be cast. The succeeding lifts are cast on a daily basis until the final fill profile is reached.

Physical Properties

Several studies investigated the physical and mechanical properties of cellular concrete cast at different densities and with or without aggregates in the mix [1,18–21]. Because the density of cellular concrete may be varied over a wide range, 320–1920 kg/m^3 (20–120 lb/ft^3), it is considered as an additional variable that significantly impacts the physical properties and the mix design of the material. The lower density cellular concrete has lower thermal conductivity (higher insulation), accompanied by lighter weight and reduced strength. As the density increases, the thermal conductivity increases, but this is compensated for by improvements in strength properties. Nevertheless, the ability to control the density of the material over a wide range suitable for various applications is the major contribution of cellular concrete.

Density

When referring to the density of cellular concrete, confusion may be avoided by stating the moisture condition of the material at that specific density. Significant moisture conditions include as-cast density (wet density or plastic concrete density), air-dry density (at a stated age and curing condition), and the oven-dry density.

The as-cast or wet density is usually determined at the point of placement in accordance with ASTM C 796. In determining the wet density, the concrete should be consolidated by tapping the sides of the container and not by rodding. The ratio of the wet density to oven-dry density for the different cellular concrete mixtures varies due to the different water content requirements. The wet density of the cellular concrete is an important job-site quality assurance tool to control uniformity of the mixtures.

The air-dry density of cellular concrete usually represents the condition of the in-place material. The change in density due to air drying is a function of temperature, duration of the drying period, humidity, the wet density of the concrete, the water-cement ratio, and the surface-area ratio of the element. Although the relationship between air-dry density and wet density seems complicated, the air-dry density of cellular concrete is usually about 80 kg/m^3 (5 lb/ft^3) less than its wet density. Cellular concrete cast, cured, and air dried under job conditions in low-humidity environments may have density losses approaching 160 kg/m^3 (10 lb/ft^3).

Oven-dry density is commonly used to relate the physical properties of various types of cellular concretes, and for the determination of the thermal conductivity by the guarded hot plate method in accordance with ASTM Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus (C 177). For the latter purpose, the oven-dry density may be calculated with sufficient accuracy from the mixture data by assuming that the water required for hydration of the cement is 20 % of the weight of the cement. The oven-dry density (D) is calculated as follows:

$$D = [1.2\ C + A]\ \text{kg/m}^3 \text{ or } [(1.2\ C + A)/27]\ \text{lb/ft}^3$$

where

C = weight of cement, kg/m^3 (lb/yd^3) of concrete; and
A = weight of aggregate, kg/m^3 (lb/yd^3) of concrete.

Workability

Cellular concrete in the low-density range (less than 800 kg/m^3 (50 lb/ft^3)) is a flowable material with excellent workability. As a result, it is handled as a liquid and poured or pumped into place without the need for consolidation. It should be pointed out that the slump test, which is used to measure the consistency in normal weight concrete, is meaningless in the case of cellular concrete since the material is placed in fluid consistency.

Thermal Conductivity

The thermal conductivity (k) of a material is the time rate of transfer of heat by conduction, through a unit thickness, across a unit area for a unit difference of temperature. The units of k are watts/meter-Kelvin or W/mK (BTU in./h ft^2 °F). The thermal conductivity of cellular concrete is primarily a function of its oven-dry density and is measured by means of the Guarded Hot Plate (ASTM C 177). Detailed studies were conducted to evaluate the thermal properties of cellular concretes of various

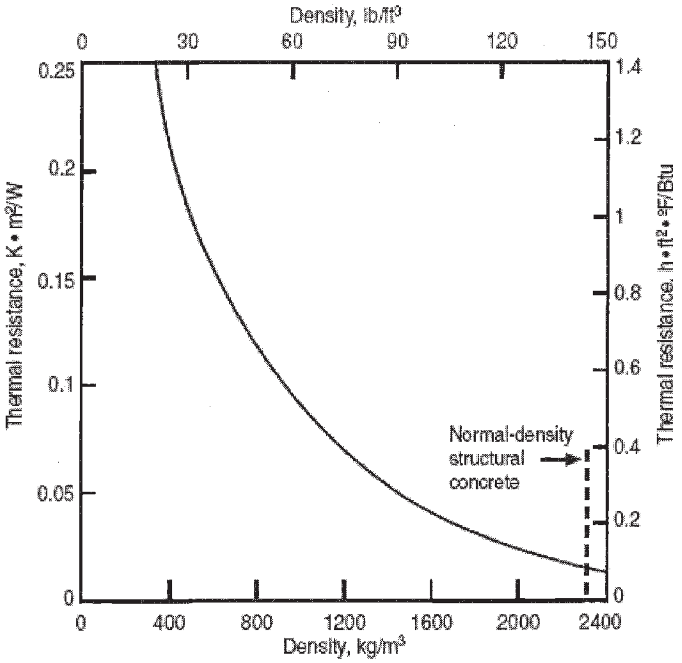

Fig. 1—Relationship between thermal conductivity and oven-dry density for cellular concretes over the entire density range [9,17].

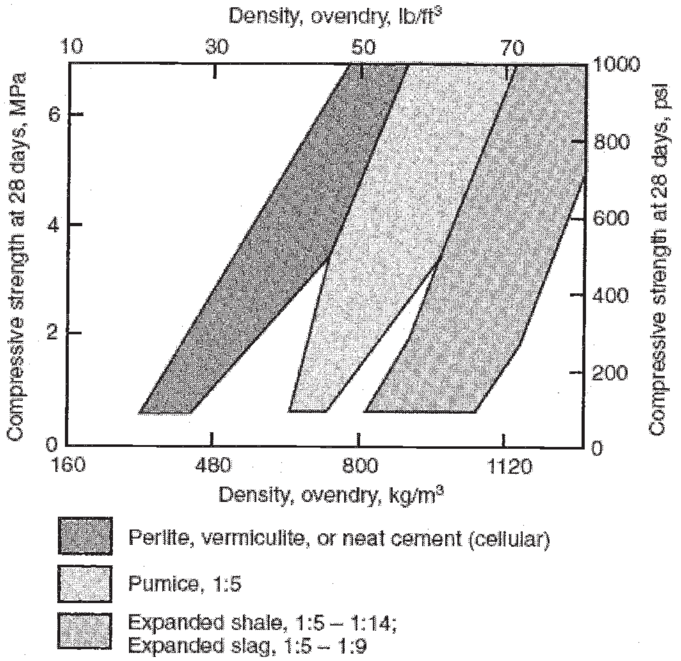

Fig. 2—Approximate relationships between density and compressive strength for different types ofcellular concretes [9].

low densities [22]. Figure 1 shows the relationship of thermal conductivity to oven-dry density for cellular concretes over the entire density range [9,17]. The thermal conductivity increases with increase in moisture content and density, with approximately 5 % increase in thermal conductivity for each percent increase in density due to free moisture.

Compressive Strength

The principal factors affecting the compressive strength of cellular concrete include cast density, cement content, water-cement ratio, aggregate type and amount, special admixtures, and curing conditions. Figure 2 shows approximate relationships between density and compressive strength for different types of cellular concretes, and Fig. 3 shows the relationship for sanded cellular concrete mixtures at different water-cement ratios. Similar curves can be developed for different cement factors, water-cement ratios, and various ingredients or admixtures. It should be pointed out that the compressive strength of cellular concrete depends on both the water-cement ratio and the air-cement ratio. The compressive strength can increase when the water-cement ratio increases, as long as there is a more significant reduction in the air-cement ratio [19,23,24]. The compressive strength of neat-cement cellular concrete was predicted by Hoff [25]. Other studies also attempted to develop relationships between the strength and mix proportions of cellular concrete [26,27].

The compressive strength for cellular concrete having an oven-dry density not exceeding 800 kg/m³ (50 lb/ft³) should be determined in accordance with ASTM Standard Test Method for Compressive Strength of Lightweight Insulating Concrete (C 495). For oven-dry densities greater than 800 kg/m³ (50 lb/ft³), it is recommended to determine the compressive strength in accordance with ASTM Standard Specification for Lightweight Aggregates for Structural Concrete (C 330), with the exception that the sides of the cylindrical mold shall be tapped with a rubber hammer while the mold is being filled. Using a vibrating table to vibrate the molds lightly may also be used in preparing the test specimens [6].

Tensile Strength

The tensile strength of cellular concrete bears a similar relationship to the compressive strength as with normal weight concrete. Tensile strength is typically 10–15 % of the compressive strength. Since low-density cellular concretes have very low tensile strengths, adding fiber to increase the tensile strength is beneficial and usually cost-effective for specific applications. The splitting tensile strength is determined in accordance with ASTM Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens (C 496).

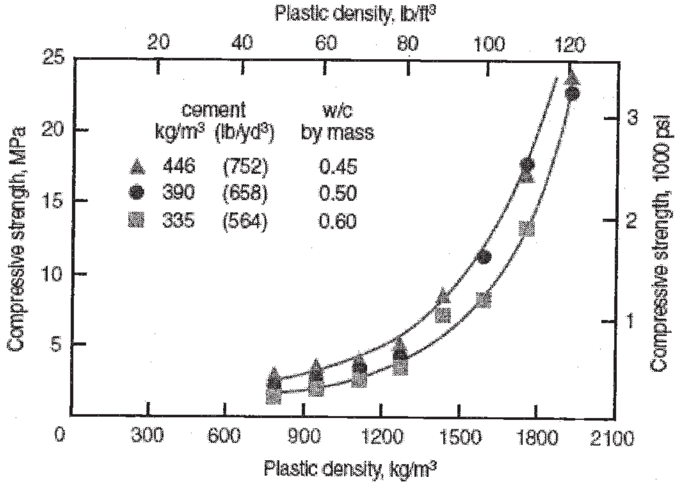

Fig. 3—Compressive strength versus density for sanded cellular concrete mixtures at different water-cement ratios [9].

Shear Strength

Very limited data are available on the shear strength of cellular concrete. Tests of cellular concrete beams with cast density ranging from 800 to 1440 kg/m^3 (90–120 lb/ft^3) have indicated that the shear strength of such mixes may be estimated using the ACI Code requirement for lightweight concrete [1]. Direct shear tests of cellular concrete reinforced with polypropylene fiber were also reported [18].

Modulus of Elasticity

The modulus of elasticity (E_c) of cellular concrete is a function of its density and compressive strength. It is reasonable that cellular concrete has a lower modulus of elasticity than concrete of the same density, but made with lightweight aggregate, because the cellular concrete has a lower compressive strength.

In a laboratory study [1] of modulus of elasticity of cellular concretes whose wet density varied from 1280 to 1872 kg/m^3 (80–117 lb/ft^3), the following equation was selected to represent best the test results

$$E_c = W^{1.5} \cdot (37.04) \cdot \sqrt{f'_c}\,(N/m^2)$$

or

$$E_c = (W^{1.5}) \cdot (28.6) \cdot \sqrt{f'_c}\,(lb/in.^2)$$

where

W = (wet density − 80) kg/m^3 or (cast density − 5) lb/ft^3, and
f'_c = 28-day compressive strength, MPa (lb/in.^{2}).

It was also reported [6,19] that the ACI 318 Code equation for the modulus of elasticity was applicable for cellular concretes ranging in density from 368 to 1872 kg/m^3 (23–117 lb/ft^3).

Water Absorption

Cellular concretes have considerably higher water absorption values than normal weight concretes, primarily due to the lower density of cellular concrete. The consequence of water absorption is a higher density. The amount of water absorbed by cellular concrete varies not only with the density of the material but also with the quality of the mixture ingredients—specifically, the cement, supplementary cementitious materials (if used), and preformed foam concentrate. Some concentrates do not produce a discrete cell structure within the cement matrix. This may be related to the chemistry of the ingredients, their dilution rate, their method of generating foam, or their expansion pressure.

If a preformed foam is not able to withstand the rigors of batching, mixing, and placing (usually by pumping), the cell structures of the mixture will interconnect resulting in channels for high water absorption. Lower quality concentrates require more mixture water that evaporates during hardening and leaves more pore spaces. Supplementary cementitious materials may decrease the amount of water absorbed because their smaller particles fill available spaces between cement particles.

Water absorption may be measured in three different ways: 24-h immersion, long-term immersion (120 days), and by tide cycle. In each case, the specimen is weighed prior to testing. At each interval of testing, the sample is reweighed and compared to the initial condition. The increase in density is the water absorption and may be expressed as a percentage by volume or a percentage of the initial weight or density. Expressing water absorption as a percentage by volume more accurately reflects the effect of sample size, density, and the surface area to volume relationship.

Drying Shrinkage

The drying shrinkage of cellular concrete is not usually critical when it is used in roof deck insulation and fill applications. It should be considered, however, in structural applications. The drying shrinkage of moist-cured cellular concrete may vary from 0.1 to 0.6 %, depending on the composition of the mixture and whether or not aggregates are used.

Fire Resistance

Cellular concrete is incombustible and has excellent fire resistance properties as compared to normal weight or lightweight aggregate concrete. Fire tests conducted on concrete slabs made of different types of concretes demonstrated a superior performance for low-density cellular concrete [28–30]. Highest fire endurance ratings were achieved at the lower densities, and as the density increased, a decrease in the fire endurance resulted for each type of concrete. Fire tests were conducted in a manner very similar to ASTM Standard Methods of Fire Tests of Builiding Construction and Materials (E 119).

Freeze-Thaw Resistance

Cellular concrete is usually covered by roofing material such as hot mopped asphalt or pitch, and therefore not exposed directly to the elements. Nevertheless, cellular concrete has good freeze-thaw resistance because of its high air content and its relatively high-cement content. Although its water absorption is high, the rate of water penetration through cellular concrete is low as the larger pores will not fill by suction. In the case of direct exposure to severe freezing and thawing environments, cellular concrete surfaces must be treated for protection.

Energy Absorption

Low-density cellular concretes possess excellent deformation resistance properties because of their cellular structure and their ability to absorb loads and crush in a controlled manner. The absorbed energy is greater at lower densities that can withstand more deformation before “bridging and locking” of the material occurs. Zollo and Hays [18,31,32] studied the impact and energy absorption capabilities of fiber-reinforced cellular concrete panels. Their work showed that high energy rapid rates of loading produced only localized damage, which was attributed in part to the fibers and in part due to the low-density void structure of the cellular concrete matrix. Also, panels subjected to high energy impact using small caliber hand guns at close range showed that the bullets were captured within the panel after penetrating to approximately two-thirds of the depth.

Walkability

The ability of a low-density cellular concrete roof deck or floor to sustain normal construction foot traffic without damage is an important factor and often referred to as “walkability.” It can be judged by examining the surface distress such as footprints caused by normal foot traffic. Walkability improves with increased density. If heavy construction traffic (such as wheelbarrow, scaffolds, material storage, etc.) is expected, the surface of the concrete should be protected by wooden boards or other means.

Nailability and Sawability

Low-density cellular concrete can be sawn, and it holds nails. It can be worked like wood. Nailing is important in the case of attaching roofing to the cellular concrete deck. Nailing within seven days of concrete placement is desirable. In general, the lower the density, the better the nailability and sawability of the material [5,33].

Applications

Cellular concrete can be used in a wide variety of applications [8,23,34]. The vast number of applications is due to the ability to control the density of the material. Typical applications include: fills for thermal and sound insulation of floors, walls, and roofs; cast-in-place walls, floors, and roofs; precast wall and floor panels; and as a low-density engineered fill for use in a variety of geotechnical applications. Specialized applications such as protective structures for advanced military weapons, fragmentation shields, blast attenuating walls, and tunnel linings were discussed in detail by Hoff [35,36]. The U.S. Department of Energy reported on a new innovative void-filling technology using low-density cellular concrete [37]. The technology entails filling the voids of nuclear vessels with a fluid low-density cellular concrete, which would support the walls of the vessels and prevent them from collapsing, even if they degrade. Only a few of the most commonly used applications are discussed in this chapter.

Floor Fills

Floor fill mixtures are job-site produced from a sand/cement grout at 1600–1760 kg/m^3 (100–110 lb/ft^3) having a cement content of 256–298 kg/m^3 (564–658 lb/yd^3). Preformed foam is added to the sand/cement grout. This grout is pumped into place over wood-frame construction that is typically wood joists and a plywood subfloor. The floor fill is cast 3.8 cm (1.5 in.) thick, the dimension of a standard wood bottom plate. The purpose of floor fill is to provide fire resistance and sound attenuation characteristics to floor/ceiling systems.

Floor fill mixtures may also be cast on top of precast concrete units for leveling the camber of these members to provide a flatter floor. Floor fills are occasionally cast over corrugated steel decks 38–51 mm (1.5–2 in.) thick above the top of the flutes. Fire ratings exist for many of these constructions.

Cellular concrete floor fills are very effective in floor rehabilitation for the renovation of older buildings. It levels the floors of these structures while minimizing added dead load. For thick fills, lighter materials can occupy much of the volume and then be topped with the cellular concrete floor fill. In floor rehabilitation, cellular concrete floor fills are cast over a variety of floor substrates such as concrete, wood, terrazzo, wood sleepers and cinder fill, tile, etc. Each application must be analyzed on its individual requirements.

Roof Deck Fills

The largest single use of low-density cellular concrete is for insulating roof deck fills. The roof deck system over which the insulating cellular concrete is cast generally becomes a permanent part of the structure. Roof deck applications for new construction are typically cast over galvanized steel decking (corrugated or fluted) to provide improved fire ratings, additional seismic resistance, thermal insulation, and a slope-to-drain roof deck. The finished deck is then covered with a waterproofing membrane. Substrates such as concrete (precast or cast-in-place) and wood decking are also common for this application.

The typical mixture utilizes a predetermined quantity of foam added to a cement/water slurry resulting in a density of 480–640 kg/m^3 (30–40 lb/ft^3). Expanded polystyrene insulation board (EPS) is often included in a sandwich construction over the structural deck. This involves filling the steel deck flutes or otherwise casting the slurry on to the deck and laying the EPS board in the slurry so that it is “cemented” or bonded to the structural deck. It is then topped with the final cellular concrete roof deck fill resulting in a slope-to-drain surface ready for the membrane roofing.

Cellular concrete is an excellent material for reroofing applications. If the existing roofing is not removed, the existing structure must be checked for its ability to support additional load. The sloping roof deck usually incorporates EPS board sandwiched within the fill. If the existing roofing system is wet or damaged, those portions must be replaced or repaired prior to casting the cellular concrete. This solution provides positive drainage for “ponded roof decks” and protects and preserves the insulation system for future reroofing.

Standard roofing membranes are used over cellular concrete roof decks. These include built-up systems as well as single-ply membranes such as ethylene propylene diene monomer (EPDM), polyvinyl chloride (PVC), polyisobutylene (PIB), and modified bitumens. It is not the purpose of this chapter to discuss these roofing membrane systems further.

Engineered Fills

Cellular concretes provide alternate solutions to standard geotechnical procedures. In many cases, engineered fills can economically replace common geotechnical solutions such as piling, removal and replacement of poor soils, surcharging, and bridging over poor soils to name a few. In most applications load balancing or weight credit techniques are combined within the engineered fill solution. For example, to reduce the load over poor soils, it is advantageous to remove part of the existing soil, typically weighing 1920 kg/m^3 (120 lb/ft^3), and replace it with 480 kg/m^3 (30 lb/ft^3) cellular concrete. Thus, for every unit of soil removed, four units of cellular concrete can raise the elevation without additional load.

The excellent flow characteristics of cellular concrete and the fact that it does not require the compaction required by granular fills are major advantages in these applications. Each application is unique and must be considered on its individual merits. Common applications include roadway rehabilitation, bridge approaches, fills behind retaining walls, load-reducing fills over structures, and various pipeline and culvert applications [37].

Precast Elements

Precast cellular concrete elements may be produced from specially designed mixtures; however, they are not commonly used due to the low strength and high drying shrinkage of moist cured low-density cellular concrete. Autoclaved aerated concrete (AAC) is more suitable for manufacturing precast low-density blocks and steel-reinforced panels that have structural properties. In AAC, autoclaving (which is steam curing under pressure and high temperature) is employed to provide the material with improved structural properties and dimensional stability. Although this chapter does not address AAC, it is worth noting that ASTM has been active since the early 1990s in developing standards for this material, and currently two ASTM subcommittees C 27.60 "Precast Autoclaved Aerated Concrete" and C 15.10 "Autoclaved Aerated Concrete Masonry" are active in producing standards for AAC. Currently published standards are ASTM Standard Specification for Precast Autoclaved Aerated Concrete (PAAC) Wall Construction Units (C 1386) and ASTM Standard Specification for Reinforced Autoclaved Aerated Concrete Elements (C 1452).

Quality Control and Testing

Field quality control procedures for cellular concrete involve measuring the wet density of the material at the point of placement and comparing this density with the design wet density of the mixture. Slight adjustments in the mixture may be necessary to bring the wet density within the specified range. If batching, mixing, and placing procedures are consistent, the material should have only minor deviations in density.

The physical properties of cellular concrete are closely related to the mixture design parameters such as cement content, water-cement ratio, and the quality of other ingredients such as sand gradation, and the use of supplementary cementitious materials. The physical properties of the mixture are reproducible within acceptable limits.

The actual quality control procedure involves securing cylindrical test specimens from the measured wet density at the point of placement. After a few days, these samples are moved from protection at the casting location to the laboratory for subsequent curing and compressive strength testing. Compressive strength and density are the primary indicators of mixture quality. Properties such as permeability, freeze-thaw resistance, and water absorption correspond to these measured parameters–strength and density.

Summary

Cellular concrete has a variety of unique properties that makes it suitable for a large number of specialized applications. The ability to introduce a large volume of macroscopic, discrete air cells uniformly distributed throughout the matrix is the key factor that provides cellular concrete with its distinctive properties. Whenever a lightweight, inert, insulative, and cost-effective material is desired, it is possible that a specific density and mixture of cellular concrete could be designed for the application.

Cellular concrete has many potential new applications. Although it has been used in construction for over 50 years, new materials and innovative mixture proportions will make it a viable option for a broader range of applications. There are significant opportunities for expanding existing applications, as well as continuing research into new areas such as specialized material performance properties.

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Concrete for Radiation Shielding

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Introduction

History and Responsibilities

SUBCOMMITTEE C09.41 IS RESPONSIBLE FOR ASTM standards dealing with concrete for radiation shielding. This concrete systems subcommittee is assigned the charter of establishing and maintaining the standards needed for regulation of pertinent radiation shielding materials used in concrete. The predecessor subcommittee, designated C09.02.08, was established as a result of a joint symposium conducted by ASTM and the American Nuclear Society in 1965. Results of three years of study by this subcommittee led to the development of ASTM standards as noted:

- Specification for Aggregates for Radiation-Shielding Concrete (C 637)
- Descriptive Nomenclature of Constituents of Aggregates for Radiation-Shielding Concrete (C 638) [1]

Preplaced-aggregate concrete is a method used to make very dense and uniformly consistent radiation barriers. Subcommittee C09.41 is responsible for the preplaced-aggregate concrete standards in addition to ASTM C 637 and ASTM C 638. The designation and titles for these standards are as follows:

- Specification for Grout Fluidifier for Preplaced-Aggregate Concrete (C 937)
- Practice for Proportioning of Grout Mixtures for Preplaced-Aggregate Concrete (C 938)
- Test Method for Flow of Grout for Preplaced-Aggregate Concrete (C 939)
- Test Method for Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory (C 940)
- Test Method for Water Retentivity of Grout Mixtures for Preplaced-Aggregate Concrete in the Laboratory (C 941)
- Test Method for Compressive Strength of Grouts for Preplaced-Aggregate Concrete in the Laboratory (C 942)
- Practice for Making Test Cylinders and Prisms for Determining Strength and Density of Preplaced-Aggregate Concrete in the Laboratory (C 943)
- Test Method for Time of Setting of Grouts for Preplaced-Aggregate Concrete in the Laboratory (C 953)

Radiation Shielding Primer

Beginning with the early days of the nuclear industry, concrete has been used as a radiation shield because its mechanical properties, chemical composition, and ease of construction make it compatible for withstanding imposed service demands. Concrete can even be enhanced as a shielding material with the use of special additives and aggregates to provide specific attenuation characteristics. In order to establish a firm basis for using the materials mentioned in ASTM C 637 and C 638, knowledge of radiation concepts is essential for designers. Basic concepts, consisting of selective nuclear physics together with applicable material properties and interactions, are developed. Building from this foundation of understanding, the significance of the tests and specifications needed to make concrete for radiation shielding is evident.

Pertinent Atomic Structure and Physics

All elements consist of unique configurations of atoms. To maintain the electrical stability of atoms, a certain number of positively charged particles, called protons, are balanced by an equal number of negatively charged particles, called electrons. In addition to protons and electrons, an atom has some number of neutrally charged particles, neutrons, associated with a particular element. Fundamental to the structure of any particular element, atoms are uniquely configured with the same number of protons for each element, but they may contain a variable number of neutrons. Like elements with differing neutron counts are called the isotopes of the element. The neutrons and protons are bound into a tight mass at the center of the atom, called the nucleus. Essentially, the entire mass of an element is concentrated in the nucleus of its atom. Electrons are also bound to the atom, but they orbit the nucleus in a set pattern of separate levels or shells. Electrons contribute virtually no mass to the atom of an element.

There are two important concepts to keep in mind with the preceding model of the atom. First is the enormous volume of empty space that is contained within the atom. As an example, think of children playing on the beach. One is holding a large plastic beach ball with both arms, while the other cradles a single grain of sand in his palm. Now visualize the grain of sand centered inside the beach ball. If the grain of sand is thought to

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TABLE 1—Number of Atoms in 4000 psi (27 580 kPa) Concrete

Element	Unit Weight Proportions	Grams per cm^3	Atoms per gram	Average Atoms per cm^3
Si	1234.16	0.7322	2.14452E+22	1.57022E+22
Al	125.36	0.0744	2.23227E+22	1.66081E+21
Fe	59.46	0.0353	1.07852E+22	3.80718E+20
Ca	311.95	0.1851	1.50282E+22	2.78172E+21
Mg	23.65	0.014	2.47809E+22	3.46933E+20
S	7.09	0.0042	1.87837E+22	7.88916E+19
Na	35.09	0.0208	2.61986E+22	5.4493E+20
K	45.84	0.0272	1.54048E+22	4.1901E+20
Ti	5.36	0.0032	1.25828E+22	4.02649E+19
P	1.30	0.0008	1.94455E+22	1.55564E+19
H	15.84	0.0094	5.97579E+23	5.61724E+21
O	1846.50	1.0955	3.76452E+22	4.12403E+22

be of comparable size to the nucleus of an atom, then the plastic covering of the beach ball can be thought to represent the electron orbits of the atom. The second important understanding regarding atoms is the enormous number of them contained in matter. Table 1 provides a listing of elements and the number of atoms per cubic centimeter for normal weight concrete used at Los Alamos National Laboratory.

Light elements, so called because their atoms are composed of the fewest number of protons, have very stable nuclei. At the opposite end of the spectrum, heavy elements, which contain large numbers of protons, are somewhat unstable. This is because heavy elements are composed of an increasingly larger numbers of neutrons, compared to the proton count in the nucleus. Subsequently, this high ratio of neutrons to protons creates the potential for instability as contrasted by the light elements. The structure of the nucleus of heavy elements is so configured to afford sufficient volumetric separation of the individual protons to accommodate the forces perpetuated by the positive charges of those particles. For a comparison of light and heavy elements, hydrogen contains one proton and no neutrons, while uranium-235 contains 92 protons and 143 neutrons [2]. Instability in the nucleus of an atom, particularly large ones, necessitates particle matter or energy, or both, to be released from the atom to achieve a more stable configuration. This process of altering atoms to achieve stability is called radioactivity. If the radioactive particles or energy have the ability to interact and alter the structure of atoms of biological tissue, this is called ionizing radiation. Only ionizing radiation needs shielding, and the two major categories are as follows:

- Electromagnetic waves
- Nuclear particles [1]

Ionizing electromagnetic waves consist essentially of X-rays and gamma rays. Gamma rays are similar to X-rays but are usually thought to have higher energy levels (more cycles per second). To understand the significance of ionizing electromagnetic waves, consider a sampling of the non-ionizing electromagnetic spectrum:

- Electricity has a frequency of 60 cycles/s (Hz) and a wavelength of 5000 km (3100 miles)
- AM radio waves have frequencies of 1 000 000 Hz and wavelengths of 300 m (1000 ft)
- Microwaves have frequencies of 2 540 000 000 Hz and wavelengths of 12 cm (5 in.)

In contrast, ionizing electromagnetic waves, such as X-rays, have a frequency starting at around 1 000 000 000 000 000 Hz and a wavelength of 100 nm [3]. Gamma rays are different from X-rays because the source of X-rays is external to the nucleus, while gamma rays are generated from the nucleus. At high energy levels, electromagnetic waves predominately display particle characteristics, rather than just wave-like phenomena. The particle component of electromagnetic waves is called a photon, and this term is essentially interchangeable with x-rays and gamma rays.

Nuclear particles, such as alpha, beta, and neutron, constitute ionizing radiation too. Other nuclear particles exist, but the three listed particles are representative of the usual type of ionizing radiation requiring shielding [4].

Alpha particles consist of positively charged helium atoms. During the decay of certain atoms, such as plutonium-239 into the more stable state of uranium-235 [2], emission of pairs of protons and neutrons (He^{++}) occur. A stable, zero charge helium atom consists of two protons, two neutrons, and two orbital electrons. During radioactive decay, the helium atomic nucleus, without orbital electrons, is released. This type of radioactive emission defines alpha particles.

Beta particles consist primarily of negatively charged electrons that are emitted from the nucleus of an atom. Sometimes a beta particle is released as a short-lived positive electron (positron), which quickly annihilates in the electric field of matter. As an example of beta radiation, consider the decay of phosphorous-32. A neutron in the atom decays into a proton and an electron. A forced release of the electron from the nucleus occurs, and the proton count of the atom increases by one to 16. This spontaneous stabilization process transforms the element into sulfur-32 [2].

Neutron particles are released during spontaneous or induced fission. These particles are important in maintaining the fission process by striking other potentially unstable atoms and creating a chain of fission activity. Notable neutron sources for instigating fission activity are beryllium and its alloys. These materials are excellent participants to initiate chain reactions because the low neutron binding energy of beryllium provides a ready source of neutrons to encourage this process [5]. In addition to fission, scientific accelerators produce beams of neutron particles for a variety of experimentation purposes. Radiation from these processes must be shielded to address health, safety, and environment issues.

Generally, there are three types of neutron particles of interest in radiation shielding. They are grouped according to the level of kinetic energy associated with the particle. Slow neutrons are referred to as thermal neutrons and have associated energy levels less than 0.5 electron volts (eV). Intermediate or epithermal neutrons have kinetic energy in the vicinity of 5000 eV. Fast neutrons have kinetic energy levels exceeding 500 000 eV [6].

Physical and Biological Perspective

Any material of sufficient thickness can be used as shielding against alpha, beta, neutron, and gamma radiation. Alpha and beta particles are deflected and absorbed by interacting with the electrical charges of the atoms comprising matter. In contrast, gamma rays are absorbed by the interaction with orbital electrons of atoms. Increasing the mass of a shielding material is the most effective method of increasing the probable interaction with orbital electrons. As with gamma rays, the electrically charged particles of atoms do not affect neutrons. Instead, these particles collide inelastically or elastically with the nuclei of heavy or light atoms, respectively.

Shielding is required to protect people and the all-encompassing environment from the effects of radiation. Both nonpenetrating and penetrating radiation can ionize or break the bonds of atoms in human body cells. However, nonpenetrating radiation is only a biological threat if the particles are introduced internally, while penetrating radiation constitutes a biological threat from sources external to the being. Radiation exposure effects on humans consist of radiation sickness, cataracts, sterility, genetic mutations, coma, cancer, and death—depending on the amount and length of time of the exposure. Penetrating radiation consists of gamma rays and neutrons. This type of radiation can penetrate the body and damage the atoms of cells composing the various organs [4].

Radon gas is an example of naturally occurring nonpenetrating alpha and beta radiation [7]. Alpha particles cannot penetrate human skin, but they are harmful if ingested. Although beta particles can penetrate and damage human skin, betas cannot penetrate body organs, unless ingested. Numerous newspaper accounts describe radon contamination of houses. This occurs because plumes of radon gas exist in-situ in the ground. These deposits develop as by-products from the decay of naturally occurring radioactive materials. Because of its gaseous form, this type of radiation is easily inhaled into the lungs where it will damage tissue. Although the floor of a house is capable of shielding against radon radiation, penetrations through the floor, such as for utilities, will allow the gas to migrate into occupied space.

Shield Mechanics

Radiation shields are designed and constructed to capture all primary and secondary radiation emitted by a source. Design of shielding focuses on the radiation requirements for neutrons and photons. Recall a previous statement that atoms have vast amounts of empty space within their confines. Consequently, neutral radiation particles and energies will simply pass through most atoms but eventually will make an impact. Therefore, the mechanics of a shield can be viewed, fundamentally, as the probabilistic occurrence that a neutron or photon will interact with a sufficient number of atoms, which will deflect, slow, and absorb the particles, without leakage of secondary radiation. Attenuation is best explained by following the hypothetical path of a high energy neutron and photon through a shield, although an actual shield will attenuate enormous numbers of radioactive particles, not just one. The following descriptions highlight the shielding evolution for neutrons and photons.

Neutron Attenuation

A radiation source emits a fast neutron into its concrete shield. Heavy materials, such as magnetite or steel, contain large nuclei from iron, which are big targets for emitted neutrons. Such materials are often used in concrete shields to increase the probability that the neutron will collide rapidly and multiple times in its path through the shield. Simply stated, the chance of hitting the big atom of a heavy element is much greater than hitting a small atom of a light element. The fast neutron impacts and bounces off the nucleus in a process called inelastic scattering. The neutron particle remains an entity from this type of collision, but photons are released from the interaction. Energy loss occurs as a result of numerous interactions as the deflected travel path slows the neutron.

Eventually, the neutron slows enough for the physics of another attenuation mechanism to prevail. Intermediate energy neutrons have a propensity for retention in the nucleus of atoms. Light elements, such as hydrogen, are particularly adept at retention of neutrons in this energy range. However, the neutron retention is not an absorption process because the particle impacts the receptive nucleus with sufficient energy to cause the release of another neutron from that nucleus. The newly emitted neutron has significantly lower energy. Attenuation by this type of interaction is called elastic scattering. An abundance of hydrogen in a radiation shield increases the probability that elastic scattering will occur quickly after neutrons attain intermediate energy levels.

Slow, low energy, thermal neutrons are absorbed by impacting the nucleus of an atom. The neutron is retained in the nucleus, and binding energy is released as secondary gamma radiation. Now, though, the target atom has an increased neutron count, making it an isotope of the element. The isotope may be in an unstable configuration, causing it to quickly decay into two different, more stable elements. Consequently, if a radiation shield contains certain elements, such as boron, that increase the probability of quick absorption, shield thickness can be reduced.

Photon Attenuation

Photon attenuation is attained from different processes from those described for neutron particles. Interaction with dense materials is the key for optimum photon attenuation. In concrete, very heavy (dense) elements, such as contained in magnetite or steel, are most important for shielding purposes. Dense elements consist of large atoms that have numerous shells of orbital electrons. The physical size, number of orbital electrons, and influence of the electrostatic field between the charged particles significantly increase the probability that photons will attenuate quicker with heavy elements. Three attenuation mechanisms are important in stopping photons.

Pair production attenuates photons through the proximity of the nucleus and the electrical (Coulomb) fields of the atom

[2]. In this process, the photon enters the electrostatic influence of the atom and disintegrates into two electrons of opposite charge. The positive electron, or positron, is anti-matter. It annihilates quickly, forming low energy photons. The physics of pair production is the predominant process of attenuation for high-energy photons with an atom [8].

A scattering attenuation mechanism involves photon interaction with an orbital electron of an atom. This effect is called Compton scattering. In this process, the impact between the photon and the orbital electron dislodges the electron from its shell in the atom. The result of this interaction causes the photon to deflect and to lose energy. This is the most significant attenuation mechanism of medium energy gamma rays, which can be generated as secondary radiation from thermal neutron capture [8].

Absorption of low energy photons occurs through photoelectric effect. As with Compton scattering, the photon interacts with an orbital electron of an atom. In this process, the energy of the photon is completely dissipated by first, breaking the electron free of its atomic shell, and second, by imparting all remaining energy to the expelled electron [2].

Terms for Shield Design Calculations

Of the numerous material properties and constants associated with shield design, two fundamental terms are selected for discussion. Knowledge of these terms is important to enhance communication exchanges between people in the concrete industry with clients or designers in the nuclear industry.

Scattering Cross Section

The scattering cross section is a function representing the probability that a neutron or a photon will be scattered by interacting with an atom of an element. Scattering cross sections are an expression of the sum of the scattering attenuation mechanisms inherent in and characteristic of a particular element. The scattering cross sections of some typical elements commonly found in concrete shields are listed as follows:

- Hydrogen, 99.0 barns
- Oxygen, 4.2 barns
- Boron, 4.0 barns
- Silicon, 1.7 barns
- Iron, 11.0 barns
- Barium, 8.0 barns [9]

An element, such as hydrogen, which has a large scattering cross section, is most beneficial when used as an elastic scattering material. Large, heavy elements, such as iron, are important for inelastic scattering of neutrons.

For gamma radiation, dense aggregates consisting of heavy elements are the best concrete materials to scatter and reduce the energy. Typically, steel, iron, and iron ores (such as magnetite, barites, ilmenite, and hematite) are used as dense aggregates in concrete for this purpose. The final density and homogeneous nature of the concrete is more important than the particular set of heavy aggregates used. For all practical purposes, thickness of concrete shields is proportional to the density of the mix [1].

Absorption Cross Section

The absorption cross section is the material property representing the probability that a neutron or photon will be absorbed by interaction with an atom of an element. Absorption cross sections are an expression of the sum of the absorption attenuation mechanisms inherent in and characteristic of a particular element. The absorption cross sections of some typical elements commonly found in concrete shields are listed as follows:

- Hydrogen, 0.3 barns
- Oxygen, 0.0 barns
- Boron, 755.0 barns
- Silicon, 0.2 barns
- Iron, 2.5 barns
- Barium, 1.2 barns [9]

An elemental isotope, such as boron-10, has a very large absorption cross section and releases low energy gamma rays as secondary radiation. The properties of boron-10 are highly valued as a shielding material.

For photons, absorption occurs through the interaction with an orbital electron of a target atom. Dense materials consisting of heavy elements are the best materials to control gamma-ray energy. For concrete mixes, steel, iron, and iron ores (such as magnetite, barytes, ilmenite, and hematite) are used to increase mix density for shielding. The final mix density, rather than any particular heavy aggregate used, is the most important aspect of gamma radiation shield design.

Concrete Materials

Normal-weight concrete consists of cement, water, sand, and coarse aggregate. The addition of natural or manufactured minerals, in the form of sand and coarse aggregate, can alter the composition of normal-weight concrete into an efficient radiation shielding material. This conglomeration of materials results in a concrete mix consisting of both light and heavy elements as is required to achieve an effective radiation barrier. Proper mixing and placement of the concrete is essential to make the barrier as homogenous as possible.

Natural Minerals to Enhance Shielding Attributes

Heavy, natural minerals used in concrete for radiation shielding consist of magnetite (elemental composition $Fe_2O_3 \cdot FeO$ with a specific gravity ranging from 4.6 to 5.2 [10]), ilmenite ($FeO \cdot TiO_2$), barite ($BaSO_4$), hematite (Fe_2O_3) [11], and witherite ($BaCO_3$) [10]. These aggregates are substituted for usual concrete aggregate to obtain a high-density concrete matrix. Such a radiation barrier is effective at scattering high-energy neutrons, as well as scattering and absorbing photons.

Light, natural materials used in concrete for radiation shielding consist of serpentine ($3MgO \cdot 2SiO_2 \cdot 2H_2O$), goethite ($Fe_2O_3 \cdot H_2O$), limonite ($Fe_2O_3 \cdot XH_2O$, also referred to as impure goethite, with a specific gravity ranging from 3.5 to 4.5 [10]), as well as colemanite ($2CaO \cdot 3B_2O_3 \cdot 5H_2O$ [11]). These materials are known as hydrous aggregates. Their use in concrete enhances the hydrogen content of the shield. Hydrous aggregates moderate neutrons through elastic scatter and also absorb lower energy neutrons.

Caution must be exercised in working with some of these special shielding minerals. For instance, serpentine contains asbestos, a known carcinogen.

Manufactured Aggregates to Enhance Shielding Attributes

Manufactured products used for gamma shielding typically consist of ferrophosphorous (FeP or Fe_2P or Fe_3P), iron or

TABLE 2—Aggregates Used in Shielding Concrete [1]

Natural Mineral		Manufactured	
Local sand/gravel		Crushed aggregate	
calcareous	(2.5–2.7)^a	heavy slags	(~5.0)
siliceous		ferrophosphorous	(5.8–6.3)
basaltic	(2.7–3.1)	ferrosilicon	(6.5–7.0)
Hydrous ore		Metallic iron products	
bauxite	(1.8–2.3)	sheared bars	(7.7–7.8)
	[15–25 %]^b	steel punching	
serpentine	(2.4–2.6)	iron or steel shot	(7.5–7.6)
	[10–13 %]		
goethite	(3.4–3.8)		
limonite	[8–12 %]^c		
Heavy ore		Boron additives	
barite	(4.0–4.4)	boron frit	(2.4–2.6)
magnetite ^{c}		ferroboron	(5.0)
ilmenite	(4.2–4.8)	borated	
		diatomaceous	
hematite	(4.6–5.2)	earth	(∼1.0)
witherite	(4.3)	boron carbide	(2.5 to 2.6)
		calcium boride	2.5
Boron additives			
calcium			
borates			
borocalcite			
colemanite	(2.3–2.4)		
gerstley	(2.0)		
borate			

[a] Specific gravity is shown in ().

[b] Water of hydration is indicated in [].

[c] Refer to chapter section on Concrete Materials

steel shot, and steel punchings [11]. Ferrophosphorous is a by-product from the production of phosphorous. Because of its poor attributes as a concrete aggregate, such as degradation, caution is advised in using this material [10].

Boron frit consists of the fritted product of silica (SiO_2) with borax ($Na_2B_4O_7 \cdot 10H_2O$) [11], as well as ferro boron. Boron carbide (B_4C) and calcium boride (CaB_6, with a specific gravity of 2.5) are manufactured products that are efficient for neutron absorption. Table 2 lists these aggregates along with the density of each material.

TABLE 3—Densities of Shielding Concretes Commonly Specified for Given Heavy Aggregates [1]

Type of Aggregate	Density, lb/ft^3	Density, kg/m^3
Barite	3450 to 3600	215 to 225
Magnetite	3500 to 3750	220 to 235
Ilmenite	3500 to 3850	220 to 240
Ferrophosphorus	4550 to 4800	285 to 300
Barite and boron additive	3200 to 3450	200 to 215
Magnetite and boron additive	3350 to 3600	210 to 225

Densities

Standard heavyweight concrete, using natural iron ore aggregate, can achieve densities up to 3850 kg/m^3 (240 lb/ft^3) [1]. Barite, magnetite, and ilmenite are the traditional materials used for this endeavor. Table 3 lists concrete densities attainable using heavy aggregate in the mix. Manufactured iron and steel aggregates are used to achieve concrete densities greater than 4000 kg/m^3 (250 lb/ft^3) [1].

Irradiation Effects

There are damaging effects to concrete used as radiation barriers from the scattering and absorption processes. Irradiation of concrete causes dissociation of water into its hydrogen and oxygen components. Compressive and rupture strengths both decrease with time due to radiation exposure and high temperature, although the effect is tolerable under normal reactor conditions [12].

Ontario Hydro Research Division researched the effects of radiation exposure on concrete. A nuclear power plant, which had been operating for over 20 years, was studied. Concrete core samples from the shielding of this plant were obtained and tested for physical and thermal properties. The results from this testing were compared with the test results of samples from the original construction, as well as laboratory test specimens.

A summary of some of the findings of this research follows:

1. The average compressive strength of the 20-year-old heavyweight concrete in the reactor shield increased in the same

progression from the radiation source through the shield. The samples, taken closest to the radiation source, showed compressive strengths 10 to 20 % less than the samples extracted on the farthest face from the radiation source.

2. The modulus of elasticity of the concrete shield also displayed variations (approximately 10 %) between samples on the farthest face compared with samples closest to the radiation source.
3. The thermal diffusivity, which helps explain the rate at which a material undergoes temperature change, and the thermal conductivity, which is a measure of heat transfer through a material, were roughly 30 % less for the 20-year-old heavyweight concrete shield samples as compared with the laboratory-tested heavyweight concrete specimen [13].

Temperature Effects

Concrete used in a nuclear reactor to shield the core may lose up to 50 % of its compressive strength when it is exposed to temperatures of 430°C (800°F) for a long period of time. For high temperature applications, concrete should be made with heat-resistant aggregates to prevent excessive compressive strength loss and to avoid dehydration. If shielding requires aggregates high in fixed water, subjecting the concrete to lower temperatures would be important. Limonite dehydrates above 200°C (400°F), while serpentine can withstand temperatures up to 370°C (700°F) [1].

In general, nuclear and mechanical properties are affected by both temperature gradients and changes in temperatures. Research indicates the maximum temperature occurs in the first 30 cm (12 in.) from the exposed face of concrete subjected to a radiation flux. For concrete shields functioning at temperatures less than 105°C (220°F), free water will be present in the concrete for 10 to 20 years. As a worst-case scenario, thermal effects from radiation have caused as much as two-thirds of the total original water content to be lost over the useful life of the concrete shielding [14]. To prevent excessive water loss from occurring as a result of high temperatures, it is common to increase the water-cement ratio (w/c) of the concrete in the initial construction of the shield. Of course, higher w/c ratios must be monitored so satisfactory performance for strength is not sacrificed.

Placement and Verification Considerations

Normal-weight concrete, with no special aggregates or additives, is satisfactory to shield any nuclear radioactive source. However, high-energy gamma rays and neutrons from a reactor or accelerator may require concrete masses that are prohibitive in size. Facility designs may impose space constraints on the design of the shield. These types of constraints necessitate the use of special shielding materials to increase effectiveness. However, these special materials also increase the cost of shields through both higher material costs and the expense associated with construction. For instance, the mixing and placing operations are more difficult because the very heavy aggregates or the special fine aggregates make uniformity harder to achieve when compared with the construction practice for normal concrete. Therefore, specifications for the material components and the construction of the concrete should be written specifically for each shielding job. The specification writer should have knowledge of cost of the various materials available for shielding and the difficulties involved in constructing the shield with these materials. The following are comparative costs for general categories of concrete shields:

- High density, natural mineral, 3.0 times the cost of normal concrete
- High density, manufactured aggregate, 4.0 to 9.0 times the cost of normal concrete
- High hydrogen content, 1.3 times the cost of normal concrete [15]

These values must be considered "Ball Park" values because material and labor costs fluctuate, competitiveness of contractors varies, and other factors such as material availability may affect cost.

Shield verification practice for nuclear facilities should follow good engineering practice and include documented procedures. One such process, used at the Idaho National Engineering Laboratory, was established to verify the integrity of a concrete shield. It incorporates the use of a test radiation source. The verification process starts with a visual inspection of the shield. All shield penetrations and other deficiencies need to be recorded in a log, so further testing with the test radiation source can follow. The test radiation source should be sufficiently large to emulate actual operating conditions of the facility. Radiation flux through the shield is then measured, recorded, and compared with calculated values. In this way shielding deficiencies can be corrected during facility construction without impacting construction schedules [16].

ASTM Standards

Cast-in-Place or Precast Concrete for Radiation Shielding

Cast-in-place concrete for radiation shielding or precast concrete shield blocks should comply with normal-weight ASTM concrete standards, as amended by ASTM C 637 for aggregates. ASTM C 638 is useful as a supplement to ASTM C 637, because it provides specific descriptions of special aggregates used in concrete for radiation shielding. The following paragraphs briefly summarize the two standards.

ASTM C 637

This standard is a specification of fine and coarse aggregates specifically used in concrete for radiation shielding. Composition, specific gravity, grading, fixed water content, deleterious substances, and abrasion resistance test requirements are listed, as well as requirement exceptions for the various referenced ASTM Standards. Natural and synthetic mineral aggregates are covered by this specification.

A key provision of the specification is the requirement for uniformity on specific gravity and fixed water content. There is a 3 % limitation on the variation of bulk specific gravity of the sample, while hydrous aggregate must not fall more than 5 % below the minimum specified.

ASTM C 638

Descriptions of the various fine and coarse aggregates specifically used in concrete for radiation shielding are presented. This standard is useful to obtain an understanding the specific aggregate components used in concrete shielding. Specifically covered are iron minerals and ores, barium minerals, ferrophosphorus, boron minerals, and boron frit glasses. The nomenclature defers to ASTM C 294, Standard Descriptive Nomenclature for Constituents of Concrete Aggregates for the common components of concrete.

Preplaced-Aggregate Concrete for Radiation Shielding

Preplaced-aggregate concrete has commercial applications beyond concrete for radiation shielding. Therefore, this topic is covered in a separate chapter within this publication. For radiation shielding usage, preplaced-aggregate concrete is used when very heavy-weight concrete, greater than 4000 kg/m^3 (250 lb/ft^3) is required for shielding radiation, or when an especially high level of uniformity of the mix is needed. [17]. Special forms are utilized in which reinforcing steel and mix aggregates are initially placed, then pressure grout fills the voids to make a homogeneous, dense concrete shield. The precise location of these components and the stringent inspection requirements guarantee a shield of uniform matrix, unmatched by normal-weight concrete placement techniques. The following ASTM standards deal specifically with making preplaced concrete.

ASTM C 937

This is a specification for fluidifier used in grout to enable the grout to maintain fluid characteristics while reducing water. Fluidifier is used in preplaced-aggregate concrete where a very dense concrete is required. This standard covers physical requirements, composition, sampling, and test methods for the fluidifier.

ASTM C 938

This practice describes the method of testing required to select correct mix proportions for grout used in preplaced-aggregate concrete. Materials, sampling, and procedures are covered.

ASTM C 939

This test method establishes the time of efflux for grout through a standard flow cone in order to determine the fluidity of the grout for preplaced-aggregate concrete. It may be performed in the laboratory or the field. Calibration of apparatus and procedures are presented.

ASTM C 940

This test determines the amount of expansion exhibited by freshly mixed hydraulic-cement grout over a period of time. Also, the amount of bleed water, or the free water that rises to the top of grout, is determined. In general, minimization of expansion and bleeding characteristics of preplaced-aggregate concrete improve shielding effectiveness.

ASTM C 941

This test method measures the property of freshly mixed hydraulic-cement grout to retain mixing water. This is used to qualify fluidifiers or to determine the effects of admixtures in grout for preplaced-aggregate concrete.

ASTM C 942

This test method provides a measure of the compressive strength of hydraulic-cement grout, which has expansive capability but hardens in conditions that tend to restrain expansion. This test also measures the effect of the fluidifier on the compressive strength of the grout for preplaced-aggregate concrete. Temperature, humidity, sampling, preparation of specimen, and procedure standards are covered.

ASTM C 943

This practice covers procedures for making standard test cylinders and prisms for preplaced-aggregate concrete. It follows the process used in the field to construct preplaced-aggregate concrete. The cylinders are used to determine compressive strength, while the density is calculated from prism measurements.

ASTM C 953

This test is essential to determine the setting time of grout, as well as the acceptability of components of grout mixed to fluid consistency for use in preplaced-aggregate concrete. Apparatus, sampling, and procedures are presented.

Conclusions

ASTM has been active in preparing and maintaining standards for concrete used in radiation shielding. Subcommittee C09.41 and its predecessor C09.02.08 have been given the task to perform this service.

Ionizing radiation is a biological hazard requiring shielding. Neutrons and photons are emitted radiation for which shields are typically designed. Although neutron and photon radiation attenuate differently, concrete is an excellent material to capture both.

Normal-weight concrete provides good radiation shielding characteristics, but altering the constituents of the mix can optimize the shielding properties. The use of iron, steel, or iron ore for aggregates produces a heavyweight concrete that is most suitable to shield electromagnetic radiation or to scatter fast neutrons. The presence of hydrogen in a shield is important to moderate neutrons. Hydrogen is abundant in concrete because of mixing water. The amount of fixed water in concrete can be supplemented by the use of hydrous aggregates, which contains high amounts of fixed water. A boron aggregate additive can be mixed in the concrete to enhance absorption of thermal neutrons.

Concrete shields are constructed using standard cast-in-place concrete techniques or by using preplaced-aggregate construction methods. The preplaced-aggregate concrete is normally used to ensure density and uniformity requirements of a shield.

Acknowledgments and Disclaimer

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The Los Alamos National Laboratory strongly supports academic freedom and a researcher's right to publish; therefore, the Laboratory as an institution does not endorse the viewpoint of a publication or guarantee its technical correctness. This chapter is designated LA-UR-05-3520.

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Fiber-Reinforced Concrete

Peter C. Tatnall[1]

Preface

IN PREPARATION OF THIS CHAPTER, THE CONTENTS of the 4th edition were drawn upon. The author acknowledges the author of the 4th edition, Colin D. Johnston. The current edition will review and update the topics as addressed by the previous author, introduce new technology that has been developed, and include up-to-date references.

Introduction

Fiber-reinforced concrete (FRC) in the context of this chapter is conventionally mixed concrete containing discontinuous fibers that initially are randomly oriented in three dimensions in the mixture, but may subsequently become partially aligned by vibratory consolidation, surface finishing, or shotcreting and by geometrical constraints at mold surfaces, formwork, or interfaces with existing concrete, rock, or subgrade. The standards applicable specifically to it are under the jurisdiction of ASTM Subcommittee C09.42 on Fiber-Reinforced Concrete, but many of the standards of ASTM Committee C9 on Concrete and Aggregates may apply to FRC with or without modification.

Thin-section, glass fiber-reinforced, cementitious mixtures that generally do not contain coarse aggregate and are prepared by the spray-up or other special processes are not discussed. They are under the jurisdiction of ASTM Committee C27 on Precast Concrete Products and ASTM Subcommittee C27.40 on Glass Fiber Reinforced Concrete Made by the Spray-Up Process. Also excluded are glass fiber-reinforced mortars for surface-bonded masonry that are under the jurisdiction of ASTM Committee C12 on Mortars for Unit Masonry and ASTM Subcommittee C12.06 on Surface Bonding.

Nature of FRC

A brief explanation of the nature of FRC insofar as it differs from concrete without fibers is necessary to understand the rationale governing development of tests and the significance of the properties determined in the tests.

Whether the fibers are steel, polypropylene, nylon, glass, carbon, or other materials yet to be evaluated, they act like very long slender needlelike particles in freshly mixed FRC. During mixing, they are subject to bending, impact, and abrasion by the action of conventional mixers, and must resist both damage caused by breakage that produces shortening and, in the case of multifilament strands, separation that causes greatly increased specific surface. The high specific surface of fibers, either monofilaments or multifilaments, and the needlelike shape, tend to impart considerable cohesion to FRC mixtures. This makes the reduction in workability associated with adding fibers to concrete appear very severe when judged under the static conditions of the standard slump test. However, many FRC mixtures with low slump flow quite easily when vibrated, so workability tests that employ vibration to produce dynamic conditions are often more appropriate than the standard slump test because placement in practice normally employs vibration.

The workability-reducing effect of fibers depends largely on fiber content and fiber aspect ratio. Aspect ratio is the ratio of fiber length to diameter, or equivalent diameter for fibers of noncircular cross section, and is defined for fiber monofilaments like steel in the ASTM Specification for Steel Fibers for Fiber-Reinforced Concrete (A 820/A 820M). Fiber content is defined most conveniently in practice in kilograms per cubic meter (lb/yd^3) of concrete, but fundamentally it is the volume fraction of fibers regardless of the density of the fiber material that influences the effect of fibers in concrete. Both fiber content and aspect ratio are often limited by workability considerations, although the advent of high-range water reducers has helped to address this problem.

Aspect ratio and fiber content also figure prominently in determining property improvements in hardened FRC. Ideally, the aspect ratio should be as small as possible to minimize loss of workability and as large as possible to maximize the resistance of fibers to pullout from the matrix and thus their reinforcing effectiveness. The fiber content should be as small as possible to minimize loss of workability and as large as possible to maximize reinforcing effectiveness.

What is possible in terms of miscibility, placeability, and property improvements, is therefore a compromise that varies widely with the nature of the proposed application. More than 1 % fibers by volume of concrete, 78 kg/m^3 of steel, 9.1 kg/m^3 of polypropylene (132 lb/yd^3, 15.3 lb/yd^3, respectively), is uncommon in conventionally mixed FRC or in fiber-reinforced shotcrete. Steel fibers are quite widely used in slabs-on-grade at as little as 0.25 % by volume, and microsynthetic fibers in slabs-on-grade at as little as 0.1 % by volume. Aspect ratios for steel and macrosynthetic fibers are generally 40 to 80, and usually less than 100. Aspect ratio is not easily determinable for microsynthetic multifilament or fibrillated fiber types such as

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polypropylene. Nominal length is generally 15–50 mm, and rarely more than 65 mm.

Innovations in fiber development that combine reductions in fiber length or aspect ratio, thus minimizing the workability-reducing effect, with improvements in the resistance of the fiber to pullout from the matrix, thus maximizing reinforcing effectiveness in the hardened state, have taken place over the years. Compared with fibers produced in the early 1970s, straight smooth monofilaments of uniform cross section are now quite rare. Examples of innovative techniques adopted specifically to optimize fiber performance in both the freshly mixed and hardened states are surface texturing, surface deformation (similar to deformed rebar), hooked or enlarged ends, crimping to a wavy rather than a straight profile, partial splitting (fibrillation) to produce multifilament strands with cross links that separate into branched monofilaments during mixing, and bundling of fibers with a water-reactive glue or paper prior to separation in the mixer. More recently, fiber blends (hybrids) consisting of either combinations of different materials such as steel and polypropylene, or combinations of macro and microfibers of the same material have been introduced, as well as macrosynthetic fibers which mimic the size and shape of steel fibers.

Fiber size determines the number of fibers per kilogram or pound of their batch weight and their number per cubic metre or cubic yard of concrete. For example, in five commercially available types of steel fibers, the number varied from 4000 to 100 000/kg (1800 to 45 000/lb) [1]. For fibrillated or monofilament polypropylene or nylon fibers, the real number dispersed as monofilaments in freshly mixed concrete is difficult to determine because it depends on how completely the bundles separate during mixing and what is their average cross-sectional size, but are estimated in the tens of millions. Obviously, the greater the number of fibers per unit volume of concrete, the lesser the average spacing between them and possibly the lower the probability of zones of weakness due to fiber deficiency arising from nonuniform distribution. However, the true influence of fiber size and number per unit volume on the performance of hardened FRC is uncertain. Perhaps the only point of fairly universal agreement regarding fiber size is that the fiber length should be at least as large as the nominal maximum aggregate size.

Readers interested in more details regarding the nature of FRC and the range of FRCs possible with different amounts and types of fibers, together with their proven and potential applications, should consult appropriate references [2–4].

Freshly Mixed FRC

Sampling and Consolidating Test Specimens

Sampling FRC mixtures for testing should be in accordance with the ASTM Practice for Sampling Freshly Mixed Concrete (C 172). Other sampling requirements specific to FRC are identified in the ASTM Test Method for Time of Flow of Fiber-Reinforced Concrete Through Inverted Slump Cone (C 995), the ASTM Test Method for Flexural Toughness and First-Crack Strength of Fiber-Reinforced Concrete (Using Beam with Third-Point Loading) (C 1018), and in the ASTM Specification for Fiber-Reinforced Concrete and Shotcrete (C 1116). Most important is the requirement that prohibits wet-sieving to remove large aggregate because of possible adverse effects on fiber content and uniformity of distribution.

For consolidation of test specimens, vibration should be chosen for FRC mixtures when ASTM standards permit a choice between rodding and vibration. When ASTM standards for conventional concrete do not provide for vibration or require rodding, external vibration is preferable for FRC when disturbing the natural fiber distribution may affect the test result. For example, uniform fiber distribution is important in beams prepared according to ASTM C 1018 or ASTM C 1399, so external vibration is required rather than internal vibration or rodding that may disrupt the fiber distribution. In contrast, disturbing the fiber distribution is much less important in, for example, air content or density tests, so internal vibration is acceptable.

Workability

Adding fibers to freshly mixed concrete significantly alters its behavior in terms of the three widely recognized rheological parameters that determine workability; namely, stability, mobility, and compactability [5]. Fibers impart considerable stability or cohesion to FRC mixtures that may cause them to appear unworkable when judged only in terms of slump, which is a measure primarily of stability under static conditions. However, when properly proportioned, FRC mixtures flow readily under the dynamic conditions produced by the internal or external vibrators normally used for placement in practice. Accordingly, they are satisfactory from the view point of mobility. Consolidation is accomplished quite easily under vibration, indicating satisfactory compactability.

One test that assesses the workability of FRC under vibration is the ASTM Test Method for Time of Flow of Fiber-Reinforced Concrete Through Inverted Slump Cone (C 995). It determines the time required for a sample of FRC to exit the narrow end of an inverted slump cone after an internal vibrator is inserted (Fig. 1). It was first recommended on the basis of using readily available equipment (that is, a slump cone, internal vibrator, and unit weight bucket) to develop a test more appropriate for FRC than the slump test because the slump of FRC mixtures is lower than that of conventional concrete mixtures with the same time of flow (Fig. 1) [6]. The test works well with steel or other rigid fibers, demonstrating the effects of fiber content and aspect ratio quite clearly (Fig. 2) [7]. Inverted cone (I.C.) flow times in the range of 8–30 s are appropriate for placement and consolidation by vibration [7]. Times more than 30 s mean that the FRC will be very difficult to place and consolidate fully. Some types of long (greater than 50 mm) flexible fibers like polypropylene may tend to wrap around the specified 25-mm-diameter internal vibrator, thus nullifying the validity of the test result. For some mixtures rendered extremely cohesive by a high fiber content, the internal vibrator may simply create a central hole in the sample with the outer portions retained indefinitely in the cone, again nullifying the validity of the test result. Otherwise, the test is a good indicator of the mobility of FRC mixtures under internal vibratory conditions representative of placement in practice.

Another test that assesses workability under vibration is the British Standard Test for the Vebe Time of Freshly Mixed Concrete, BS 1881, Part 2. It is just as effective for FRC as for low-slump and no-slump concretes without fibers. It measures primarily mobility, but slight consolidation may occur during the test implying secondary assessment of compactability. It is as effective as ASTM C 995 in demonstrating the effects of fiber content and aspect ratio on the workability of FRC with steel fibers (Fig. 2). It is probably better for FRC mixtures with long synthetic fibers or mixtures that are very cohesive, since

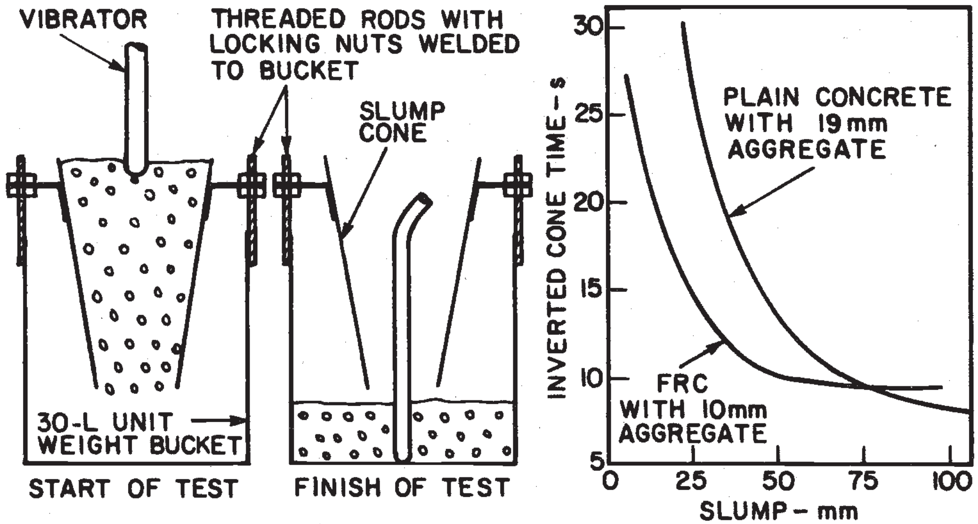

Fig. 1—Inverted slump cone test with relationships between inverted cone time and slump for FRC and plain concrete [6].

the test end-point can nearly always be reached even though the vibration time may be abnormally large. Vebe times in the range of 3–10 s represent adequate workability for placement by vibration [7].

Both the inverted slump cone and Vebe test results correlate closely, and the essentially linear relationship passing through the origin suggests that both are measuring primarily the same rheological characteristic; namely, the mobility or flow of FRC under vibration (Fig. 3, *left*) [7]. In contrast, the relationship between inverted cone time and slump is quite nonlinear (Fig. 3, *right*).

Both the inverted cone and Vebe tests are inappropriate for higher workability FRC mixtures because the test times become too short (less than 3 s) to be determinable with reasonable pre-

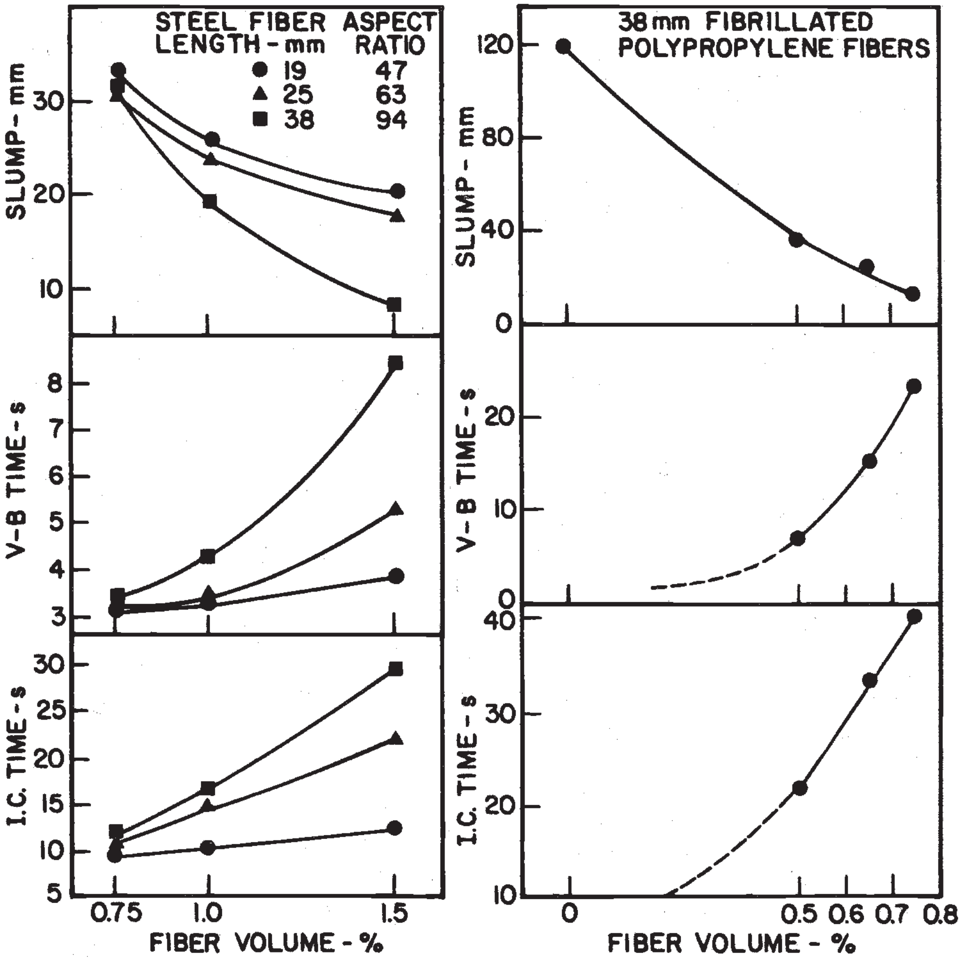

Fig. 2—Effects of fiber content and aspect ratio on slump, V-B time, and inverted cone time [7].

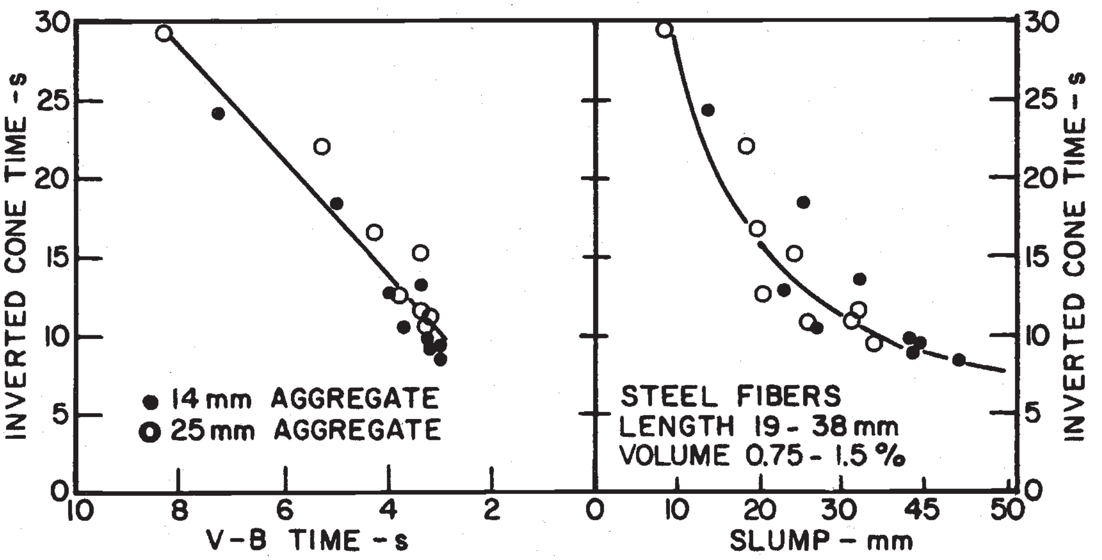

Fig. 3—Relationships between inverted cone time and slump or V-B time for FRC with steel fibers [7].

cision. For the inverted cone, times less than about 8 s may not provide valid results because the FRC may flow freely through the cone. Consequently, slump may be the most practical alternative for such mixtures that produce flowing concrete.

Air Content, Yield, and Unit Density

Air content is just as important for the durability of FRC as for conventional concrete. The standard pressure meter, C 231, and volumetric techniques, C 173/C 173M, are applicable with the modification to require consolidation by vibration when slump is less than about 75 mm (3 in.) as provided in C 231. Vibration may also be desirable for high fiber content mixtures when slump exceeds 75 mm (3 in.) if rodding becomes difficult or produces visibly unsatisfactory consolidation. The potential for excessive entrapment of air due to incomplete consolidation is greater for FRC mixtures than for conventional concretes, especially at high fiber contents, and only complete consolidation will ensure accurate assessment of entrained air content, yield, and unit weight in ASTM Test Method for Unit Weight, Yield, and Air Content (Gravimetric) of Concrete (C 138/C 138M), ASTM Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173/C 173M), and ASTM Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231).

Fiber Content

Verification of the fiber content in freshly mixed FRC and its variation from uniformity throughout a truck load or mixer batch prior to placement, or throughout the end product after placement, is a concern in quality control. Proper sampling in accordance with ASTM C 172 and C 1116 is essential. When the in-place uniformity of fiber content is to be determined, a statistically based sampling plan may be appropriate using the principles given in the ASTM Practice for Examination and Sampling of Hardened Concrete in Constructions (C 823).

In North America, the development of standard tests to reliably establish fiber content and its uniformity has not been considered a high priority because of the trend toward performance-based specifications. However, the practice of using relatively low fiber contents of steel fibers (0.25–0.38 % by volume, 20–30 kg/m^3 or 33–50 lb/yd^3) in industrial floors that originated in Europe and is now common in North America has made this issue more important, particularly when large fibers are used and the resulting number of fibers per unit volume of concrete is small. The practice of using very small fiber contents of synthetic fibers primarily to control plastic shrinkage cracking in slabs-on-grade that is now quite common in North America also highlights the issue, particularly when fiber contents are as low as 0.1 % by volume, (about 1 kg/m^3 or 1.6 lb/yd^3 of polypropylene or nylon).

For freshly mixed FRC, washout appears to be the simplest, most direct, and therefore potentially the most accurate method of determining fiber content and its variation within a load or placement unit of FRC. The Japan Society of Civil Engineers standards for testing steel FRC include a Standard Method of Test for Fiber Content of Steel Fiber Concrete, JSCE-SF7, which employs a washout procedure using a container not less than 6 L (0.2 ft^3) in volume [8]. The fibers are collected after washout using a magnet. They are then weighed to determine the weight per unit volume of concrete. An alternative procedure that indirectly determines the fiber content of a 100 by 200 mm (4 by 8 in.) cylindrical sample in a nonmagnetic (paper or plastic) mold employs an electromagnetic induction coil that surrounds the mold. Fiber content is proportional to the induced current, and the standard requires calibration of the apparatus with data obtained using the washout procedure, as shown in Fig. 4 [9]. This procedure is obviously inapplicable for stainless or other alloyed steel fibers that are not magnetic.

Other types of fiber may in principle be isolated by washout and collected by appropriate means, such as floatation in water in the case of polypropylene or other fibers that float. However, there is remarkably little information on this subject.

For routine quality control, it is arguable that if fiber inventory and concrete production are carefully monitored tests for determining fiber content are no more necessary than tests for determining the water and cement contents of conventional concrete. However, when uniformity of distribution is the concern rather than just the average fiber content, such tests become necessary.

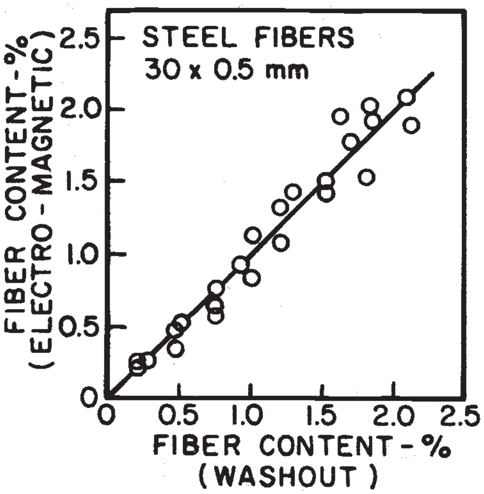

Fig. 4—Steel fiber contents of freshly mixed FRC by electromagnetic and washout techniques [8,9].

Hardened FRC

Fiber Content and Orientation

Verification of fiber content and its uniformity in hardened FRC is possible for some fibers. The electromagnetic technique standardized in JSCE-SF7 [8] is applicable to 100 by 200-mm (4 by 8-in.) cylindrical samples of hardened FRC, either molded specimens or cores, but is of course limited to the magnetic types of steel fiber. Nevertheless, the results are reported to depend somewhat on fiber orientation [8,9] and on proximity of the surrounding coil to the ends of the specimen [9]. The electromagnetic technique can also be employed using a conventional covermeter placed on a specimen surface [10]. Precast concrete products are monitored in this way in Sweden; however, account must be taken of the thickness of the section and the orientation of the fibers [10].

Fiber orientation and uniformity of distribution have been examined using X-ray radiography to produce visual images of the arrangement of steel fibers in a planar cross section of FRC [11,12]. Although apparently only employed with steel fibers, this technique is applicable in principle to any composite where the fiber and matrix densities are significantly different, as between bone and flesh in the human body. It can be useful in relating crack development under load to local deficiencies in fiber content or to fiber orientation predominantly parallel to the crack [12].

X-ray fluorescence spectrometry widely used for elemental analysis of materials has been applied to determining the fiber content of zirconia-based glass fibers in glass fiber-reinforced cement [13]. The measurement was based on detection of zirconia present in a known amount in the parent glass, but there were calibration problems attributed to the matrix and varying water contents in the samples. Interferences from other trace elements such as strontium in the cement are also possible. Nevertheless, it appears that fibers containing known amounts of elements having distinct radiation intensity bands not characteristic of anything in the matrix may in principle be detected and quantified in concentration using this technique.

Clearly, there are no techniques for determining the fiber content of hardened FRC that are conveniently practical and reliable enough to be employed on a routine basis for quality control. The techniques developed to date are too complex or unreliable, or both, but may when appropriately verified have value for investigative purposes, just as the methods for determining the water content and cement contents of conventional hardened concrete have value for investigative purposes but not for routine quality control.

Mechanical Properties (Static Loading)

The role of fibers in hardened FRC is primarily to promote crack distribution and reduce crack widths. Prior to the start of visible and continuous cracking, fibers at the concentrations that are normal in FRC (less than 1 % by volume of concrete) have little effect on mechanical behavior. However, microcracking does occur as the FRC is loaded, and there are characteristic levels of load and deformation at which the FRC eventually starts to exhibit cracks that are significant in continuity, visibility, total length, and width. At this stage, the microcracks have become macrocracks, and acoustic emission measurements confirm that the maximum acoustic event rate corresponds closely to these characteristic levels of load and deformation [14]. The condition is termed “first crack” and is clearly identifiable for direct tension and flexure by a sharp reduction in stiffness (Fig. 5). For compression, where the behavior of both matrix and FRC is often quite curvilinear, first crack is not easily identifiable, but the use of fibers only in zones of compressive stress is usually of less interest than in flexure, tension, or shear.

Strengthening

Below first crack and the associated values of load and deformation, matrix strength contributes substantially to composite FRC strength and stiffness. For FRC in tension and flexure, first-crack strength is a more important and meaningful parameter than the ultimate strength based on maximum load because it corresponds to a specific serviceability condition, that is, the onset of macrocracking. In contrast, the maximum load and any strength based upon it can correspond to widely different serviceability conditions in terms of deflection and cracking, depending on whether the FRC exhibits strain-hardening or strain-softening (Fig. 5).

Increases in first-crack strength attributable to fibers are quite small for both flexure and direct tension, even at high fiber contents well above the 1 % maximum normally possible in practice. For example, for direct tension, strength increases are also modest, only about 25 % for 1.5 % steel fiber content even when the fiber aspect ratio is as high as 100, which is only

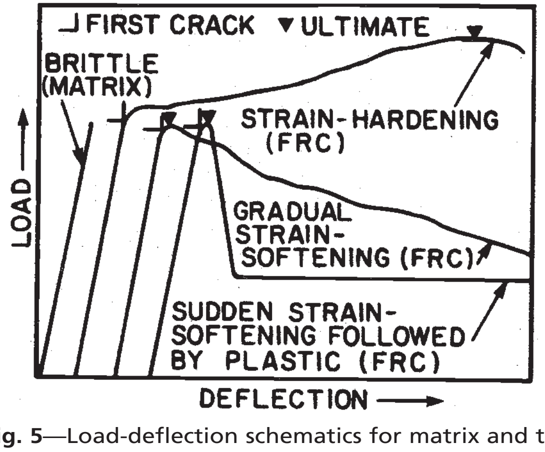

Fig. 5—Load-deflection schematics for matrix and typical FRCs in flexure.

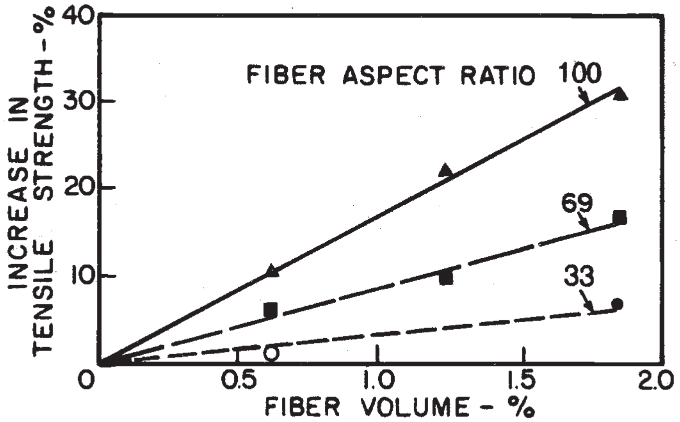

Fig. 6—Strengthening effect of steel fibers in mortar for direct tension [15,16].

possible using a mortar matrix (Fig. 6) [15,16]. Generally, increasing the fiber content or aspect ratio are not nearly as effective as measures to improve matrix strength, for example, the addition of silica fume (Fig. 7) [17]. Clearly, the first-crack strength of FRC in tension or flexure is governed primarily by matrix parameters rather than fiber parameters at the fiber contents possible in concrete in practice.

For compression, the relationship between strength and fiber content is not statistically significant (Fig. 8), confirming the widely held view that fibers have minimal effect on compressive strength. They affect only the strain-softening phase of FRC behavior after the maximum load has been reached (Fig. 8) [18]. Again, matrix strength as determined by matrix parameters (like water-cement ratio, the presence of silica fume, etc.) is the primary factor governing FRC compressive strength.

Toughening

As the load-carrying capability of the matrix becomes progressively reduced by macro-cracking, the behavior of the FRC composite after first crack becomes largely attributable to load carried by the fibers. Depending on fiber type, amount, and aspect ratio, it can vary over a wide range in terms of the degree of strain-hardening or strain-softening that follows first crack, and in terms of the strength retained over any selected interval of deformation (Figs. 5 and 8).

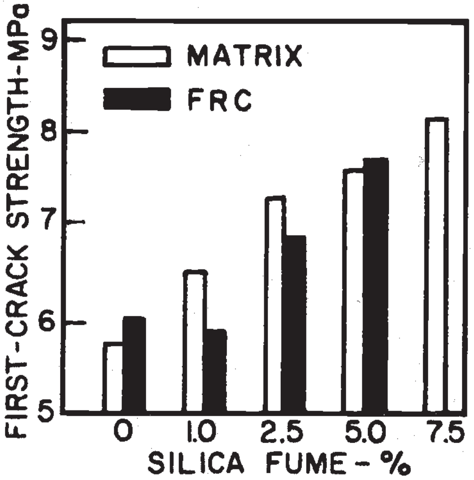

Fig. 7—First-crack flexural strengths of FRC and matrix for concretes with silica fume and 0.5 % steel fibers [17].

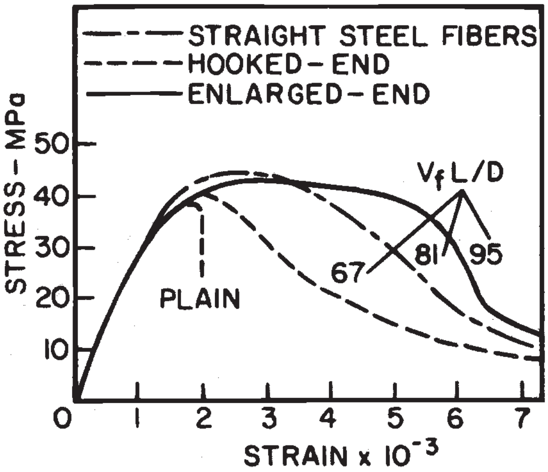

Fig. 8—Typical stress-strain curves for FRC with steel fibers in compression [18].

Toughness, which is determined largely from the area under the stress-strain or load-deformation curve after first crack (Figs. 5 and 8), is the characteristic of FRC that most clearly distinguishes it from concrete without fibers. Its importance is more difficult to relate to engineering design and practice than, for example, flexural or compressive strength, which have long been accepted as the basis for selecting allowable concrete stresses in design and material acceptance in quality control. It is qualitatively demonstrable by comparing the flexural failure patterns of beams with and without fibers (Fig. 9). This manifestation of toughness or energy absorption capability is relevant in some engineering applications, such as anticipated earthquake exposure where preservation of structural integrity even with severe damage is the primary concern. In many other applications where the serviceability conditions are less severe and catastrophic structural damage is not anticipated, performance up to a specified permissible level of serviceability in terms of deformation or cracking may be more relevant. In such cases, retained strength at this serviceability level may be a more useful and acceptable design criterion than toughness. Accordingly, most of the standard tests evaluate the toughening effects of fibers up to specified defor-

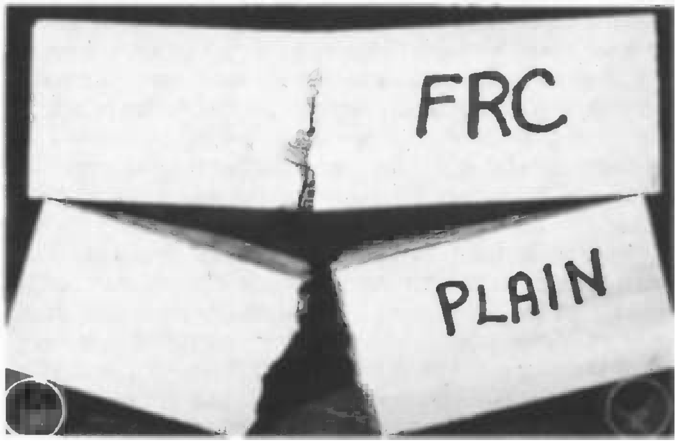

Fig. 9—Toughness of FRC in flexure compared with plain concrete.

mation or load limits rather than to complete failure of the test specimen.

Standardized Tests for Strength and Toughness of FRC

Flexural performance receives the most attention because of its relevance to many FRC applications, as in ASTM C 1018, ASTM C 1399, ASTM C 1550, and the Japan Society of Civil Engineers Method of Test for Flexural Strength and Flexural Toughness of Steel Fiber Reinforced Concrete, JSCE-SF4 [8]. Performance in compression and shear receives less attention, but Japanese standards exist for FRC with steel fibers [8]. They are the Method of Test for Compressive Strength and Compressive Toughness of Steel Fiber Reinforced Concrete, JSCE-SF5, and the Method of Test for Shear Strength of Steel Fiber Reinforced Concrete, JSCE-SF6.

With regard to evaluation of strength, these standards differ considerably in that the test result may be expressed as the strength at first crack, or at the maximum load, or as an average over a prescribed strain or deformation interval. In some of them, the rationale for determining strength after first crack is questionable because load is converted to stress assuming the material behavior is linear elastic, notably in flexure when the maximum load is not reached until well after first crack (Fig. 5) and material behavior is no longer linear elastic.

With regard to toughness, the standards differ in that the test result may be expressed either simply as the area under the load-deformation or stress-strain curve or as a toughness factor or toughness index derived from portions of the area under these curves. The problem with tests that determine only the area under these curves is that the result is specimen-specific, that is highly dependent on specimen size, shape, and loading configuration. Consequently, the test result fails to characterize material behavior in a manner conceptually independent of specimen and testing variables, unlike, for example, compressive strength that has the same significance (but not necessarily the same value) whether determined on a 100-mm (4 in.) cube or a 150 by 300-mm (6 by 12-in.) cylinder.

In most existing standards for toughness testing, the choice of test end-point in terms of deformation or deflection is quite arbitrary and therefore unrelatable to anticipated serviceability conditions. However, in principle, the test end-point may be specified to reflect one or more specific aspects of serviceability. In general, these may be stated qualitatively as somewhere between the two extremes of "very small deformation/deflection and very fine cracks permissible" (for example, in bridge decks with deicer exposure) or "very large deformation/deflection and very wide cracks permissible" (for example, in earthquake loading of a structure or in shotcretes for tunnel or rock slope applications where short-term structural integrity is of paramount importance). Naturally, there is the option to quantify them by specifying numerical limits on deflection or crack width, or both, anywhere within these qualitative extremes.

In the Japanese standard for flexural testing, JSCE-SF4 [8], the test end-point deflection, δ_{tb} (Fig. 10), is specified arbitrarily as $\frac{1}{150}$ of the span. Therefore, there is no possibility of selecting the test end-point to reflect anticipated serviceability conditions. The specimen size, shape, and span limits (only two square cross-sectional sizes, 100 or 150 mm (4 and 6 in.), and spans, 300 or 450 mm (12 or 18 in.), permitted, depending on fiber length) restrict the span-to-depth ratio to 3.0. Since the formula for determining first-crack deflection shows that it is proportional to L^2/D where L and D are the beam span and depth, respectively, it follows that the test end-point deflection represented by $L/150$ is a fixed multiple of the first-crack deflection (approximately 50 depending on the modulus of elasticity and flexural strength of the matrix) so long as the span-to-depth ratio, L/D, is fixed at 3.0 [19]. If L/D changes, the multiple changes in inverse proportion to it, and the area, T_b (Fig. 10), and parameters derived from it do not have the same meaning in terms of end-point serviceability from one specimen shape to another and from one span to another. Consequently, the standard is not adaptable in principle to circumstances where the minimum standard thickness of 100 mm (4 in.) or an L/D of 3.0 are not possible for practical reasons, as in thin specimens representative of shotcrete or bridge deck overlays, or thick specimens sampled from thick FRC placements like pavements. The main parameter derived from T_b, the equivalent flexural strength

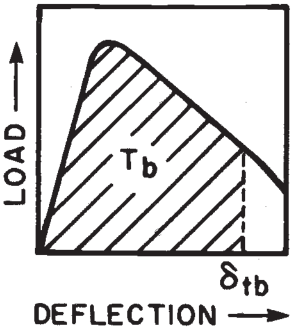

Fig. 10—Toughness in flexure, JSCE-SF4 [8].

$$\bar{\sigma}_b = \frac{T_b L}{\pi_{tb} b D^2}$$

is subject to the same limitations. It represents an average load (T_b/δ_{tb}) for the whole deflection interval, comprising the segments both before and after first crack, converted to a stress using the linear elastic formula for flexural stress. While the load retained at or near a test end-point selected on the basis of serviceability may be useful for design, it is difficult to rationalize how the overall average load can be of direct use in design. Moreover, converting it to a stress using a formula that is clearly invalid after first crack is analytically questionable. Clearly, both T_b and $\bar{\sigma}$ are specimen-specific parameters that cannot reflect FRC material behavior independent of specimen size, shape, and span.

The corresponding standard for compression, JSCE-SF5 [8], is much the same in principle with T_c measured to a limiting deformation, δ_{tc}, corresponding to a strain of 0.75 % (Fig. 11). An equivalent compressive strength

$$\bar{\sigma}_c = \frac{4T_c}{\pi d^2 \delta_{tc}}$$

is derived as the average load divided by the cylinder cross-sectional area to convert it to a stress. Again, it is difficult to rationalize how the overall average rather than the strength retained at or near the test end-point can be useful in design, but the conversion of load to strength is simpler and more justifiable than for flexure.

The standard for shear, JSCE-SF6 [8], provides for double shear of a prismatic specimen (essentially the same as the beams of JSCE-SF4) using an appropriate specified apparatus (Fig. 12).

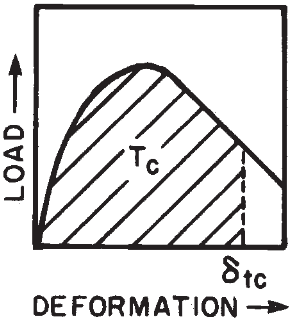

Fig. 11—Toughness in compression, JSCE-SF5 [8].

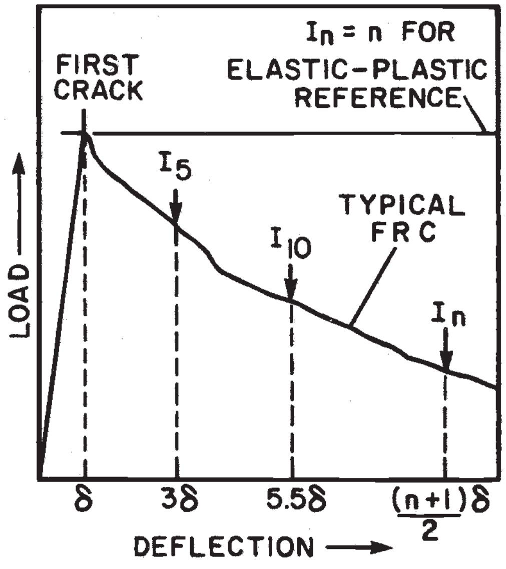

Fig. 13—Flexural toughness indices, ASTM C 1018 [21].

Shear strength is determined simply as load divided by cross-sectional area. No measure of toughness is established in this test.

In the ASTM C 1018 standard for flexural testing of FRC, material performance is characterized in terms of three parameters or sets of parameters defined to ensure in principle their independence of specimen size, shape, span-depth ratio, and loading configuration [19–21]. They are therefore equally adaptable to test specimens representing thin- or thick-section FRC construction. The rationale for defining these parameters provides for selection of the test end-point deflection to reflect a wide range of anticipated serviceability conditions (Fig. 13). It also ensures that each parameter has a readily understandable meaning and significance relative to an established reference level of material performance, elastic-plastic (Fig. 13), typified by mild steel in tension. This is a level of performance to which practicing engineers can easily relate, and one that is readily achievable in FRCs with amounts and types of fibers that produce superior reinforcing effectiveness, for example, 80 kg/m^3 (135 lb/yd^3) of HE 60 fibers in Fig. 14.

The first parameter of importance is the first-crack flexural strength (Fig. 13), which is analogous to the yield point for steel. It represents the maximum stress that can be sustained without serious macrocracking and is largely dependent on the matrix, as previously discussed (Fig. 7).

The second important set of parameters are toughness indices, I_5, I_{10}, I_{20}, etc. (Fig. 13), derived as the area under the curve up to the specified end-point deflection divided by the area up to first crack. The index subscript, the corresponding end-point deflection, and the index value are related in such a way that index subscripts and actual values correspond for the elastic-plastic reference level of material performance, that is, $I_n = n$ (Fig. 13). Index values more or less than the subscript value indicate the average slope of the curve and degree of strain-hardening or strain-softening behavior observed after first crack (Fig. 14). Performance differences attributable to fiber type (Fig. 14) or amount [1] are clearly identifiable, for example, in Fig. 14 between the HE 60 fibers with approximately elastic-plastic behavior ($I_5 = 4.9$, $I_{20} = 20.4$) and the MS fibers with marked strain-softening ($I_5 = 3.8$, $I_{20} = 8.1$).

The third important set of parameters are residual strength factors, $R_{5,10}$, $R_{10,20}$, etc., derived directly from toughness indices. They represent the average strength retained over a specified deflection interval expressed as a percentage of the first-crack strength and are derived so that elastic-plastic or yield-like material performance corresponds to residual strength factors of 100 and fully brittle material performance after first crack to a factor of 0. They are non-dimensional as derived, but are readily converted to a retained load capability using the first-crack strength and its corresponding specimen-

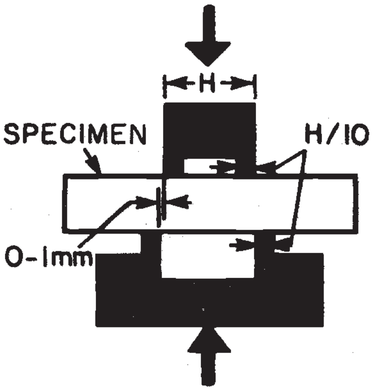

Fig. 12—Shear strength test, JSCE-SF6 [8].

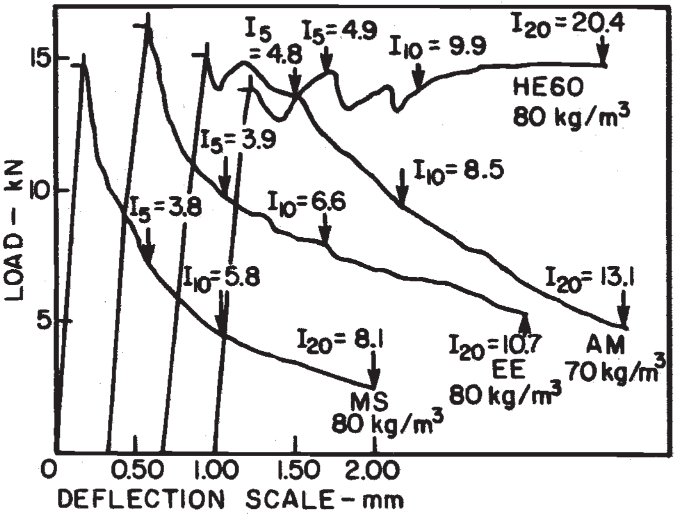

Fig. 14—Influence of steel fiber type on ASTM C 1018 toughness indices for 750 by 150 by 100-mm beams [1].

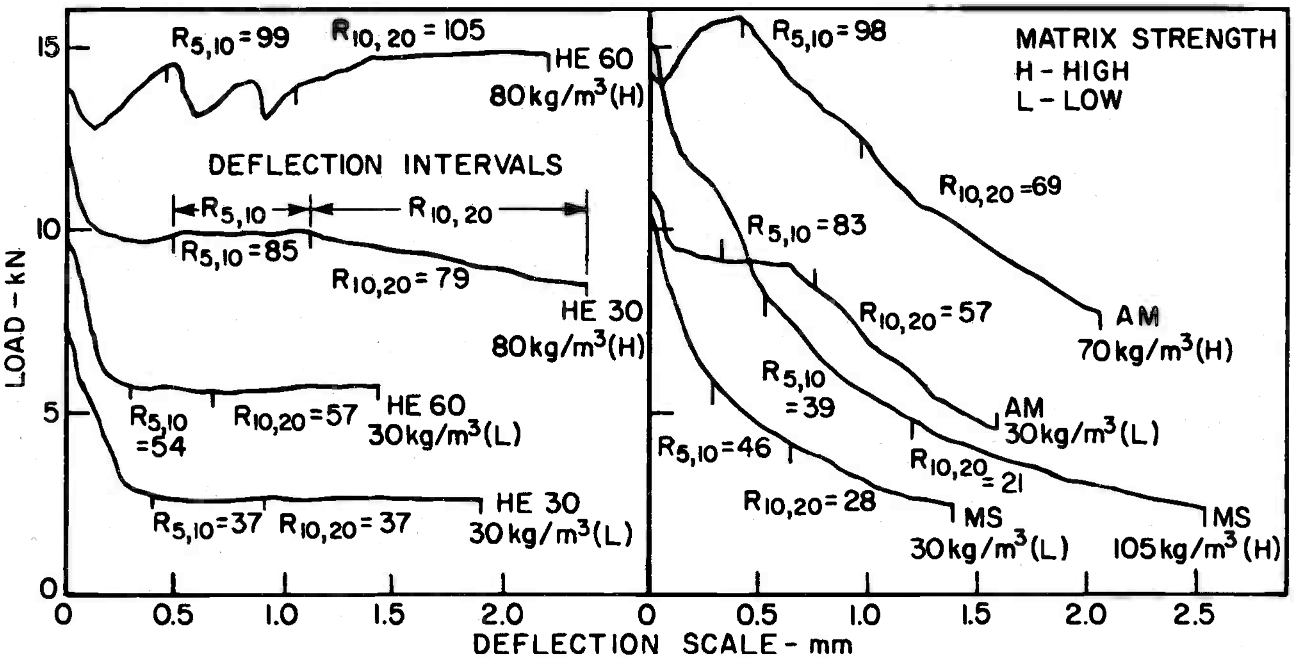

Fig. 15—Influence of steel fiber type and amount on ASTM C 1018 residual strength factors [1].

specific load. Thus, they have the potential for use in design where serviceability limits have been established, and their significance is probably more easily understood by practicing engineers than toughness indices. They are probably most useful for characterizing FRCs that exhibit sudden strain-softening followed by essentially plastic behavior (Fig. 5), where successive factors, $R_{5,10}$, $R_{10,30}$, etc., remain approximately constant and performance differences by fiber type or amount are clearly distinguishable; for example, in Fig. 15 (*left*) between equal amounts of HE 60 and HE 30 fibers and between 30 kg/m^3 and 80 kg/m^3 (50 lb/yd^3 and 135 lb/yd^3) of the same fiber. For FRCs characterized by gradual strain-softening (Fig. 5), residual strength factors decrease with increase in deflection (Fig. 15, *right*), and are probably only useful for design when deflection serviceability limits are known.

ASTM C 1018 requires reporting of first-crack strength; toughness indices, I_5 and I_{10}; and residual strength factor, $R_{5,10}$ as a minimum, and reporting of I_{20} and $R_{10,20}$ is optional. Testing to larger deflections as appropriate to anticipated serviceability conditions is also recommended, and an appendix provides the rationale for establishing appropriate toughness indices and residual strength factors.

In Sweden, task groups dealing with industrial floor design and shotcrete tunnel linings have prepared recommendations giving values of residual strength calculated as the product of first-crack strength and residual strength factors determined as in ASTM C 1018 but on different specimen sizes. In Belgium, the design of FRC slabs based on residual strength factors is also being discussed [22]. In Canada, the ASTM C 1018 toughness indices have been used in design in many FRC shotcrete projects [23]. In Japan, the design of FRC with steel fibers is recommended on the basis of minima for conventional flexural strength and the equivalent flexural strength, $\bar{\sigma}_b$ [8].

When comparing the ASTM C 1018 and JSCE-SF4 standards, it is important to recognize that, in principle and in experimental reality (Fig. 16), it is possible to have two different FRCs with the same T_b and $\bar{\sigma}_b$ at the end-point deflection stipulated in JSCE-SF4. Yet, they can have quite different performance characteristics that are clearly distinguished by all ASTM C 1018 toughness parameters, except perhaps I_5, as shown in Fig. 16, where the end-point deflection is $1/300$ of the span instead of $1/150$ of the span as specified in JSCE-SF4.

In 1997 ASTM C 1018 was updated to require closed-loop, servo-controlled testing machines. In this setup, testing nonfibrous concrete can result in significant values of I_5, and perhaps I_{10}. For this reason the test method is being modified to mediate this abnormality, and users are cautioned to use the I_5 and I_{10} values with this fact in mind until the update is published.

Because ASTM C 1018 is a fairly expensive setup, with few commercial laboratories able to conduct the method, ASTM C 1399 was developed to provide a simpler method to determine the post-cracking load carrying capability of FRC. ASTM C

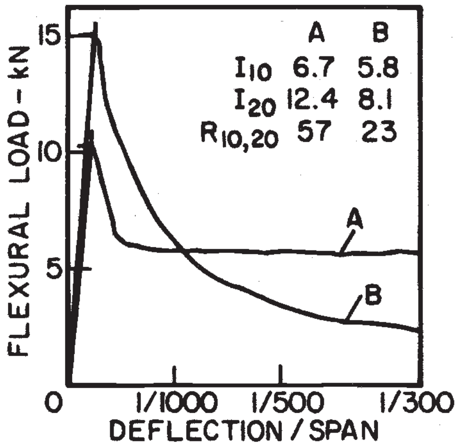

Fig. 16—Equal toughness based on JSCE-SF4 [8] but not in terms of ASTM C 1018 parameters [21].

1399 utilizes testing equipment normally found in most commercial laboratories. The method conditions the specimen by pre-cracking the beam specimen in a controlled manner. Then a load-deformation curve is generated by testing the conditioned specimen. An average residual strength is determined from this curve, with the end-point deformation fixed. Comparison of results using this test method with residual strengths at the same net deflections from Test Method C 1018 have been reported to be an average of 6.4 % lower than Test Method C 1018 test results [24].

A more recent development is ASTM Test Method C 1550, which assesses the toughness of a thin panel in terms of energy absorption. The method was developed initially to monitor performance of thin fiber reinforced shotcrete linings in underground support applications such as deep hard rock mine support, but is applicable to all fiber reinforced concretes. Since this test method utilizes a "structure," the method provides for scaling of results whenever specimens do not comply with the target thickness and diameter within given limits, and thus the results are specimen-specific. Toughness in this test method is defined at various central deflections providing post-cracking flexural behavior assessment for differing serviceability criteria. Results of testing with this method have been used to specify FRC requirements and to monitor FRC compliance with construction specifications, and to compare various fiber-reinforced concretes during the mixture proportioning process or in research and development work.

Mechanical Properties (Dynamic Loading)

Single-Cycle, High-Rate Loading

Uses of FRC that involve resistance to explosives or earthquake loading, or impacts on pavements and bridges due to vehicles or aircraft, may justify impact testing. Weighted pendulum rigs based on modified Charpy impact testers and instrumented drop-weight arrangements with fast-response load and deformation-measuring equipment coupled to appropriate data-acquisition systems have been used in research to evaluate flexural impact performance [25–27]. However, the experimental complexity and expense of the instrumentation needed to eliminate the influence of extraneous energy losses mitigates against widespread acceptance and routine use of such tests in quality control of FRC. Furthermore, comparison of areas under the load-deflection curves for impact and slow flexure shows that the slow (static) flexure test provides a conservative estimate of toughness determined in the more complex and expensive instrumented impact test (Fig. 17) [3,19,25–27]. Impact values can exceed static test values by as little as 20–70 % [25] up to as much as several hundred percent [27], reflecting the widely different strain rates actually imposed by a diversity of impact testing rigs, each with its own equipment-specific strain rate. Strain-rate sensitivity of data may also be affected by the rate sensitivity of the fibers, which probably is greater for polymers like polypropylene than for metals like steel. These factors make it unlikely that a fully instrumented impact test will be standardized for FRC in the near future.

Multiple-Cycle High-Rate Loading

A form of empirical impact drop-weight test is described in ACI 544.2R [6], in which impact is recorded simply as the number of blows sustained by the specimen to first crack or failure. Determination of the energy actually absorbed by the specimen exclusive of extraneous losses to the support system is not possible. This test was proposed as a standard by the ASTM Subcommittee on Fiber-Reinforced Concrete, but was not accepted for several reasons, including its empirical nature and its reportedly very poor precision.

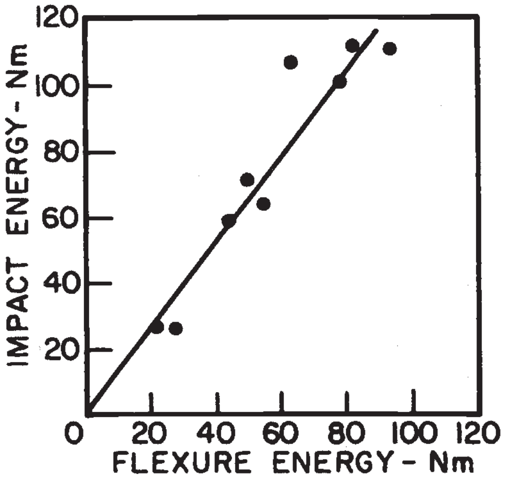

Fig. 17—Energy absorption or toughness compared for impact and slow flexure [19,24].

Uses of FRC in airport and highway pavements subjected to high-frequency traffic loading may justify flexural fatigue testing. Again, the complexity and expense of the equipment and the time needed to conduct such tests mitigate against widespread acceptance and routine use in quality control. Flexural fatigue tests in FRC have shown some benefits attributable to fibers in terms of stress or strength that are to some extent equipment-specific because of differences in testing variables such as reversing/non-reversing stress, stress range, cycling rate, etc. (Fig. 18) [6,28–30]. Incorporating toughness measurements into the test has been attempted [31], but adds further to its complexity. The combined influence of all these factors makes it unlikely that fatigue testing will be standardized for FRC in the near future.

Resistance to Cracking

Cracking can develop in FRC either directly due to application of load, or as a result of loss of moisture and associated differential shrinkage of the exterior of the FRC against restraint provided by the relatively more moist interior.

Cracking Under Restrained Shrinkage

Most investigations have employed a steel ring or cylindrical core around which the FRC is cast and against which it subsequently undergoes drying shrinkage [32–34]. Drying may take place through all three exposed surfaces or may be restricted to the outer one by applying sealant to the others. In all cases, the number, width, location, and spacing of cracks are monitored as drying progresses. Free (unrestrained) shrinkage measurements on companion prisms tested according ASTM Test Method for Length Change of Hardened Hydraulic Cement Mortar and Concrete (C 157/C 157M) are also possible. Typical results show that fibers have much more effect on crack development in the ring test than on free shrinkage (Fig. 19) [32]. Both effects increase with increase in fiber content and aspect ratio (Fig. 19) [32,35]. Since the detection and measurement of very fine cracks depends on the means of detection and magnification available, the development of refinements

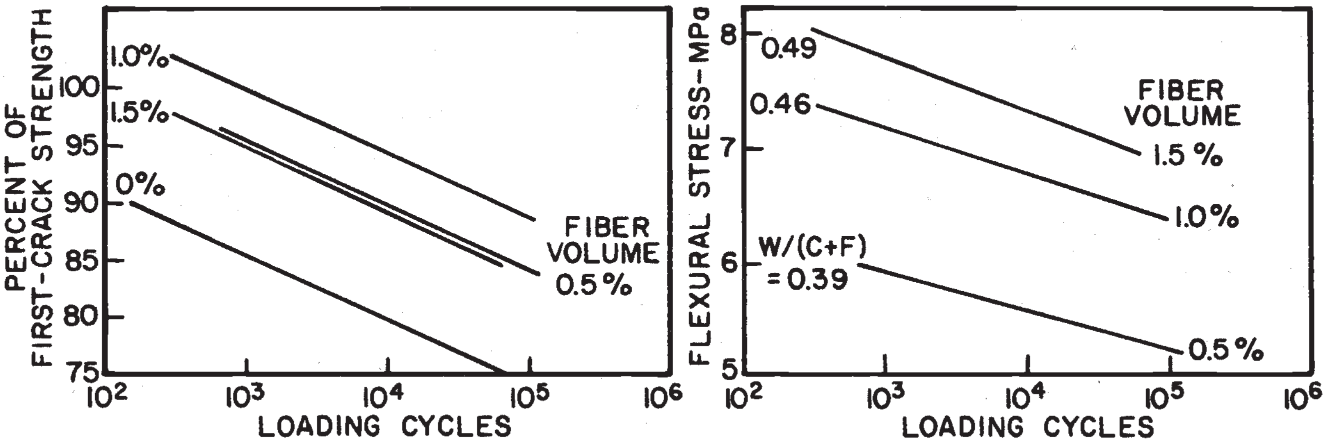

Fig. 18—Flexural fatigue performance of FRC with steel fibers relative to first-crack strength and in terms of actual stress [29].

incorporating laser holography for detection and high-power microscopes for crack width measurement have facilitated more comprehensive and reliable testing [34]. This has helped to quantify the influence of fiber parameters and fiber properties such as modulus of elasticity, *E,* and frictional bond strength, τ_f, on cracking (Fig. 20).

In principle, the ring test can also be used to monitor the plastic shrinkage and associated cracking that sometimes occur within a few hours of placement under adverse conditions of temperature, humidity, and wind speed causing high surface evaporation. Claims by manufacturers that fibers, particularly small volumes of polypropylene or nylon fibers, are effective in preventing this problem are questionable, and there is a need for some form of standard test to confirm such claims [36]. In addition to the ring test, alternatives that require further study are slabs restrained at the edges or fitted with a centrally placed cylindrical insert [37,38]. A standard to assess the plastic shrinkage potential of restrained FRC slabs is being developed by the ASTM Subcommittee for Fiber-Reinforced Concrete. It compares the area of cracking, determined as the product of crack length times width, for fiber-reinforced and unreinforced concrete slabs. It involves fan-forced air flow over the surface of newly molded slabs under controlled conditions of temperature and humidity sufficiently severe to induce plastic shrinkage cracking. Recent results obtained with this method using fibrillated polypropylene fibers of 13-, 19-, and 51-mm (0.5-, 0.75-, and 2-in.) lengths in amounts of 0.05, 0.1, and 0.2 % by volume indicate that plastic shrinkage cracking can be reduced from 20 to 90 %, but was not entirely eliminated under the conditions of the test (water-cement ratio = 0.48, relative humidity = 40 %, and temperature = 35°C (95°F)) [39]. The best results were achieved using 0.2 % of 19- and 51-mm (0.75- and 2-in.) fibers.

Cracking Under Load

Crack widths and spacings between cracks can be measured quite easily with appropriate visual magnification of the cracked surface; for example, on the tension face of a test specimen loaded in slow flexure. Monitoring crack development in this way makes it possible to quantify the ability of fibers to control cracking in concrete, the third benefit imparted by them in addition to strengthening and toughening. It also allows serviceability in terms of crack development to be related to the end-point deflection selected in toughness tests,

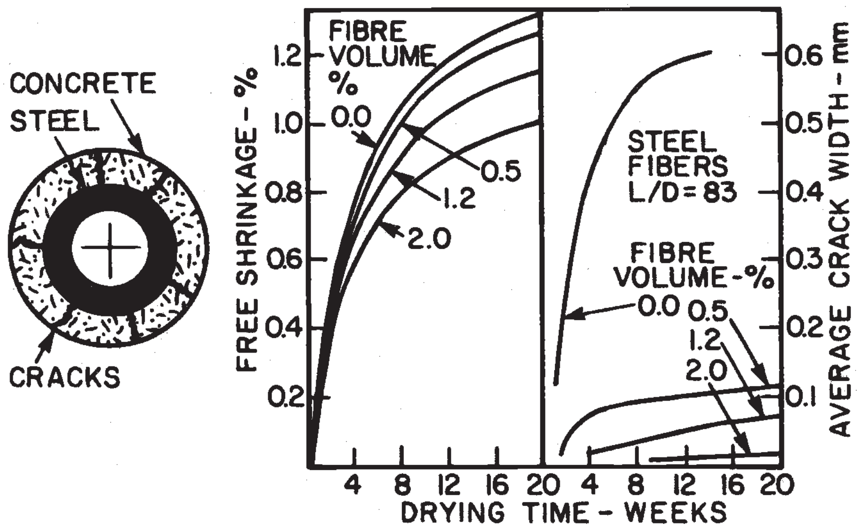

Fig. 19—Free shrinkage and crack development under restrained shrinkage in ring test for matrix and FRC with steel fibers [31].

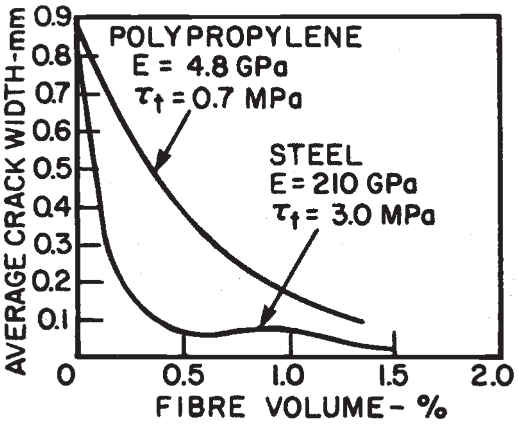

Fig. 20—Crack development compared for 19-mm polypropylene and 25-mm steel fibers in ring test [33].

such as ASTM C 1018 and ASTM C 1550. However, monitoring crack development is not part of the standards, and no comprehensive and conclusive data on the effects of fiber content or type on crack development have yet been obtained, although it seems reasonable to expect such effects from the example shown in Fig. 21 [21].

Durability

No durability tests have been standardized specifically for FRC. The durability of the matrix is assured if it meets the relevant ASTM standards for conventional concrete subjected to freezing and thawing, salt scaling, sulfate resistance, and aggregate reactivity tests. Fiber-specific problems can arise in uncracked FRC if the fibers are chemically incompatible with cement paste or admixtures, and fiber compatibility tests for fibers that may be suspect are being considered by the responsible ASTM subcommittee. In cracked FRC, the fibers may deteriorate in certain exposure environments.

The fibers commonly used in FRC, carbon-steel and polypropylene, pose no problem in uncracked FRC, but, in cracked FRC, steel fibers may rust if exposed to moisture and air and polypropylene fibers may deteriorate if exposed to sufficient ultraviolet radiation. Alloyed steels address the rusting problem, and stabilizing additives may inhibit any ultraviolet attack for polypropylene. As new fibers emerge, there is a need for vigilance and appropriate tests to avoid possible fiber-specific durability problems, such as those that are now well known for glass fibers and many natural cellulose-based fibers and those that remain to be fully investigated for aramid, polyester, nylon, and acrylic fibers [2,3,40]. In the absence of an established satisfactory performance record, ASTM C 1116 requires credible evidence that unfamiliar fiber types will not react adversely with the cementitious matrix or any chemical or mineral admixtures it contains. The ASTM Subcommittee on Fiber-Reinforced Concrete is developing a specification for polymer fibers for use in concrete that will address most of these concerns.

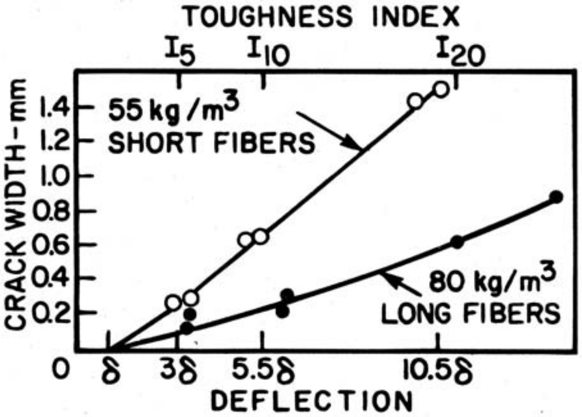

Fig. 21—Crack development in the ASTM C 1018 flexure test for two types of steel fiber [21].

Conclusion

Inconsistent and confusing performance tests in existence diminishes the confidence that potential users of FRC have in this material. The poor reliability of data produced using some of the existing test methods often leads to contradictory outcomes. The arbitrary nature of some performance parameters also obscures the significance of the data. The result is widespread skepticism in the construction industry about performance of FRC that is in sharp contrast to the unquestioning confidence placed in reinforcing bars and tendons [41].

As new fiber types or modifications of existing types may be proposed for use in FRC hindsight suggests that compatibility of fibers with the moist alkaline environment of cement paste and tests to confirm it should be a high priority. The fibers of the future must be engineered to minimize mixing and placement difficulties, maximize short-term property improvements, and ensure that these improvements are sustained over the long term. Much remains to be done in developing standard test methods to assess performance of fibers and FRC relevant to design, and then to develop performance-based specifications for FRC to meet all of these objectives.

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Preplaced Aggregate Concrete

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Preface

THIS CHAPTER ON PREPLACED AGGREGATE (PA) concrete is basically the information from the previous editions of *ASTM 169*. The original chapter was written by B. A. Lamberton. While there have been some improvements in the chemical composition and performance of the grout fluidifiers, the basic method and testing of PA concrete has not significantly changed since this chapter was last updated by Raymond E. Davis, Jr. in *ASTM 169C*.

Introduction

The term "preplaced aggregate" (PA) concrete refers to a method of making concrete that involves placing coarse aggregate in a confined space and later injecting a fluid grout into the voids. The technique has been used since 1937 and is generally credited to Lee Turzillo and Louis Wertz [1]. In 1943 Wertz was granted a U.S. Patent for the method of filling cavities using (PA) concrete [2].

PA concrete is an alternative to blending the fine and coarse aggregate, cement, and water and placing the mixture in a form. In the case of PA concrete the coarse gap-graded aggregate is preplaced in the form and a structural grout is injected into the coarse aggregate mass in such a way as to fill void spaces where it hardens to form dense homogeneous concrete. The drying shrinkage of PA concrete is about 50 % less than that of conventionally placed concrete with similar material proportions [3]. This is due apparently to the point-to-point contact of coarse aggregate particles. Because of this low shrinkage characteristic, it was first used as a method of repairing concrete and masonry structures, particularly railroad bridge piers and tunnel linings [4,5]. The method is now widely accepted for other specialized applications such as placement of mass concrete under water, in heavily reinforced structures, and for high density biological shielding [6–8].

While the two-step placement procedure would increase costs over that of conventional methods in a great majority of routine concrete work, it has the advantage of permitting placement of coarse aggregate, representing roughly 60 % of the total concrete volume, in a timed sequence independent of mixing and placing cementitious constituents. The coarse aggregate fraction can be preplaced either very rapidly with bulk handling equipment or slowly and carefully by hand labor, depending on the application, followed by grout injection at a time convenient to the overall construction schedule. The method is particularly applicable to placement of concrete in structures containing a profusion of inserted fixtures [9].

Materials

The slurry containing a mixture of portland cement, sand, admixtures, and water that is injected into the coarse aggregate mass is referred to as "grout." Actually a highly fluid structural mortar, it bears no resemblance to the low-strength, generally high water/cement (w/c) ratio grouts that are pressure injected into soil or rock formations to increase strength or reduce permeability. Grout used in PA concrete work is a structural material. Injected into coarse aggregate, it produces concrete comparable in strength and other physical characteristics to that mixed and placed by conventional methods. Several manufacturers of fluid grouts that meet the ASTM C1107 Specification for Packaged Dry, Hydraulic-Cement Grout (Nonshrink) claim that their product can be used as the slurry. The same working stresses used in conventional concrete design are applicable to PA concrete.

Cementing Materials

Cementing materials suitable for use in conventionally placed concrete may be used with equal confidence for placement by the PA concrete method. It is customary practice to substitute pozzolanic quality fly ash for part of the portland cement. Substitution in the range of 15 to 35 % by weight of portland cement is common. In addition to the beneficial properties normally imparted by any pozzolan to portland cement concrete, such as lower heat generation, reduced permeability, and improved resistance to chemical attack, fly ash is particularly beneficial in the PA concrete process in that it improves pumpability and retards initial set. Fly ash should meet the Class F requirements of the ASTM C618 Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete.

Fly ash is often omitted when concrete is to be used for high density biological shielding. Since the specific gravity of this material is appreciably less than that of cement, partial replacement of cement with fly ash causes a slight reduction in unit weight of the concrete. Fly ash is also less effective than cement in chemically fixing water and so is undesirable in shielding subject to high neutron flux which is more effectively attenuated by hydrogen atoms.

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Fine Aggregate

Fine aggregate, properly graded, is essential to successful execution of the work. Oversize material will cause obstruction of the void channels in the coarse aggregate mass. Excess fines will increase water requirement and so reduce compressive strength and increase drying shrinkage. Gap-graded fine aggregate may cause excessive bleeding. A typical gradation appears in Table 2, Grading 1 of ASTM C637 Specification for Aggregates for Radiation-Shielding Concrete. Although described in this specification in connection with high-density aggregate for biological shielding, the grading is equally suitable for use with normal-density aggregates. Either natural or manufactured sand may be used, although the former is usually preferable from the standpoint of pumpability. As indicated in Table 2, Grading 2 of ASTM C 637, somewhat finer grading is required where high-density sand is used or where coarse aggregate grading is finer than normal.

Coarse Aggregate

Coarse aggregate grading is far less critical than is fine aggregate grading. The only absolute requirements are that it be (*a*) free of surface dust that would prevent bond of grout to the aggregate particles, (*b*) sufficiently saturated that it will not absorb water from the grout and so cause premature thickening, and (*c*) of such a grading that grout will flow readily by gravity alone through the void system. The particles should be of such toughness and hardness that they do not fracture or degrade during transport and placement into the forms. Extra caution must be exercised during placement to avoid degrading friable aggregate.

Normal aggregate grading limits for most structural applications are shown in Table 2, Grading 1 of ASTM C 637. Aggregate graded within these limits will exhibit a void content in the range of 43 to 48 %. In the absence of very closely spaced reinforcing or restricted form configuration, it is common practice to scalp coarse aggregate on a 20-mm (3/4-in.) wash screen. A trommel-type screen is generally more effective than a deck screen in washing the aggregate particles to the required surface cleanliness.

A wide variety of high-density aggregate, both fine and coarse as described in ASTM C 637, have been placed by the PA concrete method, which is particularly well-suited for construction of high-density biological shielding. The advantage of the method for this type of work is two-fold. The heavy particles of mineral ore or steel punchings or a blend of these coarse aggregates are placed in the form with no possibility of segregation such as may occur when coarse aggregate and grout is combined in a plastic mass and placed by conventional methods. Second, the method is well-suited to concrete placement around multiple embedments, closely spaced reinforcing, and within the tight confines of complex formwork characteristic of most shielding installations.

Grout Fluidifier

Grout fluidifier is required on all PA concrete work to promote fluidity of the grout mixture and, most importantly, to cause expansion of the grout prior to initial set in an amount sufficient to more than offset setting shrinkage of the grout that would otherwise take place beneath coarse aggregate particles. Grout fluidifier is customarily used as a commercially preblended material conforming to ASTM C937 Specification for Grout Fluidifier for Preplaced-Aggregate Concrete. It contains a water-reducing agent, a suspending agent, aluminum powder, and a chemical buffer to assure properly timed reaction of the aluminum powder with the alkalies of the cement. This reaction results in the formation of hydrogen gas to expand the fluid grout, usually in the range of 5 to 8 %. This gas-forming reaction should be so timed that, at 21°C (70°F), it is approximately 50 % complete in 1 h and 95 % complete in 3 h. At a slow expansion rate, bleeding tends to increase. If the expansion time is more rapid, grout mixing and pumping time are shortened unnecessarily.

Since the expansive characteristic of the grout is dependent on the alkali content of the cement, it may be depressed when the standard grout fluidifier formulation is used with low alkali cements. Performance of grout fluidifier in combination with job cement should be determined, therefore, and the product custom formulated by the manufacturer if necessary when use of cement with an alkali content less than 0.40 is anticipated. The release of hydrogen bubbles and the expansive reaction have an effect similar to that of an air-entraining agent with respect to freeze-thaw durability. An air-entraining agent or air-entrained cement should not be used routinely in the PA concrete process. However, where freeze-thaw conditions are severe and a specific minimum air content is required, an air content determination should be made using grout fluidifier and job cement and an air-entraining agent incorporated in the grout mix if required.

Grout Mix Proportions

Grout mix proportions may be selected in accordance with ASTM C938 Practice for Proportioning Grout Mixtures for Preplaced-Aggregate Concrete. For typical structural applications, proportions are normally in the range of 2:1:3 to 4:1:5 by weight in which the ratio represents, respectively, the weights of cement, fly ash, and sand. The ratio of water to cementing materials (cement plus fly ash) is in the range of 0.43 to 0.47. Grout fluidifier is added in an amount equal to 1 % by weight of the cementing materials. For mass concrete applications, the ratio of cementing materials to sand may be reduced to as low as 1:2. Further decrease in this ratio is usually precluded by pumpability limitations.

Specifications

Specifications for PA concrete placement vary widely in detail depending on job complexity. For routine structural repair of concrete or masonry, performance-type specifications normally are employed, requiring only that a minimum stipulated compressive strength be exhibited by test cylinders and that good construction practice be followed in accordance with such accepted publications as ACI Standard 304 [10]. For difficult projects such as large heavily reinforced structures or biological shielding, specifications may be entirely of the prescription type, setting forth not only compressive strength requirements but aggregate composition and grading, grout insert spacing, grout surface monitoring procedures, placement temperature, and in-place density.

High-Density Aggregates

High-density aggregates typically used in PA concrete construction are described in the ASTM C638 Descriptive Nomenclature of Constituents of Aggregates for Radiation-Shielding Concrete and typical specifications for these aggregates are presented in ASTM C 637. All aggregates described in ASTM C 638 are suitable for placement by the PA concrete method, although particular care should be exercised with the softer natural minerals such as barite, geothite, and limonite. Ferrophosphorous varies widely in hardness and some of it may be so soft and friable as to be unsuitable for placement by the PA concrete method. All of these materials are obviously far more expensive than normal density aggregate and purchase specifications for these materials should be written with particular care. They should not only call for specific gravity limitations but also carefully describe sampling procedure with respect to quantity represented by each sample, frequency of sampling, and the location where tests are to be performed. Aggregates should always be tested prior to shipment, with conformance sampling and testing performed at the job site. If the aggregate is relatively friable and breakage during transit is high, consideration should be given to reclaiming undersize material and incorporating it in concrete to be placed by conventional methods.

Where blended aggregates are to be used, such as a combination of iron ore and steel punchings, the two materials must be inspected separately and the final blend again inspected for particle size gradation and homogeneity of the blend. Aggregates may be blended continuously by streaming through metering gates onto a belt ahead of a washing screen that completes the blending process, or they may be batch blended by a few rotations in a concrete mixer followed by washing and screening. Although the latter method may appear to be somewhat more accurate, in practice either type of blending operation causes some size degradation of the more friable of the two aggregate fractions, and allowance must be made for a loss of some of this friable material in the blending operation.

All high-density aggregate should be weighed before placement and sampled frequently to determine unit density and void content. Based on these determinations, calculations can be made to determine required grout density. Where watertight steel forms are used, void content of the entire coarse aggregate mass may be determined in-place by metering water into and out of the aggregate-filled forms. Using careful testing and sampling procedures, it is possible to specify in-place density of concrete placed by the PA concrete method as close as ±1 %, although a tolerance of ±2 % is a more common limitation.

Temperatures

Temperatures at the time of grout injection are often of great importance where minimum thermal shrinkage is an important consideration. The aggregate may be precooled in the form by flooding it with chilled water or covering the coarse aggregate with flaked ice and allowing the ice to melt and drip down through the aggregate mass. Using this technique, in combination with precooling the grout ingredients and using ice in the mixing water, permits placement temperatures to be achieved within the range of 5 ± 3°C (40 ± 5°F).

Grout Surface Monitoring

Grout surface monitoring is of particular importance in ensuring complete penetration of all voids within the coarse aggregate mass and complete contact with embedments and penetrations. For routine repair work and for many structural concrete applications, location of grout surface can be determined with sufficient accuracy by simply observing grout or moisture seepage through the forms or through insert or inspection holes drilled in the side of the forms. Slotted sounding wells may also be used, particularly for deep mass concrete placements, in which a float on the end of a line is dropped into the sounding well and the depth to the grout surface physically measured. Using such methods, location of the grout surface can be determined with an accuracy of about ±300 mm (±12 in.). For critical applications, location and frequency of sounding wells may be specified, as well as location and spacing of grout insert pipes and vent pipes. The Time Domain Reflectometer provides a more accurate means of determining grout surface location in a nonflooded aggregate mass. Short voltage pulses are transmitted on electrically calibrated detector wires located within the aggregate mass and the pulse reflections assembled and displayed on an oscilloscope. Grout or water around the sensor wires alters the impedance permitting an experienced operator to locate the grout surface with an accuracy of about ±80 mm (±3 in.) [10].

Fluid Grout Characteristics

Fluid grout characteristics are always routinely tested during any placement by the PA concrete method. Measurements of grout viscosity or flow, measuring the time in seconds for discharge of 1725 cm^3 of grout through a 12.7-mm (1/2-in.) orifice in accordance with ASTM C939 Test Method for Flow of Grout for Preplaced-Aggregate Concrete (Flow Cone Method) should be made routinely at about 2-h intervals. This test is analogous to the slump test for conventionally placed concrete. Typical consistencies range from 21 to 30 s with the upper end of the range suitable for use with plus 20 mm (3/4 in.) coarse aggregate and the lower end of the range required for plus 12 mm (1/2 in.) coarse aggregate. Specifications usually require grout consistency to be controlled within a range of ±2 of a stipulated value.

The test for expansion in accordance with ASTM C 940 Test Method for Expansion and Bleeding of Freshly Mixed Grouts for Preplaced-Aggregate Concrete in the Laboratory is performed less frequently, often no more than once a day. This test requires only observation of total grout expansion 3 h after mixing. The utility and significance of the test procedure is greatly enhanced if observations are made of both total grout expansion and accumulated bleed water at four 15-min intervals following mixing and at 30-min intervals thereafter until all expansion has ceased. Although with normal cement and at normal temperature, expansion is virtually complete at 3 h, expansion may continue as long as 4 h at lower temperatures and with some low alkali cements.

Following the procedures of ASTM C 940, approximately 800 cm^3 of fresh grout are placed in a 1000-cm^3 graduate. The observer should then note and report not only the total volume of the specimen, including grout and accumulated bleed water, at the aforementioned intervals, but the total volume of grout and the total volume of bleed water. Expansion, expressed in percent, is calculated as total volume minus initial volume divided by initial volume. Bleeding, expressed in percent, is reported as the volume of bleed water (total volume minus grout volume) divided by initial volume.

Water retentivity of freshly mixed grouts should be determined in accordance with ASTM C 941 Test Method for Water Retentivity of Grout Mixtures for Preplaced-Aggregate Concrete in the Laboratory. Grout bleeding, grout expansion, and grout consistency are all strongly influenced by the effectiveness of mixing equipment and procedures. Before any adjustment is made to the mix design, adequacy of the mixing procedure should be evaluated carefully.

Compressive Strength

Compressive strength of hardened grouts should be determined in accordance with ASTM C 942 Test Method for Compressive Strength of Grouts for Preplaced-Aggregate Concrete in the Laboratory.

Time of Setting

Time of setting of grouts should be determined in accordance with ASTM C 953 Test Method for Time of Setting of Grouts for Preplaced-Aggregate Concrete in the Laboratory.

Density

Density of hardened concrete is normally determined by casting test cylinders in accordance with ASTM C 943 Practice for Making Test Cylinders and Prisms for Determining Strength and Density of Preplaced-Aggregate Concrete in the Laboratory. From these test cylinders, 100 by 100 by 150-mm (4 by 4 by 6-in.) prisms, on which final acceptance tests are performed, are cut. Density determinations on the standard 150 by 300-mm (6 by 12-in.) test cylinder will result in an erroneously low figure. This procedure is particularly important in counterweight construction where excessive density may be as objectionable as deficient density.

Hardened Concrete

Hardened concrete placed by the PA method is evaluated by the same methods employed with conventionally placed concrete. However, two notes of caution are in order. In accordance with good practice, cores extracted from conventionally placed concrete should be of a diameter equal to at least four times that of the maximum coarse aggregate particle size. This limitation on minimum core diameter is even more important with PA concrete by reason of the fact that there is a higher percentage of large aggregate particles with a correspondingly greater possibility that these particles will be torn loose from the grout matrix. Secondly, nondestructive testing procedures such as the Schmidt hammer and the Windsor probe should be used and interpreted with particular caution. Because of the fact that densely packed large coarse aggregate particles are in close proximity to the formed surface, covered only with a thin layer of structural mortar, such tests may show a substantial variation in strength between what is essentially a test on an aggregate particle and another test, fractions of an inch away, performed on the structural mortar. Discrepancies in test results might be expected, for example, on attempting to determine 28-day strength on concrete placed by the PA method using very dense aggregate and grout with a relatively high percentage of fly ash replacement of the cement.

Closure

The PA concrete process is an internationally accepted method of concrete placement appropriate to certain specialized construction problems. Test methods for evaluating the properties of the fresh grout and coarse aggregate fractions employed in this method are well established.

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Roller-Compacted Concrete (RCC)

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Preface

THE ORIGINAL CHAPTER ON ROLLER-COMPACTED concrete was authored by Kenneth L. Saucier with the U.S. Army Engineer Waterway Experiment Station and first appeared in the previous edition of *ASTM STP 169C* in 1994. Much of the content of the original work was drawn on in preparing the current edition. The most significant changes are in the sections on mixture proportioning, durability, construction, and quality control.

Definition

The American Concrete Institute (ACI) in *Cement and Concrete Terminology* (ACI 116R-99) defines roller-compacted concrete (RCC) as, "concrete compacted by roller compaction; concrete that, in its unhardened state, will support a roller while being compacted" [1]. RCC can further be defined as a stiff, extremely dry concrete that has the consistency of damp gravel. It is typically mixed using high-capacity continuous mixing or batching equipment, delivered with trucks or conveyors, and spread, in the case of mass concrete such as dams, with one or more bulldozers. For paving applications, RCC is spread with heavy-duty asphalt type pavers. Large vibratory rollers are used to externally consolidate or compact the roller-compacted concrete. Properties of fully compacted, hardened RCC are similar to those of conventionally placed concrete. However, the low-water content and absence of entrained air in most RCC affects some physical properties such as shrinkage and freeze-thaw durability.

Introduction

RCC may be considered for applications where no-slump concrete can be transported, placed, and compacted using earth- and rock-fill construction equipment or, in the case of pavements, asphalt laydown equipment. Ideal RCC projects will involve large placement areas with few interferences or discontinuities or restrictions on placement rate. Application of RCC is often considered when it is economically competitive with other construction methods. The two major applications for RCC are for mass concrete such as dams and heavy-duty pavement applications including intermodal yards, port facilities, warehouse and other industrial parking and storage areas. Other applications include overtopping protection for earth fill dams, buttressing of existing concrete dams, grade control structures in riverbeds, low permeable liners, and a variety of pavement applications.

Dams

RCC developed as a result of efforts to design and build concrete dams that could be constructed rapidly and economically. At the Rapid Construction of Concrete Dams Conference in 1970, Raphael [2] presented a paper in which he extrapolated from soil-cement applications the concept of placement and compaction of an embankment with cement-enriched granular bank or pit-run material using high-capacity earthmoving and compaction equipment. He noted that the increase in shear strength of cement-stabilized material would result in a significant reduction of the cross section when compared with a typical embankment dam and that use of continuous placement methods, similar to those used in earth dams, would generate savings in time and money as compared with traditional concrete gravity dam construction.

In 1972, Cannon [3] presented results of tests on a lean concrete using 75-mm maximum size aggregates transported by truck, spread by a front-end loader, and compacted by a vibratory roller at a Tennessee Valley Authority (TVA) project. The U.S. Corps of Engineers (USCE) soon thereafter constructed RCC field test sections at Jackson, Mississippi [4] and Lost Creek Dam in Oregon [5] in 1972 and 1973.

The 52-m-high Willow Creek Dam confirmed the economy and rapid construction possible with RCC. The structure contained 330 000 m^3 of RCC and was placed in less than five months. The in-place RCC cost averaged about \$26 per m^3 when considering all the different mixes used [6]. The U.S. Bureau of Reclamation's (USBR) 90-m high Upper Stillwater Dam, completed in 1987, contains 1.12 million m^3 of RCC placed within horizontally slip-formed, air-entrained concrete facing elements [7].

Worldwide, there are more than 280 RCC dams in 39 countries. Forty-seven of these dams are greater than 90-m high and located predominantly in Japan and China. The United States has 37 RCC dams with the highest being Olivenhain Dam in San Diego, CA. Completed in 2003; the dam is 97-m high and contains 1070 m^3 of RCC. Worldwide the highest RCC dam is Miel I in Columbia at 188 m. [8]. Figure 1 shows construction of the 40-m high C. E. Siegrist Dam in Lebanon, PA.

In addition to new dams, RCC has also been used extensively in the rehabilitation of existing dams. Applications include increasing spillway capacity for earth fill dams, grade

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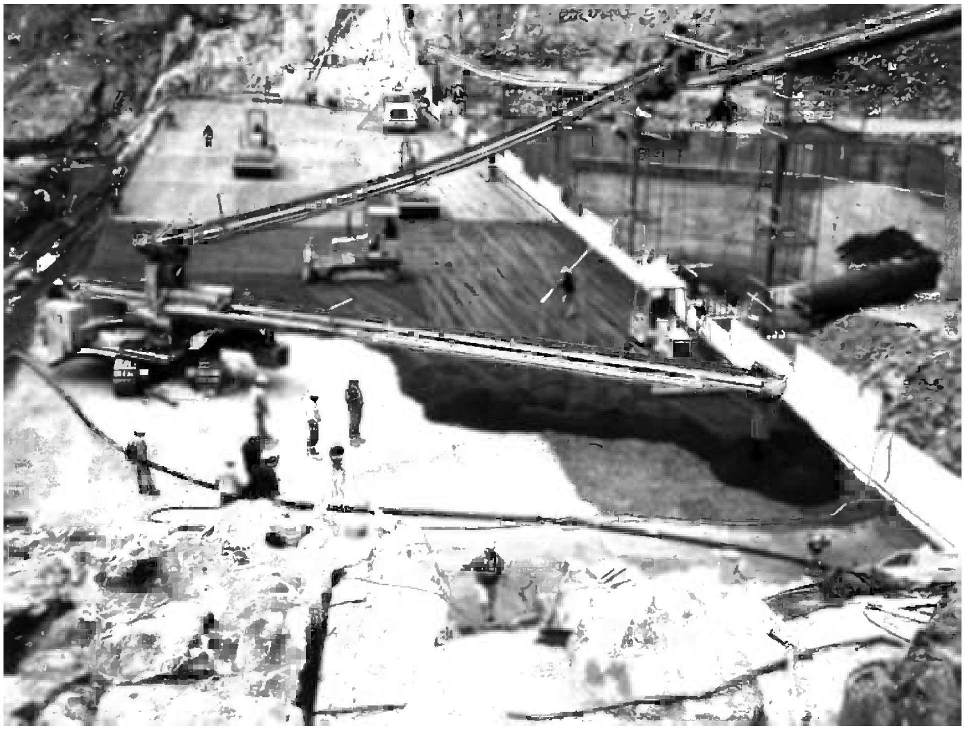

Fig. 1—Construction of C. E. Siegrist Dam, PA. Note use of conveyors to place roller-compacted concrete (photo courtesy of Gannett Fleming).

control structures in rivers, and seismic reinforcement for existing concrete dams. The Tennessee Valley Authority in 1980 was the first to use RCC as overtopping protection to rehabilitate Ocoee Dam No. 2. Since then, RCC has been used in more than 200 dam rehabilitation projects. Information on designing RCC for spillways and overtopping protection applications can be found in Ref 9.

Pavement

The use of RCC for pavements evolved from the use of soil cement and cement-treated base (CTB) material. Although equipment for batching, mixing, and transporting roller-compacted concrete is similar to CTB, RCC is designed to have the strength of conventional concrete. RCC has considerably more cementitious material than CTB, and differs from most soil cement in that it contains a well-graded coarse and fine aggregate. To enhance surface texture, the maximum size aggregate is limited to 16–19 mm. In addition, most RCC pavement projects are placed with heavy-duty asphalt type pavers. According to the Seattle office of the U.S. Corps of Engineers (USACE), the first use of RCC pavement in North America was a runway at Yakima, WA, constructed in 1942 [10]. The USACE at Vicksburg, Mississippi installed the first known RCC test pavement in the United States in 1975. This 4-m by 80-m service road proved the feasibility of RCC for use in pavement construction [11]. The first use of RCC pavement in Canada was built in 1976 at a log-sort yard at Caycuse, British Columbia. The project included 16 350 m^2 of 0.35-m-thick RCC pavement placed in a two-lift operation on a crushed-rock base. The yard size was doubled in 1979 with a second RCC application. When inspected in 1984, these pavements were in excellent condition [12].

In 1984 the Corps of Engineers constructed the first significant RCC pavement in the United States at Fort Hood, Texas. This was a large parking area for tanks and other tracked vehicles surrounding a maintenance shop. The 15 000 m^2 area of 0.25-m pavement was placed in one lift and achieved a flexural strength of 5.5 MPa. [12]. In general, RCC has been used for heavy-duty pavements such as tank hardstands, log handling yards, intermodal yards, freight depots, and other special applications. However, in the past ten years RCC has also been proven to be a cost-effective pavement for many conventional pavement applications including warehouse facilities, industrial access roads, large commercial parking areas, intersection replacements, roadway inlays, and residential streets.

Two of the largest paving projects to date have been for the auto industry. The Saturn automobile plant in Tennessee was completed in 1989. Approximately 500 000 m^2 of a 180-mm-thick pavement was placed for parking areas and access roads [13]. Approximately 830 000 m^2 of 175-mm-thick pavement was placed in 2003 for the Honda manufacturing facility in Alabama. According to the contractor on this project, RCC typically costs between \$19 and \$24 per m^2 [14].

Advantages

The primary advantage of RCC over conventional construction is in the speed of construction and cost savings. Construction cost histories of RCC and conventional concrete show that the unit cost per cubic meter of RCC is considerably less than conventionally placed concrete. Approximate costs of RCC pavement range from 20–30 % less than conventionally placed concrete [12,15,16]. The difference in percentage savings usually depends on complexity of placement and on total quantities of concrete placed. Savings associated with RCC are primarily due to reduced cement content, forming, placement, and compaction costs, as well as reduced construction times. To achieve the highest measure of cost effectiveness and achieve a high-quality product similar to what is expected of conventional concrete structures, the following design and construction objectives are desired: (1) RCC should be placed as quickly as practical after mixing; (2) operations should include as few laborers as possible; (3) design should avoid, as much as possible, multiple mixtures and other construction or forming requirements that tend to interfere with production; and (4) the design should not require complex construction procedures.

Typically, RCC needs no forms or finishing. In the case of pavements, there are no dowels, tie rods, or steel reinforcement. To minimize the treatment of cold joints, rapid placement is desired. In pavements, that may mean the use of two paving machines working in tandem. With dams and other mass placement projects, the contractor will typically run two 10-h shifts per day, six days a week. At Olivenhain Dam, a record placement rate of 224 800 m^3 per month was achieved [17]. High production rates make dam construction in one season readily achievable for even large structures. When compared to embankment or conventional concrete dams, construction time for large projects can be reduced by one to two years. Other benefits from rapid construction include reduced administration costs, reduced risk of flooding, and earlier operation of the facility. Basically, RCC construction offers economic advantages in all aspects of construction that are related to time.

Materials

Cementitious Materials

RCC can be made with any of the basic types of hydraulic cement or a combination of hydraulic cement and pozzolan. Selection of materials for chemical resistance to sulfate attack, potential alkali reactivity, and resistance to abrasion with certain aggregates should follow procedures used for conventional concrete construction.

The strength of RCC is primarily dependent on the quality and gradation of the aggregate, degree of compaction, and the proportions of cement, pozzolan, and water. The type of cementitious material has a significant effect on the rate of hydration and the rate of strength development and, therefore, significantly affects strengths at early ages.

Cement

RCC can be made using any of the basic types of portland cement given in ASTM Specification for Portland Cement (C 150) or blends of these with ground granulated blast-furnace slag as specified in ASTM Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete and Mortar (C 989). To minimize thermal cracking in mass applications, portland cements with lower heat-generation characteristics than Type I are often specified. They include Type II (moderate heat), Type IP (portland-pozzolan cement), and Type IS (portland blast-furnace slag cement). Type IV (low-heat) cement is not generally available in the United States. Before specifying a low-heat type cement, the engineer should determine its availability in the project area. Also the strength development for these lower-heat cements is usually slower than for Type I.

Pozzolans

The selection of a pozzolan suitable for RCC should be based on its conformance with ASTM Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete (C 618). Class F and Class N pozzolans are usually preferred especially for dams, since they normally contribute less heat of hydration than Class C and have greater sulfate resistance. They also help reduce expansion due to alkali-silica reactivity (ASR). For Class C pozzolans, more attention may be needed with regard to set time, sulfate resistance, ASR and free lime content. Use of pozzolans in RCC mixtures may serve one or more of the following purposes: (1) as a partial replacement for portland cement to reduce heat generation; (2) to reduce cost; and (3) as a mineral filler to provide supplemental fines for mixture workability and paste volume. The use of pozzolan will depend on required material performance as well as on its cost and availability at each project [18].

Aggregates

As with conventional concrete, aggregates for RCC should be evaluated for quality and grading. Aggregate for RCC should meet the same standards for quality and grading as required for conventional concrete construction. However, aggregates that do not meet the normal standards or requirements for conventional concrete have also been successfully used in RCC dam construction [19]. Changes from the grading or quality requirements of ASTM Specification for Concrete Aggregates (C 33) should be supported by laboratory or field test results that show that the concrete produced from the proposed materials fulfills the requirements of the project as is provided for in ASTM C 33.

Early RCC mass concrete dam projects in the United States used a 75-mm nominal maximum size of aggregate (NMSA); however, a 50-mm NMSA, preferably crushed coarse aggregate, is less prone to segregation and is becoming more widely used. Although larger sizes have been successfully used in Japan and at Tarbela Dam in Pakistan, the use of larger aggregate greatly increases the probability of segregation during transporting and spreading, and seldom significantly reduces the cost [15].

Maximum size aggregate for other applications include overtopping protection for embankment dams, which frequently use a NMSA of 25 mm, since lifts are typically thinner than for mass concrete placement [18]. For RCC pavement projects a NMSA of 16–19 mm is typically specified. In addition to minimizing the chance for segregation during handling and placement, a smaller NMSA provides a relatively smooth pavement surface texture.

Grading

The grading limits of individual coarse aggregate size fractions should comply with those used in conventional concrete. Individual size groups are normally combined to produce gradings approaching those given in Table 1. Fine aggregate gradings

TABLE 1—Ideal Coarse Aggregate Grading [15]

	Cumulative Percent Passing		
Sieve Size (mm)	**4.75–75 mm**	**4.75–50 mm**	**4.75–19.0 mm**
75	100		
63	88		
50	76	100	
37.5	61	81	
25.0	44	58	
19.0	33	44	100
12.5	21	28	63
9.5	14	18	41
4.75	...	...	...

TABLE 2—Fine Aggregate Grading Limits

Sieve Size	Cumulative Percent Passing [15]	Cumulative Percent Passing [ASTM C 33]
9.5 mm	100	100
4.75 mm	95–100	95–100
2.36 mm	75–95	80–100
1.18 mm	55–80	5–85
600 μm	35–60	2–60
300 μm	24–40	5–30
150 μm	12–28	0–10
75 μm	6–18	...
Fineness modulus	2.10–2.75	...

are also specified as shown in Table 2. Approximate fine aggregate contents, expressed as a percentage of the total aggregate volume, for mass RCC are given in Table 3. Typical gradation range for RCC pavements is shown in Table 4.

Some designers, however, have used locally available road base material with grading requirements similar to that contained in ASTM D 2940. However, the grading band for road base material can be quite open resulting in possible gap grading and segregation. Where close control of grading of the coarse aggregate and RCC production are desired, size separations should follow normal concrete practice, as recommended in ACI 304R.

The required amount of material passing the 75 μm may be greater for RCC than acceptable for conventional concrete. The larger percentage of fines is used to fill voids and contribute to compactibility. The additional fines are usually made up of naturally occurring non-plastic silt and fine sand, manufactured fines, or extra pozzolan. Depending on the volume of cementitious material and the NMSA, the required total minus 75-μm fines may be as much as 10 % of the total aggregate volume, with most mixtures using approximately 3–8 % [18].

Admixtures

Water-Reducing and Retarding Admixtures

The use of a water-reducing and retarding admixture or a retarding admixture as specified in the ASTM Specification for Chemical Admixtures for Concrete (C 494) may be considered for any RCC placement. Water-reducing and retarding admixtures have proven beneficial for improving and extending the workability of RCC beyond the typical 45 min to 1 h specified on most projects. The extended workability is especially beneficial during warmer weather, during RCC startup activities, longer haul distances, and for placement of thick lifts. It is also beneficial in maintaining lift surfaces in an unhardened state until the next layer or adjacent layer of RCC is placed, thereby creating a better bond. By improving the workability, RCC can be more easily mixed in conventional central plant drum mixers and transit truck mixers. Required dosages of water-reducing and retarding admixtures are normally several times as much as recommended for conventionally placed concrete.

TABLE 3—Approximate Ratio of Fine to Total Aggregate Volume [15]

Nominal Maximum Size and Type of Coarse Aggregate	Fine Aggregate Ratio, Percent of Total Aggregate Volume
75 mm, crushed	29–36
75 mm, rounded	27–34
37.5 mm, crushed	39–47
37.5 mm, rounded	35–45
19.0 mm, crushed	48–59
19.0 mm, rounded	41–45

Air-Entraining Admixtures

Air-entrainment of RCC has had only limited application to date. Most of the problem comes from the difficulty of entraining a good air-void system in such a low-paste, dry concrete. Research has indicated that air-entrainment may be limited to the more workable mixes with Vebe consistency times less than about 35 s [30,35]. Also, ASTM Test Method for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173) and Test Method for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231) determine total air content and do not differentiate between entrained and entrapped air voids. The entrapped air content in RCC mixtures will vary depending on the compactive effort applied in consolidating the material.

Minimizing frost damage in RCC has been achieved by proportioning mixtures with sufficient low-water-cementitious material ratios (w/c) so that the permeability of the paste is low. Once concrete has dried through self-desiccation, it is difficult to again become critically saturated by outside moisture. The use of proper compaction techniques that lower the entrapped air-void content, increase strength, and lower the permeability of the concrete should also improve the pavement's frost resistance [10].

Mixture Proportioning

As with conventional concrete construction, the primary considerations for mixture proportioning are durability, strength, workability, and, in the case of RCC, compactibility. Another important consideration for mass RCC is the minimization of heat rise due to the chemical reactions of the cementitious ingredients. Again, as with conventional mass concrete, factors such as use of (1) the largest nominal maximum-size of aggregate; (2) minimum amount of cementitious material; (3) pozzolans or

TABLE 4—Typical Combined Aggregate Grading Limits for RCC Pavement Mixture [20]

Sieve Size	Cumulative Percent Passing
19.0 mm	83–100
12.5 mm	72–93
9.5 mm	66–85
4.75 mm	51–69
2.36 mm	38–56
1.18 mm	28–46
600 μm	18–36
300 μm	11–27
150 μm	6–18
75 μm	2–8

blended cements; and (4) cooling procedures for the materials are evaluated on a job-specific basis.

A number of mixture-proportioning methods have been successfully used for RCC structures throughout the world, making it difficult to generalize any one procedure as being standard. Most mixture-proportioning methods are variations of two general approaches: (1) a w/cm approach with the mixture determined by solid volume; and (2) a cemented-aggregate approach with the mixture determined by either solid volume or moisture-density relationship. ACI 207.5R discusses four predominant mixture-proportioning methods:

Corps of Engineers Method—This proportioning method is based on w/cm and strength relationship. The method calculates mixture quantities from solid volume determinations, as used in proportioning most conventional concrete. The approximate water demand is based on nominal maximum size aggregate and desired modified Vebe time. A recommended fine aggregate as a percentage of the total aggregate volume is based on the nominal maximum size and nature of the course aggregate. Once the volume of each ingredient is calculated, a comparison of the mortar content to recommended values maybe made to check the proportions.

High Paste Method—This method results in mixtures that generally contain high proportions of cementitious materials, high pozzolan contents, clean and normally graded aggregates and high workability. The optimum water, fine aggregate, and coarse aggregate ratios are determined by trial batches Vebe consistencies are typically determined in accordance with ASTM Test Method for Determining the Consistency and Density of Roller-Compacted Concrete (C 1170). The major advantage of the high paste method is to provide excellent lift-joint bond strength and low joint permeability by providing sufficient cementitious paste in the mixture to enhance performance at the lift joints.

Roller-Compacted Dam Method—The roller-compacted dam (RCD) method is used primarily in Japan. The method is similar to proportioning conventional concrete in accordance with ACI 211.1 except that it incorporates the use of a consistency meter. The procedure consists of determining relationships between the consistency, termed VC value, and the water content, sand-aggregate ratio, unit weight of mortar, and compressive strength. Because of the consistency test equipment requirements and differences in the nature of RCD design and construction, this method is not widely used in proportioning RCC mixtures outside of Japan.

Maximum Density Method—This method is a geotechnical approach similar to that used for selecting soil-cement and cement stabilized base mixtures. Proportioning by this approach is also covered in Appendix 4 of ACI 211.3. Instead of determining the water content by Vebe time or visual performance, the desired water content is determined by moisture-density relationship of compacted specimens, using ASTM Test Method for Laboratory Compaction Characteristics of Soil Using Modified Effort (D 1557).

Another method for proportioning nonair-entrained RCCP mixtures is referred to as the optimal paste volume method. The premise behind the method is that workability and strength requirements are treated in two independent steps. The method is based on the assumption that an optimal RCC should have just enough paste to completely fill the interstices remaining when the granular skeleton has reached its maximum density under compaction.

The procedure includes three major steps. The first step is to select an aggregate grading that contains a minimum volume of voids for a given compaction energy. The next step is to adjust the paste volume to obtain the required workability. The final step involves the selection of the w/cm ratio and the proportions of cement and pozzolanic materials to produce a paste with enough binding capacity to satisfy the strength requirements [21,22].

All of the methods include the preparation of trial mixtures to confirm that the workability, compactibility and engineering properties are suitable for the particular project. This is usually confirmed in a test section using the placing methods and equipment that are planned for use on the job. If the laboratory-proportioned mixture proves unsuitable for construction, the mixture is adjusted accordingly. Although mixture proportions are project-specific, Table 5 provides typical values for estimating RCC trial mixture proportions.

TABLE 5—Typical Values for Estimating RCC Trial Mixture Proportions [15]

	Nominal Maximum Size of Aggregate[a]					
	19.0 mm		50 mm		75 mm	
Contents	Average	Range	Average	Range	Average	Range
Water content[b], kg/m^3						
a) Vebe <30 sec	150	133–181	122	107–140	107	85–128
b) Vebe >30 sec	134	110–154	119	104–125	100	97–112
Sand content, % of total aggregate volume						
a) crushed aggregate	55	49–59	43	32–49	34	29–35
b) rounded aggregate	43	38–45	41	35–45	31	27–34
Mortar content, % by volume						
a) crushed aggregate	70	63–73	55	43–67	45	39–50
b) rounded aggregate	55	53–57	51	47–59	43	39–48
Paste: mortar ratio, Vp/Vm, by volume	0.41	0.27–0.55	0.41	0.31–0.56	0.44	0.33–0.59
Entrapped air content on 37.5-mm fraction, %	1.5	0.1–4.2	1.1	0.2–4.1	1.1	0.5–3.3

[a] Quantities for use in estimating water, sand, mortar, and entrapped air content for trial RCC mixture proportioning studies.

[b] Lower range of values should be used for natural rounded aggregates and mixtures with low cementitious material or aggregate fines content.

Properties of Hardened RCC

The significant material properties of hardened RCC include compressive strength, tensile and shear strength, elastic modulus, tensile strain capacity, Poisson's ratio, volume change (thermal, drying, and autogenous), thermal coefficient of expansion, specific heat, creep, permeability, and durability. The hardened properties of RCC and conventional concrete are quite similar and differences are primarily due to differences in mixture proportions, aggregate grading, and voids content. A wide range of RCC mixtures can be proportioned, just as there is a wide range of mixtures for conventionally placed concrete. It is difficult to quantify typical values in either case. In general, RCC will have lower cement, paste, and water contents and may contain nonplastic fines to fill aggregate voids. Aggregate quality, grading, and physical properties have a major influence on the physical properties of RCC.

Compressive Strength

Compressive strength tests are conducted in the design phase to determine mixture proportion requirements, and to optimize combinations of cementitious materials, water and aggregate. The percentage of pozzolan has a significant influence on the strength development of RCC especially at early ages. The compressive strength of RCC is determined by several factors including the water to cementitious materials ratio, quality and grading of aggregate, degree of compaction, and curing. Degree of compaction has a significant influence on compressive strength. Because of its dry consistency, compaction (consolidation) of RCC requires more effort than conventional concrete. Without full compaction, increased voids will occur within the matrix of the concrete resulting in decreased strength. Delays in compaction may also result in a decrease in compressive strength. Finally, consideration must be given to the fact that most specifications accept 96–98 % of maximum density. As a result, compressive strengths of RCC compacted at less than maximum density will be reduced.

Volume Change

The two significant changes in volume experienced with RCC are due to drying shrinkage (primarily in pavements) and thermal expansion and contraction in mass concrete. Volume change associated with drying shrinkage is normally less than that in comparable conventional concrete mixtures due to the lower water content. This lower shrinkage has resulted in less cracking and revised design considerations for RCC pavements [23]. With respect to thermal considerations, heat rise that causes expansion of a massive concrete structure is due almost entirely to the chemical reactions of the cementitious material. Therefore, the use of lesser amounts of cementitious material in mass RCC construction lowers the potential for thermal cracking. For large dams a common practice is to install contraction joints in the individual lifts of the freshly placed RCC.

Permeability

The permeability of RCC is largely dependent on voids in the compacted mass, together with porosity of the mortar matrix, and therefore is almost totally controlled by mixture proportioning, placement method, and degree of compaction. Hardened RCC permeability is comparable to conventional concrete, although one researcher has indicated the permeability of RCC to be greater than conventional concrete [24]. Typical values for mass RCC range from 0.15 to 15×10^{-9} cm/s [18]. For higher cementitous mixtures such as those for RCC pavements, the permeability tends to be lower.

Durability

Abrasion/Erosion Resistance

Compressive strength and aggregate size, grading, and quality primarily govern abrasion/erosion resistance. Erosion tests in test flumes have indicated the excellent erosion resistance for RCC [*25*]. ASTM Test Method for Abrasion Resistance of Concrete (Underwater Method) (C 1138) has been used to evaluate the performance of RCC for use as streambank protection. Research has indicated that the abrasion resistance of RCC increased with increasing strength and maximum aggregate size. In fact, some studies indicated that aggregates contributed more to abrasion resistance than cement content [26].

Observations of various projects from heavy-duty pavements such as log sort yards to RCC spillways have also indicated excellent resistance to abrasion/erosion. However, ACI 207 on Roller Compacted Mass Concrete recommends that for overflow spillways of RCC dams subjected to frequent use, the RCC should generally be lined with high-quality concrete to prevent abrasion/erosion damage [18].

Freezing and Thawing

Because of its dry consistency, it has not been practical to entrain air in RCC mixtures. Laboratory specimens of nonair-entrained RCC tested according to ASTM Test Method for Resistance of Concrete to Rapid Freezing and Thawing (C 666), Procedure A (in water) and large blocks of mass RCC material exposed to natural weathering of Treat Island, Maine [18], have typically performed very poorly. Nevertheless, there are numerous examples of good performance of nonair-entrained RCC in the field [12,18,27–29]. In Piggott's study [29] a total of 34 RCC pavement projects in the United States and Canada were visually inspected. The projects ranged in age from 3–20 years. The study concluded that except for some surface wear (fines were removed up to a depth 2 mm), the performance of the RCC was very good. The coarse aggregate at the surface remained firmly embedded in the RCC matrix. The study also noted that surface wear typically occurred within the first 2–3 years of service and then stabilized.

Similar to other no-slump, nonair-entrained concrete products such as concrete pavers and precast concrete pipe, RCC derives its durability from its high strength and low permeability. Acceptance criteria for durability tests of concrete pavers according to ASTM Specification for Solid Interlocking Concrete Paving Units (C 936) and ASTM Test Method of Sampling and Testing Brick and Structural Clay Tile (C 67) rely on a combination of minimum compressive strength and moisture absorption. ASTM C 666, which has been used to evaluate the freeze/thaw durability of RCC, is a much harsher test that relies heavily on the presence of air-entrainment for acceptability. Acceptance of RCC according to C 666 criteria usually results in mixtures of very high strength not typical or economically viable for most RCC paving projects.

With regard to air entrainment in RCC, laboratory and field applications have shown an air-entraining admixture can effectively be used to provide good freeze/thaw durability, even when subjected to ASTM C 666 testing [18,30,31]. The difficulty comes in trying to incorporate the tiny air-entrained bubbles uniformly throughout the no-slump RCC mixture. Attempts to entrain air are most effective in RCC mixtures with a Vebe consistency time less than about 35 s using clean, ASTM graded fine aggregate.

Construction

A major benefit of RCC is the cost savings that result from optimizing material selection and the speed of construction. The entire process of batching, mixing, transporting, placing, spreading, compacting, and curing is accomplished as rapidly as possible. There are no forms, reinforcing steel, or finishing. Placement and compaction of the very dry mixture is typically done using equipment and techniques similar to those used for earthwork placement, in the case of mass concrete, and asphalt placement, in the case of RCC pavement. As a result, large quantities of concrete can be placed rapidly with minimum labor and equipment.

Batching and Mixing

The batching and mixing plant requirements for a project to be constructed using RCC are essentially the same as for a project built with conventional concrete [15]. The production, stockpiling, and reclamation of aggregate from the stockpiles are done in the same way and with the same equipment as for conventional concrete. RCC can be produced in any type of plant that will provide uniform mixing of the cementitious materials, aggregates, and water. Often the size of the project and plant availability will dictate which type of mixing method to use.

Horizontal Shaft Mixers—Whether single or dual shaft, portable or permanent, continuous flow (such as a pugmill) or batch, horizontal shaft mixers provide the most intense and fastest mixing action of any mixing plants. Many pugmills are equipped with transfer or gob hoppers to temporarily store the mixed RCC between truck loadings so that the least amount of plant stoppages is required. Due to the speed and quantity of material mixed, horizontal shaft mixers are the preferred mixing method especially for large projects.

Tilt Drum Mixers—The most common central mixing plant for conventional concrete are tilt drum mixers. These mixers are generally available locally and can be used effectively to produce RCC. Because of its dry consistency, RCC batch quantities are typically less than the drum capacity and mixing times are increased.

Transit Mixers—While transit or truck mixers are the most widely available and are capable of producing a quality RCC, difficulties in getting uniform mixing and discharging the dry consistency mixture generally make this type of mixing method unsuitable except for small projects. The recent use of water-reducing and retarding admixtures to improve workability has allowed greater use of transit mixers.

Transporting

The most common methods for transporting RCC from the mixing plant to the placement area are dump trucks, conveyors, or a combination of both. Dump trucks are the most common form of transportation. Depending on weather conditions, protective covers should be provided to minimize moisture loss. In confined areas where dump trucks may be difficult to maneuver, conveyors, front-end loaders, or backhoes may be required to supply RCC to the placement area. Conveyor systems are typically used on large dam projects and where there is a concern that truck hauling may contaminate the previously placed RCC layer.

Special care must be taken during transportation and placement to avoid segregation. Mounding of the RCC during loading and unloading operations should be avoided. Conveyor systems must be designed to minimize segregation at transfer points. RCC mixtures with a 75 mm NMSA have a greater tendency to segregate when they are dumped unto a hard surface, but with care and proper procedures, these mixtures have been hauled, dumped and remixed successfully. Design of wetter consistency mixes tends to reduce segregation.

Placement

Tracked dozers are the fastest, most cost-effective method for spreading RCC. Dozers are the preferred method of placement for dams and other nonpavement applications. Typical lift thickness range from a minimum of 0.15 m (compacted thickness), to over 1 m although no general production in the United States has exceeded 0.6 m. The design of dams where lift thickness greater than 0.3 m have been used has been based on the realization that the spreading of the RCC with heavy dozers not only remixes and redistributes the concrete to overcome segregation but also provides compaction. These procedures have been established and proven by large-scale, well-controlled test section construction and testing, as well as in full-scale production of RCC for dams in Japan and at Elk Creek Dam [15].

Placement of RCC pavements is typically accomplished by the use of heavy-duty asphalt type paving machines. Conventional asphalt pavers have been used; however, they are only equipped with vibrating screeds. As a result, almost all the compaction has to be provided by the vibratory rollers. Heavy-duty asphalt pavers are equipped with tamping and vibrating screeds, which allows for much higher initial compaction from the paver resulting in less compacted effort required from the vibratory rollers. Conventional pavers provide 80–90 % of modified Proctor density, whereas heavy-duty pavers have achieved up to 95 %.

Continuous operation of the paver is critical to achieving a smooth surface without bumps. Trucks delivering RCC to the paver must be scheduled to provide a continuous supply of concrete, but spaced so that they will not be delayed at the paving machine and thus permit the mixed concrete to dry out and loss workability. The use of a transfer device is also recommended whenever practical to eliminate starting and stopping (Fig. 2).

Compaction

One of the most important steps in RCC construction is compaction. RCC is usually compacted with self-propelled vibratory steel drum rollers. Rubber-tire rollers have also used successfully especially as a final pass to remove surface cracks and tears and provide a smooth tight surface. In tight areas such as adjacent to forms, large power tamper jumping jacks are most suitable.

Compaction of RCC should be accomplished as soon as possible after it is spread, especially in hot weather. Typically, compaction should be completed within 15 min of spreading and 45 min from the time of initial mixing. Substantial reduction in strength can be expected if RCC is compacted when it is more than 30–45 min old and the mix temperature is above 21°C. These times can be increased for RCC mixtures with extended set times due to pozzolans, admixtures, or cooler temperatures [18].

Each RCC mixture will have its own characteristic behavior for compaction depending on temperature, humidity, wind, plasticity of the aggregate fines, overall grading, and the

Fig. 2—RCC being placed with heavy-duty paver. Intermediate transfer device used to maintain a constant flow of material to the paver.

NMSA. Typically, four to six passes of a dual-drum 10-ton vibratory roller will achieve the desired density of at least 98 % for RCC lifts in the range of 150–300 mm. Over compaction or excessive rolling should be avoided, since it may reduce the density of the upper portion of the lift.

Curing

Because of the relatively low-water content of RCC, moist curing has been used for most projects. Water cure may be applied by water trucks equipped with fine mist spray nozzles, sprinkling systems or complete submersion. Use of open-ended hoses or coarse sprays that may erode the paste and fine aggregates from the surface should not be used.

Other methods of curing include plastic sheeting, burlap and membrane-forming curing compounds. A white pigment-curing compound conforming to ASTM Specification for Liquid Membrane-Forming Compounds for Curing Concrete (C 309) has become popular for RCC pavement projects. Because of the more open textured surface with RCC compared to conventional concrete, curing compounds are typically applied at higher application rates then for conventional concrete. The application must ensure a uniform void-free membrane exists across the entire RCC pavement surface.

Quality Control

For most RCC projects it is essential to have a quality control program that addresses the activities, procedures, and responsibilities for the specific project. The quality control program is typically the joint responsibility of the contractor, engineer, owner, or owner's representative. The extent of the inspection and testing program will depend on the nature and size of the project. It may be as simple as visual observations or as elaborate as constructing a test section and having an on-site testing lab. A thorough discussion of quality control procedures is presented in references 10, 15, 18, and 32.

Preconstruction inspection and testing typically include sampling and testing the quality of the raw materials; verifying that the type and size of the mixing plant, transportation, placing and compaction equipment meets the project requirements; and inspecting and calibrating the production and testing equipment to ensure proper operation.

Constructing a test section is also part of the preconstruction quality control program. The test section provides for evaluation of the mix design and allows the contractor to develop and demonstrate the proposed techniques for mixing, transporting, placing, compacting, jointing, and curing the RCC during production operations. The test section should be constructed sufficiently early in the contract to allow the contractor time to adjust the size of his batching, mixing, or transporting system; to modify placing, spreading, and compaction techniques; and to change any other operation that is considered essential to the success of the job.

During construction a number of quality control procedures are typically specified. Among them are regular plant calibrations, gradation tests, moisture tests, consistency and density tests, and fabrication and testing of beams and cylinders. Visual inspection for signs of segregation during placement, surface cracking or consistency changes may be indicators of construction deficiencies that need to be corrected. Another important element of inspection is to monitor the time within the various stages of construction. Most specifications require that the RCC mixture be compacted within 45–60 min of mixing and about the same time limit is used to ensure adequate bonding for placement of multiple lifts or adjacent paving lanes.

Consistency and Compactability

The Vebe or similar apparatus is used to measure the consistency or workability of many RCC mass concrete mixtures; however, it is usually not applicable for the drier RCC pavement mixtures. A modified Vebe test, conducted according to Method A of ASTM Test Methods for Consistency and Density of Roller-Compacted Concrete Using a Vibrating Table (C

Fig. 3—Determining Vebe time according to ASTM C 1170. Note ring of mortar along side of container.

1170) is used to determine consistency and compactibility of the freshly mixed RCC. The Vebe test measures the time required for a ring of mortar to appear around the periphery of the surcharge plate (Fig. 3). This test is suitable for RCC mixes with a Vebe time between 10 and 60 s. The test can be used for overall assessment of the RCC workability, but is generally not suitable for control of the uniformity of the mix during production and placement.

The modified Proctor compaction test, ASTM D 1557, is a well-established test for soils that can also be applicable with RCC. The test is used to determine the relationship between the moisture content and dry density of a material for a specific compactive effort, and results in the establishment of a maximum dry density at optimum moisture content. This test method is more applicable for the drier RCC mixtures typically used for pavement applications.

In-Place Density

One of the most important quality control parameters to monitor is compacted density. Density measurements are taken during placing of RCC using a nuclear density gage. The in-place density in the field is compared with the theoretical maximum density or maximum density achieved from a test section or in the laboratory to determine the degree of compaction To ensure the accuracy of the nuclear gages being used, a test block is made during the early stages of the project and kept available for calibration purposes. Specifications generally require the in-place density of the RCC to achieve a minimum of 95–98 % of maximum wet density.

Preparation of Test Cylinders

The primary objective of cylinder preparation is to duplicate the compaction (consolidation) effort and consequently the in-place density of the RCC after compaction in the field. Cylindrical test specimens for determination of compressive strength of RCC cannot be fabricated using the standard procedures used for conventional concrete. As a result, several alternative methods have been developed for RCC and are being used successfully, including (1) Vebe method, ASTM Practice for Making Roller-Compacted Concrete in Cylinder Molds Using a Vibrating Table (C 1176), (2) vibrating hammer method, ASTM Practice for Molding Roller-Compacted Concrete in Cylinder Molds Using a Vibrating Hammer (C 1435), (3) modified Proctor method, ASTM D 1557, (4) pneumatic tamper method, and (5) gyratory compaction method [33]. Each of these methods has advantages and disadvantages. The vibrating hammer (C 1435) and pneumatic tamper work for a wide range of RCC mixture consistencies. The Vebe test is used for wetter mixtures generally with Vebe times of 35 s or less [34]. Both the modified Proctor (ASTM D 1557) and gyratory compaction method are used for the drier RCC mixtures.

Closure

Over the past 30 years, roller-compacted concrete has advanced significantly as a viable construction technique. Primary applications are for dams, spillways, overtopping protection, and pavements. The main advantage of RCC over conventional construction is in the speed of construction and cost savings. Performance of RCC has been very good even under freeze-thaw conditions. Additional research and development is needed to: (1) improve surface texture, skid resistance, and joint construction methods in pavements; (2) establish standardized joint design spacing; (3) establish standardize mixture design methods; (4) develop representative freeze-thaw durability test procedures; (5) determine methods for air-entrainment, (6) improve mixing efficiency using conventional concrete mixing equipment; and (7) expand the use of admixtures including retarders and water reducers to extend working time and enhance performance.

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Polymer-Modified Concrete and Mortar

D. Gerry Walters[1]

Preface

THIS CHAPTER ON POLYMER MODIFIED CONCRETE and Mortar is a revision of the original chapter in *ASTM STP 169C* by Kuhlmann and O'Brien. In January 1987, a Task Group was formed in Subcommittee C09.03.18 (C09.25) due to the need identified for standards development in the area of Polymer Modified Cementitious materials. This Task Group started the process, and then in June of 1989 this activity was organized under a new subcommittee, C09.03.19 (C09.44). Membership in this subcommittee numbers about 50. Committee C09.44 issued Latex and Powder Polymer Modifiers for Hydraulic Cement Concrete and Mortar (C 1438) and Test Methods for Polymer-Modified Mortar and Concrete (C 1439) in 1999. These polymer modifiers mainly contribute to adhesion, water resistance, reduced permeability, and increased durability. Polymer modifiers are used in a variety of applications, such as patching compounds, stucco, ceramic tile thin sets and grouts, and bridge deck overlays. The first ASTM sponsored Symposium on Polymer Modified Concrete and Mortar was held in Louisville in June of 1992 with the papers being published in *STP 1176* [1]. The current edition reviewed and updated the topics of the previous authors introduced new technology that has been developed and included up to date references.

Introduction

Latex is a dispersion of small organic polymer particles in water. When latex is used as an additive to portland cement mixes, the resultant mixture is called polymer-modified mortar or concrete. The polymer particles in a latex are spheres and typically between 0.05–0.50 μm in diameter. (A cubic centimeter of a latex containing 50 % polymer solids of 0.20 μm-diameter particles would contain approximately 10×10^{12} particles.) Surfactants are added to the latex formulation in the manufacturing process (emulsion polymerization) to prevent coagulation of the particles from the mechanical stress of the process, as well as premature chemical reactions with the portland cement. These surfactants also function as water reducers, thus contributing to the improved properties that the latex adds to the mortar and concrete. However, it is the nature of these surfactants to foam when agitated. It is therefore necessary to incorporate an antifoam agent in the latex prior to use in order to control the air content of the portland cement mix.

Latex was originally produced from the sap of the rubber tree and, prior to World War II, this "natural" latex from Southeast Asia was the raw material that established the rubber industry. The war stimulated research into synthetic latex processes resulting in the production of latex by the emulsion process. This original process used a styrene-butadiene (SB) polymer, and the product therefore became known as styrene-butadiene rubber (SBR), a designation that has frequently been used incorrectly today when referring to all latexes, no matter what polymer is dispersed.

One of the first widespread uses for latex-modified portland cement was as mortar for bridge deck overlays. In 1957, 12.7-mm (½-in.) thick mortar modified with SB latex was installed as an experimental coating on a bridge deck in Cheboygan, Michigan, to determine if this would provide a long-lasting wearing surface. Since then, thousands of bridges have been overlaid with latex-modified mixes, initially with 20-mm (¾-in.) mortar, and now with 38-mm (1½-in.) concrete, all of them relying on the adhesion properties of the latex to permanently bond the overlay to the deck concrete [2].

Acrylic polymers have also been used widely for more than 30 years to increase the strength properties and durability of mortar in thin sections [3]. Latex-modified mortars are used in a variety of functional and decorative coating applications such as ceramic tile thinsets and grouts, water-resistant coatings for basement masonry walls, overlayments, and self-leveling flooring applications. These latex-modified mortars are also used in many applications where aesthetics are important. Acrylic latexes display resistance to ultraviolet radiation so they resist yellowing and chalking.

Latex Types

Although there are many types and formulations of latexes manufactured, only those formulated specifically for use in portland cement are suitable in mortar and concrete applications. Descriptions of the formulations and manufacturing processes are available in the literature [4–6]. The latexes most commonly used are copolymers of styrene-butadiene (SB), acrylates (PAE), styrene-acrylate (SA), polyvinyl acetate (PVA), and vinyl acetate-ethylene (VAE). Typical properties of these latexes are given in Table 1. As the names indicate, these latexes are composed of organic polymers that are combinations of various monomers, that is, styrene, acrylate, butadiene, vinyl acetate, etc.

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TABLE 1—Typical Physical Properties of Latexes

Latex Type	Acronym	% Solids	Viscosity, Hz	MFFT[a], °C	pH
Styrene-butadiene	SB	47	20–50	5–15	9–11
Acrylic copolymers	PAE	47	20–100	10–12	9–10
Styrene-acrylic copolymers	SA	48	75–5000	10–18	6–9
Polyvinyl acetate	PVA	55	1000–2500	15–30	4–5
Vinyl acetate-ethylene	VAE	55	500–2500	10–15	5–6

[a] Minimum film forming temperature.

Styrene-butadiene is the most commonly used latex for concrete overlays on bridge decks and is the only latex that has been evaluated for overlays by the Federal Highway Administration [7]. The other latexes, plus SB, are used in mortar applications, such as patching, floor leveling, tile grout, and stucco.

Advancements in processing technology allow producers to now convert some latexes into a dry, powder form, reducing shipping cost and simplifying the mixing process [8,9]. As a powder, the product is used by mixing with water and the other dry ingredients (cement and fine aggregate). These powders have typically been made from polyvinyl acetate and were only applicable to areas where there would be no exposure to moisture. Recently, however, powders from other polymers have been produced that allow the use to be extended to applications where moisture is present. In all cases, manufacturers' recommendations should be followed to assure proper application of these products.

Mechanism of Latex Modification

The mechanism of latex modification of portland cement involves two processes: the hydration of the cement and the coalescence of the latex. The chemistry and reaction processes of cement hydration occur the same as in conventional mortar and concrete. However, while this is taking place, water is being consumed and removed from the latex, concentrating the latex particles and bringing them closer together. With continual water removal, both by cement hydration and evaporation, the latex particles eventually coalesce into a film that is interwoven throughout the hydrated cement particles, coating these particles and the aggregate surfaces with a semi-continuous plastic film. This coalescence results in partially filled void spaces (Fig. 1), as well as adhesion between the aggregate and cement hydrates.

The success of this process is dependent on several factors:

- physical properties of the latex, that is, particle size and quality of dispersion;
- composition of the latex, that is, chemical and physical structure of the polymer; and
- environment, that is, time and temperature of application.

A compact configuration of the latex particles is a necessary stage in the process of film formation, and this occurs only if the particles are dispersed sufficiently to allow them to easily move past each other in the water phase. With a poor dispersion, in which the particles are flocculated, the particles will not become closely packed but will form intermittent voids, and thus a poor quality (spongy) film. Figure 2 is a schematic of the proper film formation process.

Isenburg et al. [10] proposed the following hypothesis for the mechanism of latex reinforcement of portland cement:

- Latex provides equal workability at significantly lower water/cement ratios.
- Latex particles coat the cement grains and aggregate forming a continuous polymer matrix throughout the structure.
- Microcracks form to relieve shrinkage strain due to drying at less than 80–100 % relative humidity.
- Microcrack propagations are restrained and held together by the polymer network.

Laboratory studies have been conducted that confirm most of the features of Vanderhoff's mechanism [10].

Proper film formation of the latex is required to retard water loss. This film ensures an adequate supply of water to allow hydration of the cement as well as the development of adhesion and impermeability properties. This film formation is governed by the latexes' minimum film formation temperature (MFFT), the temperature below which the polymer spheres will not coalesce to form a film. The MFFT of latexes used in cement will vary with latex composition but typically ranges between 4–10°C (40–50°F). Application temperature, therefore, should be maintained above the MFFT so that film formation can occur. This is typically 24 h for thin sections, ≤13 mm (≤ ½ in.) thick, and three to seven days for thicker sections.

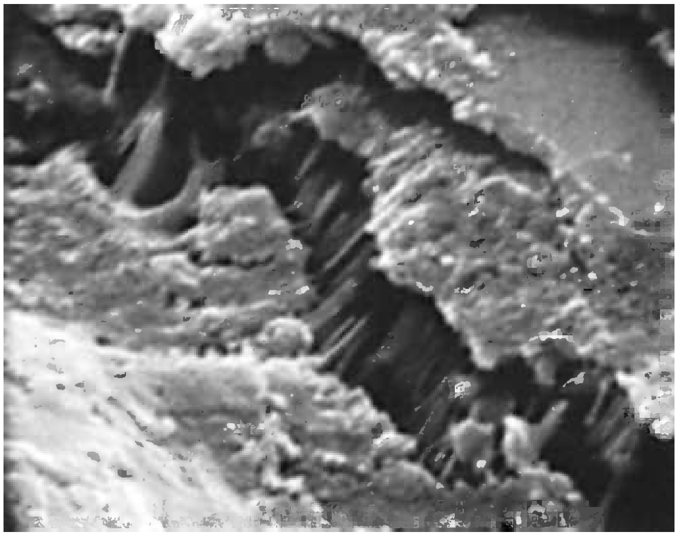

Fig. 1—Electron microphotograph of latex-modified concrete (×11 600).

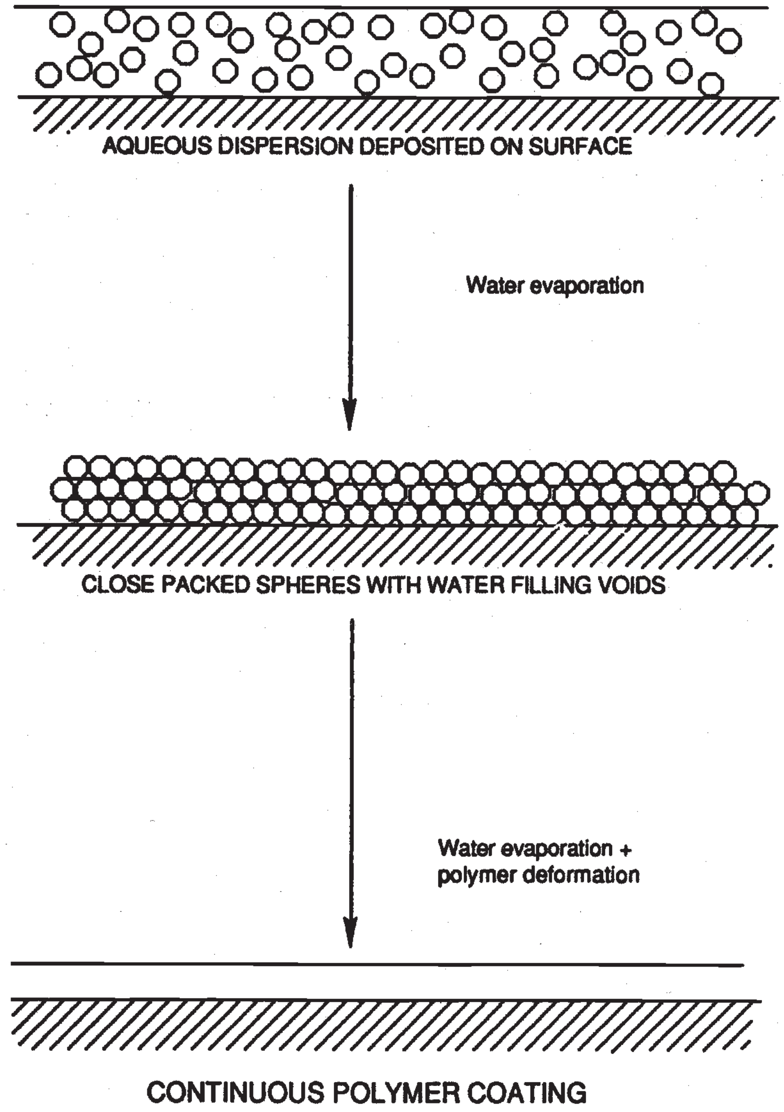

Fig. 2—Process of latex film formation.

Formulating with Latexes

The level of latex modification is usually measured as the weight ratio of polymer solids to cement. The level of latex selected is dependent on the level of performance required. Most performance properties plateau between 15–20 % solids. The 10–20 % level represents the range for optimum, cost effective performance.

Some other formulation considerations that are important are the type and amount of antifoam added, water content, aggregate/cement ratio, and the particular choice of aggregate.

When latex-modified mortar and concrete are mixed, air is entrained within the mix due to the surfactants in the latex. The amount of air generated can be controlled by the inclusion of an antifoam in the latex. The requirements of the end use determine how much air is required and therefore how much antifoam to use. The latex producers can make recommendations for the proper antifoam to be used with their product. And since the cement and aggregate can affect the air content, a trial mix should also be made with all of the recommended ingredients. The kind of air that is entrained in latex-modified mixes has been shown to be small, discrete bubbles [11].

The amount of water used in the mix should be kept to the minimum required to obtain the desired workability. The water level of a latex-modified mix should be lower than a corresponding unmodified mix at equal workability.

The choice of the cement, fine aggregate, and stone used in the appropriate mix is based on the requirements of the application.

Materials

Cement

For most applications, ASTM Specification for Portland Cement (C 150) Type I or II portland cement is suitable with latex-modified mixes, or, where a faster setting time is required, Type III cement can be utilized. Calcium suloaluminate and dicalcium silicate cements can be used to obtain the fastest setting times [12]. ASTM Specification for Expansive Cement (C 845), Type K cement has been used to reduce shrinkage cracking in overlays [13]. Although most brands of portland cement are compatible with the latexes used in mortar and concrete, there are a few that will entrain excessive air. For this reason, it is advisable that the cement be evaluated prior to the work for compatibility with the particular latex being used.

Fine Aggregate

Most fine aggregates that are suitable for mortar or concrete are suitable for latex-modified mixes. Cleanliness is particularly important, since fine contaminants such as dirt or clay can increase the demand for water in the mix and adversely affect workability. Good gradation is important since there is little excess water available in these mixes for workability. This is particularly true for concrete sand, where a large amount of fines can require excessive amounts of water to achieve slump, and thus degrade the properties of the cured concrete. Fine aggregate should conform to ASTM Specification for Concrete Aggregates (C 33). Trial mixes should be done to confirm the suitability of the fine aggregate to produce quality concrete.

Coarse Aggregate

The choice of coarse aggregate for polymer-modified concrete should be based on concrete practices and comply with ASTM Specification for Concrete Aggregates (C 33). The coarse aggregate should be clean, non-reactive, and sized for the thickness of the application, that is, maximum size should be no greater than one-third the thickness.

Water

Water should be clean, potable, and meet the minimum chloride requirements of the application.

Admixtures

No admixtures should be used in conjunction with a latex mix, without prior knowledge and approval of the latex manufacturer. In self-leveling, polymer-modified concrete mixtures, a high-range water-reducing admixture can be used.

Polymer Modifiers

There are two types of polymer modifiers covered in ASTM Specification for Latex and Powder Modifiers for Hydraulic Cement Concrete (C 1438). One type is for use in areas not exposed to moisture and the other for general use. Polymer modifier is tested in accordance with ASTM Test Methods for Polymer-Modified Mortar and Concrete (C 1439).

Mixture Proportions

Mortar

Mortar incorporating latex modification consists of portland cement, fine aggregate, latex, and water. The selection of the aggregate size will depend on the thickness of the application, just as in non-modified mortar, that is, maximum size should be no greater than one-half the thickness of the application.

TABLE 2—Representative Mixture Proportions for Latex-Modified Mortar Suitable for Concrete Patching

	Parts, by weight
Portland cement	1.00
Fine aggregate	3.50
Latex (48 % solids)	0.31
Water, approximately	0.24
Formulation constants: latex solids/cement = 0.15; water/cement = 0.40	

TABLE 3—Sprayable Formulation for an Acrylic Latex Cementitious Metal Primer

Materials	Parts, by weight
Silica flour 120	200
Portland cement Type I	100
Acrylic latex (48 % solids)	62.5
Defoamer	0.3
Water	90
Formulation Constants	
Solids content, %	72.7
Weight, kg/L (lb/gal)	1.8 (15.2)
Polymer solids/cement	0.30
Filler/cement	2

It is important to know the solids content of each particular latex product before designing the mix. From this, the amount of latex required can be determined, based on a specified latex solids-cement ratio. The next step would be to select an appropriate water-cement ratio. (Because most latex formulations function as water reducers, a water-cement ratio of less than 0.40 is common.) From this, the amount of additional water required can be calculated, keeping in mind that the water in the latex is included in the total water calculation.

Portland cement is used with fine aggregate at a range of ratios, from 1:1 to 1:4, depending on the end use. The specific quantity used will depend on the strength required, with higher cement contents generally providing an increase in strength, but also a tendency for more shrinkage. Of course, an increase in cement will also increase the total amount of latex in the mixture if the latex-cement ratio is constant.

A range of mixture proportions are possible for mortar, depending not only on the application being considered, but also on the latex being used. Table 2 gives a representative mixture proportion for mortar to be used to repair concrete. Table 3 gives a formulation for a metal coating.

Concrete

Mixture proportion procedures for latex-modified concrete are usually based on a latex solids/cement ratio of 0.15, and a maximum water-cement ratio of 0.40. Most latex-modified concrete mixture proportions have followed conventional concrete criteria for quality and used a cement factor of 300 kg/m^3 (658 lbs/yd^3), although deviations from this can be accommodated. Table 4 gives a typical concrete mixture that is used for overlays.

TABLE 4—Representative Mixture Proportions for Latex-Modified Concrete for an Overlay

Material	Quantity	
Portland cement	300 kg	(658 lb)
Fine aggregate	785 kg	(1725 lb)
Coarse aggregate	520 kg	(1150 lb)
Latex (48 % solids)	93 L	(24.5 gal)
Water	72 L, max	(19 gal)

NOTE—This mix provides a latex solids/cement of 0.15 and a water/cement of 0.37.

Properties

The addition of latexes to portland cement mixes generally improves the final product in two ways: (1) by reducing the amount of water required in the freshly mixed concrete and (2) by providing dispersed polymer in the matrix of the hardened state. Combined, these result in the following improved properties: bond strength, flexural strength, freeze/thaw durability, and a reduction in permeability. The degree of improvement of any particular property varies somewhat with the particular latex used. Complete and detailed information on these latexes can be found in Ref 14.

Freshly Mixed Properties

The water-reducing property of the latexes is evidenced by good workability at a low water-cement ratio. Mortar with a flow of 120 cm and concrete with a slump of 15–20 cm (6–8 in.) is typical for mixes made with a water-cement ratio of 0.37.

The air content of the mixes can vary widely, depending on the requirements of the end use. For some thin-layer applications, air contents above 10 % are desired to improve trowelability. For mortar and concrete repair work, air contents in the range of 4–6 % are desired. All of these can be achieved by the proper selection of the amount of latex and antifoam used.

The setting time of latex-modified mixes is controlled primarily by the reaction of portland cement with water and is similar to that of unmodified mixtures (see Fig. 3) [15,16]. There are some differences, however, due to the influence of each particular polymer type. The available working time for finishing, however, can be considerably shorter than unmodified mixes due to evaporation of water from the surface of the mix. This evaporation causes the formation of a film, or crust, on the concrete or mortar surface that can tear if overfinished. Producers usually recommend that trial mixes be made to evaluate the characteristics of the components of each particular mix to ensure that the mix fully meets the customer's requirements.

Hardened Properties

Air Void System

Air voids in latex-modified concrete do not always conform to the size criteria established for conventional concrete [11,17]. The same void spacing factor required for resistance to freezing and thawing in conventional concrete is not necessary in latex-modified concrete containing 10–20 % polymer solids/cement, since the latex polymer tends to seal the matrix and limit the amount of water permeating the concrete. The voids are, however, all spherical, and tend to decrease in diameter as the total air content increases.

Bond

The adhesion property of latex-modified mixes has been demonstrated both in the laboratory and in the field. A study of SB latex-modified mortar indicated that its bond to the concrete surface is chemical in nature, thus creating a somewhat homogeneous combination of the two materials [10]. Bond tests of this mortar have demonstrated that ultimate failure should be in the parent concrete, that is, the bond strength of a modified mortar to a properly cleaned concrete surface usually exceeds the tensile strength of the substrate concrete [18]. Bond studies of other latexes have also been reported. In a five-year out-door exposure study, where samples experienced over 70 freeze/thaw cycles and 130 cm (50 in.) of rain per year, shear bond values of acrylic modified

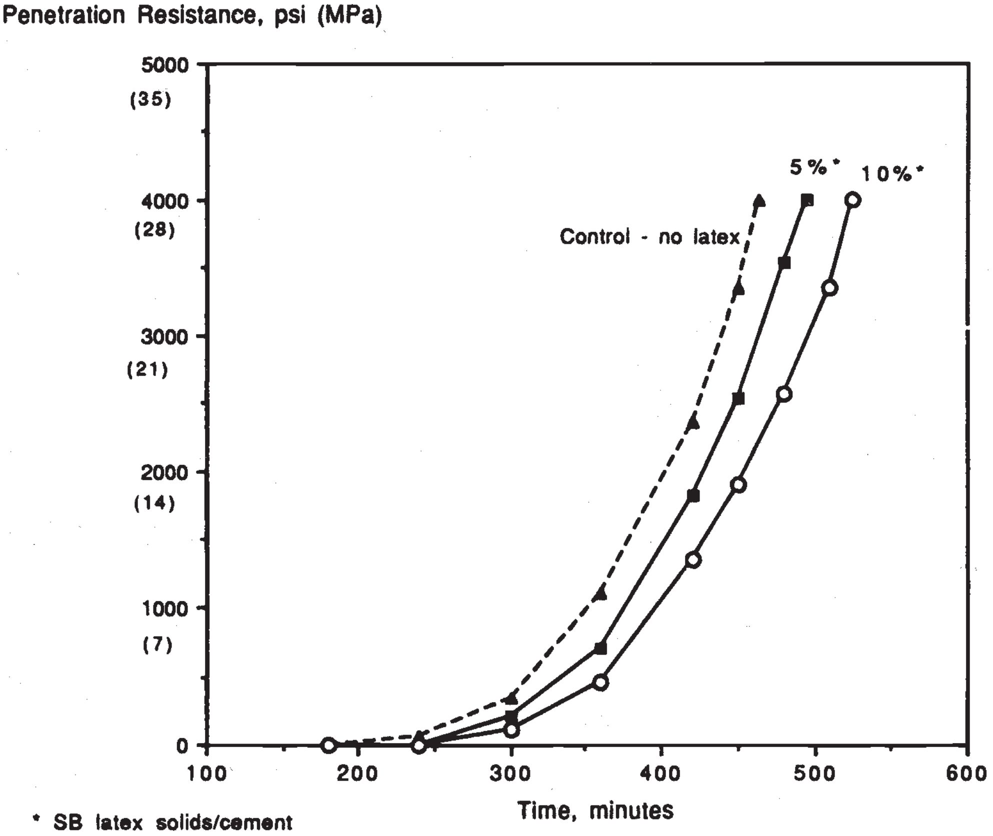

Fig. 3—Setting time of latex-modified concrete.

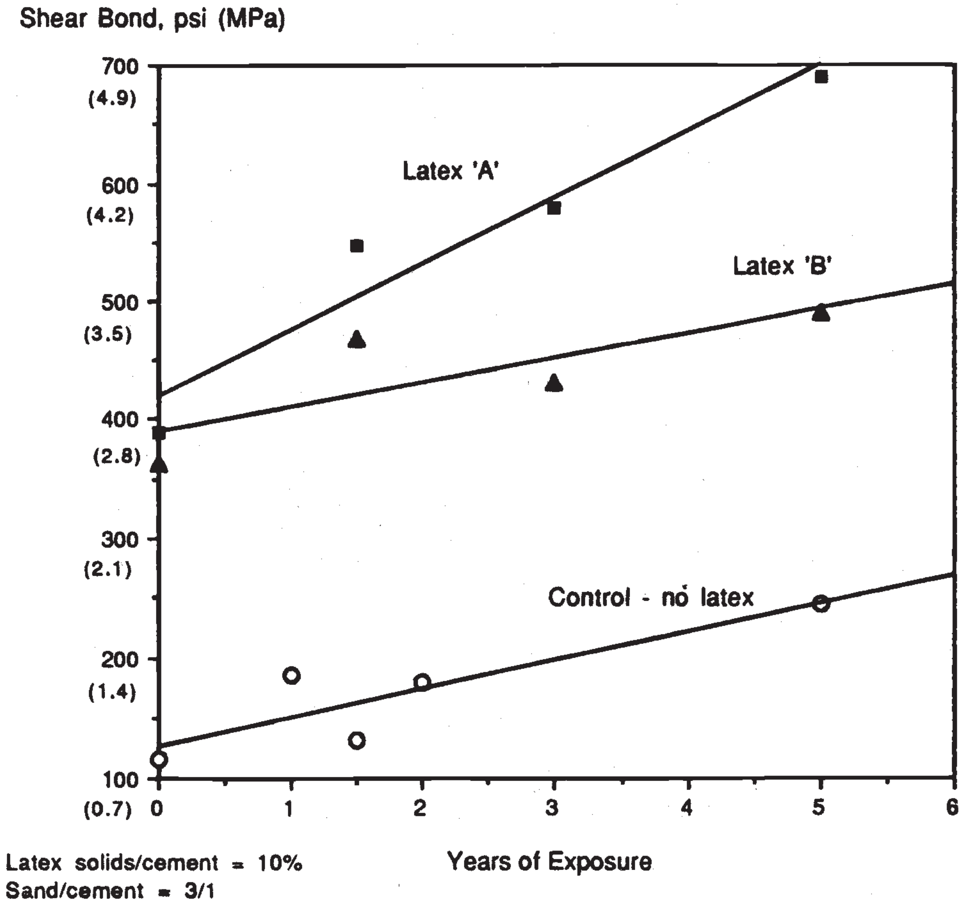

Fig. 4—Effect of weathering on adhesion of acrylic-modified mortar.

mortar more than doubled those of the unmodified control (Fig. 4) [3]. Additionally, when the bonds were tested, the latex-modified samples failed cohesively, whereas the controls failed at the bond line. In adhesion tests of samples cured normally in air, Nihon University reported that mortars modified with SB, PAE, and EVA latexes exceeded the bond strength of the unmodified control by factors ranging from 2 to 3 [15]. In this study, samples were also submerged in water before testing. All showed a decrease in strength, although the modified mortars always exceeded the control. Another laboratory study compared the bond strengths of five different latexes [19]. The results, shown in Fig. 5, indicate that SB and VAE had the highest values and that all but the PVA failed in the parent (substrate) material. The PVA failed at the bond line at a strength of 1.28 MPa (185 psi). Adhesion performance in the field was reported in a study of 20-year old bridge deck overlays, two of which had SB latex mortar overlays [20]. Cores were tested for overlay adhesion and resulted in failure of the base concrete. The bond properties of concrete modified with SB latex have been reported frequently since this material has been used extensively for thin-bonded overlays on concrete bridge and parking garage decks [21–24]. Values of 2.07–2.76 MPa (300–400 psi) at 28 days' cure for direct tensile bond strength are typical, with failure occurring in the substrate if surface preparation is proper.

Freeze-Thaw and Scaling Resistance

The durability of concrete modified with SB latex has been demonstrated by superior resistance to freezing and thawing, and wearability. The low permeability to water results in improved resistance to freezing and thawing as measured by ASTM Test Method for Resistance of Concrete to Rapid Freezing and Thawing (C 666) and ASTM Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals (C 672) [3,7]. The concrete sometimes fails C 666 because it does not have the proper air void system [13]. This is not considered a problem, as 30 years of field experience has not shown any freeze-thaw deterioration. Wear resistance, as evidenced by the thousands of bridge decks in service, was documented by the Oregon Department of Transportation [25]. Their study of the wear characteristics of latex-modified concrete bridge deck overlays indicated a life expectancy of 23–45 years for a lane having an average daily traffic of over 20 000 vehicles.

Permeability

The pore-sealing effect of latex in a concrete mix results in a major reduction of its permeability to both gases and liquids. For instance, carbonation studies have shown that inclusion of latexes in concrete significantly reduces the carbonation depth of the concrete (Fig. 6) [26]. Chloride permeability is another property that has been measured frequently on latex-modified concrete, primarily on SB-modified concrete, since this is of major interest for bridge and parking deck applications [3,16,27]. Figure 7 gives the results of a study of SB-modified compared to conventional concretes, using the ASTM Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration (C 1202) [28]. Figure 8 shows

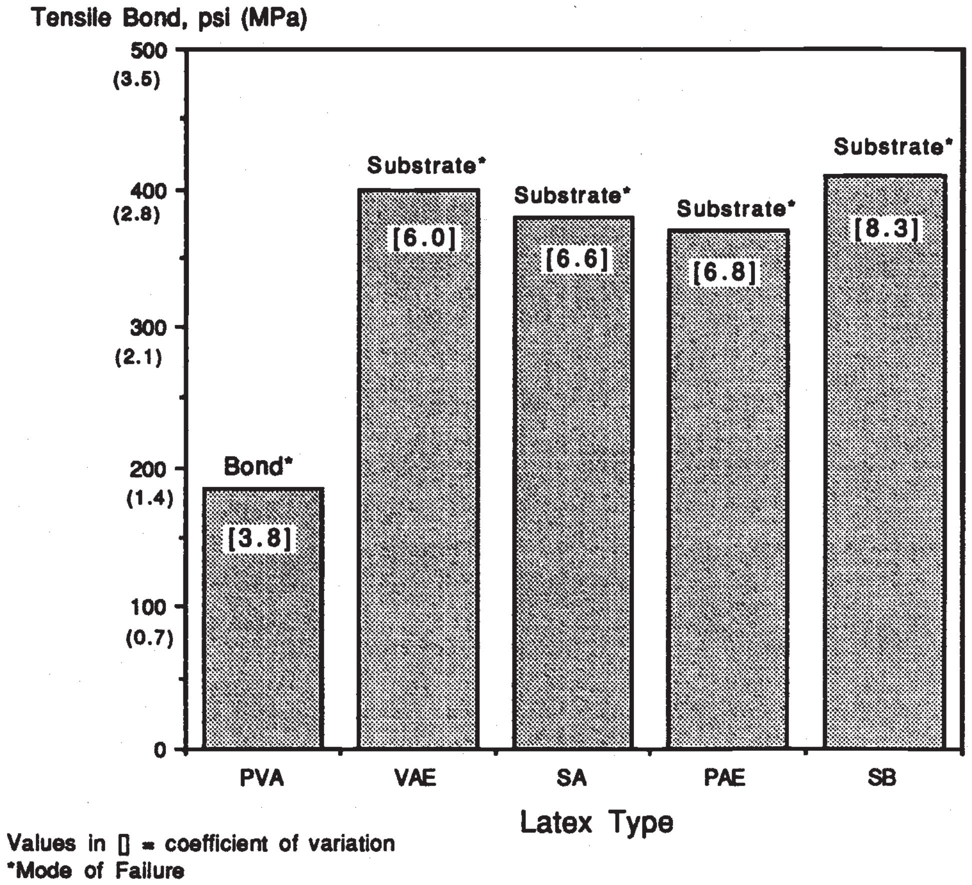

Fig. 5—Bond strength of latex-modified mortars.

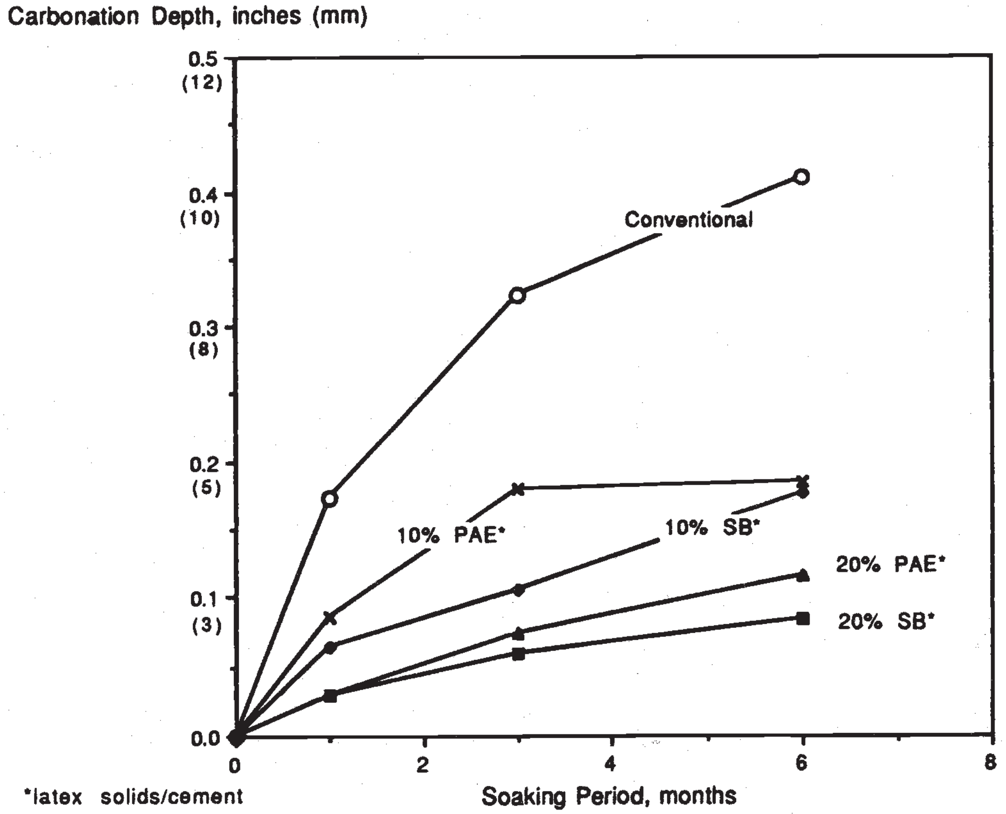

Fig. 6—Carbonation resistance of latex-modified concretes.

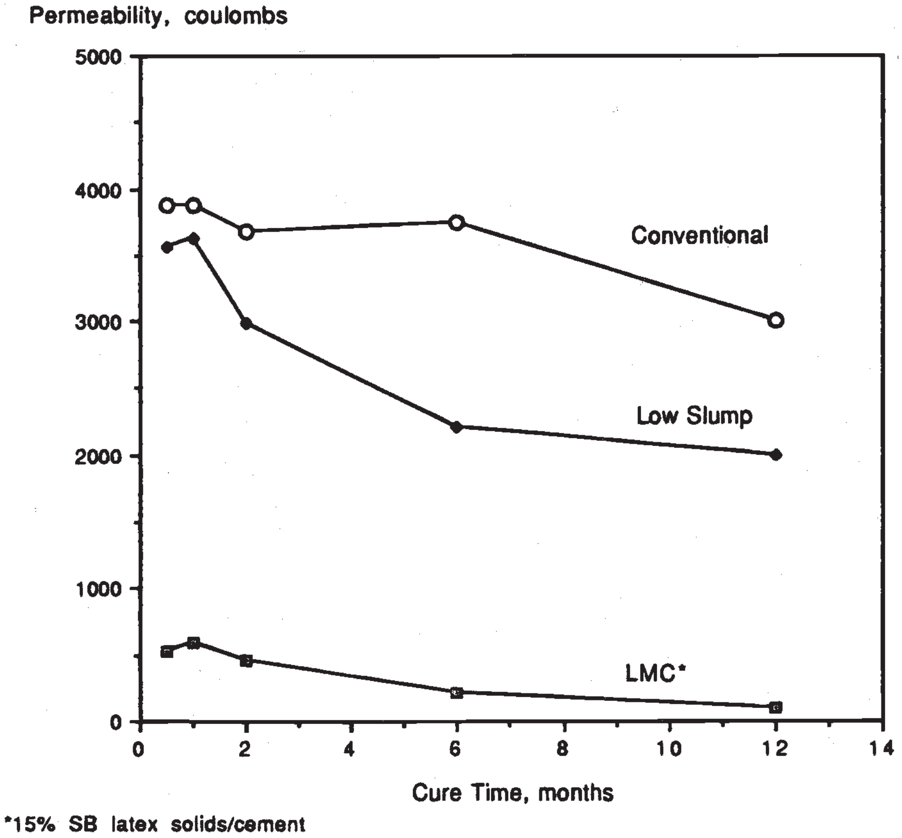

Fig. 7—Permeability of concretes versus cure time.

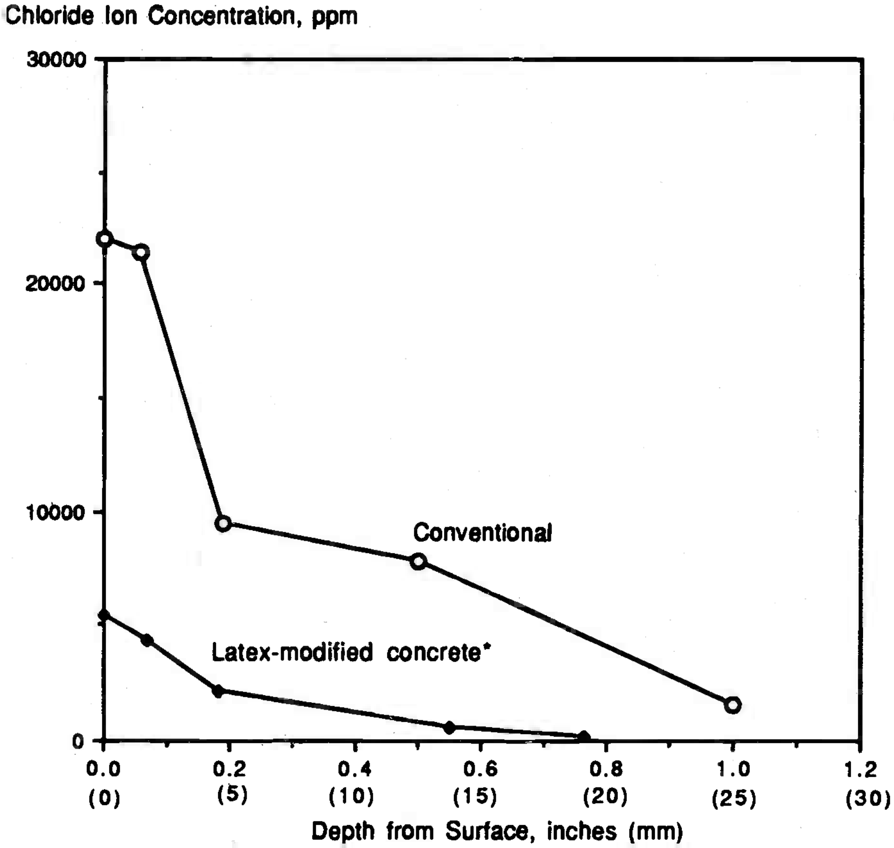

Fig. 8—Chloride ion resistance of latex-modified concrete.

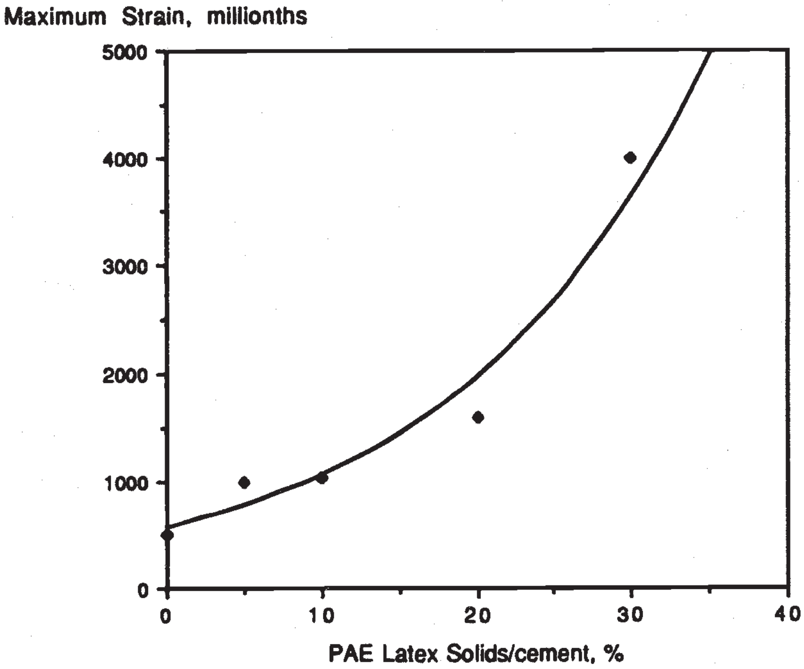

Fig. 9—Effect of latex content on strain capacity of mortar.

chloride penetration from a ponding study of PAE-modified concrete [3]. In all cases, the resistance to permeability performance of latex-modified concrete is evident.

Strain Capacity

The ability to absorb movement (strain capacity) is an important feature of latex-modified mixes. Figure 9 clearly indicates that the strain capacity of mortar that is modified with an acrylate copolymer increases as latex content increases [13].

Applications

Portland cement mixes containing latex are typically used where the following properties are desired: adhesion, durability, low permeability, weatherability, and flexibility.

As with conventional portland cement systems, the choice of whether to use mortar or concrete is based on the thickness of the application–for thin layers, 20 mm (¾ in.) or less, mortar is used, and for cross sections greater than 20 mm (¾ in.) concrete is appropriate.

Mortar

Because of its adhesion characteristics, latex-modified mortar has no minimum thickness requirement. Mortar coatings, as thin as featheredge, are possible as long as the appropriate fine aggregate is selected and an adequate amount of latex is used. A wide variety of applications have been developed since the material was introduced in the 1950s. These include:

- concrete repair for parking garage floors, bridge decks, swimming pools, and industrial floors; and
- functional coatings for water-resistant basement coatings, decorative spray coatings for exterior walls, skid-resistant coatings for pavement and ship decks, adhesives and base coats for exterior insulation finish systems, and maintenance coatings for metal.

Skid-Resistant Coatings

A unique concrete pavement application, taking advantage of the adhesion properties of latex, utilized a slurry of SB latex, portland cement, and a fine blast furnace slag to restore skid resistance to pavement that had been worn smooth by traffic [29,30]. The slurry was broomed and screeded onto the blasted concrete surface and cured for one day under burlap and polyethylene film.

Thin coatings of acrylic and SB-modified cement mortars are applied to decks of ships in order to provide a skid-resistant and protective surface to the steel substrate. The adhesion of the latex-modified mortars make them especially suitable for this use.

Decorative Coatings

Cement-based coatings have been formulated with an acrylic latex for application over a variety of substrates in order to improve appearance as well as performance. Wood, concrete, and steel are some surfaces that are typically coated. Because of the adhesion of the latex, coatings can be relatively thin, approximately 3 mm (⅛ in.), and still provide weather resistance and long-term performance.

Adhesives and Base Coats for Exterior Insulation Finish Systems

Exterior insulation finish systems (EIFS) are applications for usually acrylic latex-modified cement mortar. In this application, an insulating material such as expanded polystyrene foam is attached to the outside surfaces of walls of buildings. The insulating foam is typically attached to the substrate with an acrylate latex-modified cement mortar. The insulating foam is then covered with a polymer-modified cementitious layer reinforced with a fiberglass scrim. This reinforcement provides the foam with structural integrity as well as protection from moisture and sunlight.

Water-Resistant Basement Coatings

Portland cement coatings have been formulated with acrylic latex to be used on concrete block basement walls. Applied to the outside of the wall, these coatings provide resistance to water penetration. A laboratory procedure, based on Federal Specification TT-P-1411, has been used to test the water-resistant properties of these coatings [3].

Latex-Modified Cement Maintenance Paint

Latexes have been used to formulate two-component cementitious primers that provide low-cost protection of metal substrates. The water-based cementitious mortars are low odor and non-flammable and afford a number of benefits:

- Corrosion and water resistance.
- Flash and early rust resistance.
- Protection of rusty as well as clean metal surfaces.
- Capability of curing in damp enclosed areas.
- Adhesion to ferrous metal surfaces.
- Flexibility.
- Weatherability.
- Solvent-free composition.
- Ambient cure.

Cementitious metal primers modified with latex can be applied by brush, roll, and airless spray. Thick films, 0.50–0.75 mm (20–30 mils), can be easily applied without sag as these primers are high in solids. Two-component systems are commonly used in the maintenance and marine industries. A polymer-modified cementitious coating can be prepared by blending the dry ingredient (filler/cement) in one container and mixing it with the liquid (latex/defoamer/water) in another. A power mixer typically can be used for mixing the two components together.

Latex-modified mortar in thin coatings adheres to a metal surface, demonstrating good adhesion and flexibility. Improvement in properties is observed as the polymer level is increased. For this application, 30 % latex solids has demonstrated corrosion resistance as evaluated in a salt spray cabinet (Method of Salt Spray (Fog) Testing (ASTM 117–90)) as well as by several years' exposure at an Atlantic Sea coast test facility.

Ceramic Tile Thinsets and Grouts

Latex-modifiers are used to formulate thin-set adhesives and grouts that meet the ANSI Specification for Latex Portland Cement Mortar A118.4 and A118.6 for thinsets and grouts, respectively. Latexes and powder polymers provide high adhesion, improved water resistance, flexibility, and impact strength.

Shotcrete

Latex-modified shotcrete has been used for vertical and overhead concrete repair, but only to a limited degree due to the lack of trained contractors.

Concrete

The majority of concrete applications incorporating a latex modifier have been bridge and parking garage deck overlays. The SB latex has been used for this application for over 20 years [20]. Thousands of bridges and hundreds of parking garages in the United States have been protected with this type of overlay during this time.

Other applications for this modified concrete are structural restoration and concrete pavement repair. The restoration of Soldier Field [21], a 58-year-old concrete stadium in downtown Chicago, occurred in 1981. The existing concrete tread-and-risers were used as the form for pouring new reinforced concrete containing SB latex. This new modified concrete was 75 mm (3 in.) thick on the horizontal tread, and 150 mm (6 in.) against the vertical surface. One of the unique features of this application was that the 3000 m^3 (4000 yd^3) of concrete were placed by a pump.

On concrete pavement, polymer-modified concrete has been used to repair deterioration at the centerline of 56 km (35 miles) of interstate highways in Pennsylvania [31]. A 50-cm (20-in.) wide strip, 25 mm (1 in.) deep, was removed by a scarifier and replaced with concrete containing SB latex. The construction techniques were similar to those of an overlay; that is, the old surface was blast cleaned, the paste was scrubbed in to assure bond, and it was cured for one day under wet burlap and polyethylene. Because of the adhesion properties of the modified concrete, this thin section is well bonded and serves as an expedient repair.

Equipment Considerations

Most equipment and tools used for conventional mortar and concrete have been used successfully with latex-modified systems. This includes pumps, buggies, trowels, and screeds, to name a few. Whatever equipment is being used, it is important to minimize the time that the surface of the freshly mixed material is exposed to the atmosphere so that crusting does not occur. This would be of more concern, for instance, if a buggy were used rather than a pump to transport latex-modified concrete. Whereas no drying would occur while the modified material is in the pump hose, the buggy might have to be covered to prevent drying if the transport time were long.

Because latex-modified mortars and concretes bond so well, a major concern with any equipment that is used with these systems is cleaning. It is important, therefore, that equipment be cleaned thoroughly with water immediately after use and before drying occurs.

Limitations

For exterior applications, or where exposure to moisture is possible, PVA latex should not be used. For outdoor applications where color is important, such as stucco that uses white portland cement, the SB is not the preferred choice due to a discoloration from the effect of sunlight on the butadiene. Those latexes/polymers not containing butadiene, that is, SA, PAE, and VAE, are recommended.

As with conventional portland cement, these materials should be used with caution during extreme weather conditions, that is, between 4–30°C (40–85°F). During conditions of rapid drying, these latex-modified systems are more sensitive than conventional mortars and concrete with low water contents because of the film-forming characteristics of the latex. Rapid drying causes a skin (or crust) to form on the surface if it is allowed to dry, making the finishing operation difficult. Care should be exercised when ambient conditions, that is, relative humidity, wind, and temperature, create an environment for rapid evaporation of water.

Since latex-modified mortars and concretes require air drying to achieve their optimum properties, these systems are not suitable for underwater applications unless sufficient cure time is allowed for latex coalescence. Typically, 28 days is recommended. This cure time can be monitored by samples cured under identical conditions at the field installation and tested periodically until the design properties are achieved.

Specifications, Guides, and Reports

An ACI specification for using SB latex-modified concrete overlays has been published [32]. The American Concrete Institute has also published guides and reports [33–35]. As stated earlier, ASTM Specification for Latex and Powder Polymer Modifiers for Hydraulic Cement Concrete and Mortar (C 1438) was published in 1999.

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Shotcrete*

John H. Pye[1]

Preface

THIS CHAPTER, AUTHORED BY I. LEON GLASSGOLD, was originally published in *STP 169C* [1]. In the preparation of this chapter, the contents of the 4th edition were drawn upon. The current edition will review and update topics as addressed by the previous author, introduce new technology and current industry practice, and include up-to-date references and a bibliography.

Although shotcrete has been a viable and important element in concrete construction for more than 90 years, it has become universally accepted only in the last 30 years or so. Its versatility and its applicability to a large variety of singular uses provide economical alternatives and complements to conventional concrete construction.

Currently, the design and selection of shotcrete as a material, structural system, or component are more likely to be based on empirical and heuristic methods than on codified design or rigorous analytical or numerical design approaches. Confirmation of the performance of shotcrete, albeit generally subjective, has in turn led to greater interest and acceptance of shotcrete and to a general recognition of its capabilities, limitations, and unique characteristics as a construction process. Nevertheless, it has become apparent in recent years that there is a need for continuous improvement and assurance of the quality of shotcrete installations, facilitated by the development and application of appropriate standards.

In the United States, responsibility for creating and maintaining standards for concrete and its associated technologies, including shotcrete, has been shared by the American Concrete Institute (ACI) and ASTM. In a memorandum of understanding in 1936, ASTM agreed to address material specifications and test methods, and ACI agreed to cover concrete design and construction practice. ACI has been publishing articles and reports on shotcrete since 1911 and standards on shotcrete practice since 1951. In addition, the ACI documents have been referencing ASTM standards for material specifications and test methods. As early as 1920, Professor M. O. Fuller of Lehigh University performed experimental tests on gunite (shotcrete) slabs using ASTM specifications C 9–17 for cement [2], which were replaced by ASTM Specification for Portland Cement (C 150). Moreover, until 1988, the primary ASTM standards used for shotcrete construction were based on practices for concrete.

Since 1988, subcommittees of ASTM Committee C09 have published ten standards covering shotcrete. Both the number of standards and the rate at which they were generated needed to adequately cover current technology and the industry's need for new standards. In 1990, ASTM Committee C09 decided to concentrate its efforts through a subcommittee on shotcrete, C09.46. The scope and mission of subcommittee C09.46 was stated in January 2000: "To develop and maintain standards for Shotcrete." Subcommittee C09.46 is adapting existing shotcrete standards or developing new ASTM standards, as described later in this chapter in the section on ASTM and shotcrete. A recent review of U.S. shotcrete standards includes a summary of the various organizations currently developing standards and recommendations for the shotcrete industry [3].

Introduction

Shotcrete Defined

The term "shotcrete" was coined by the American Railway Engineering Association (AREA) around 1930 to provide a single, generic term that encompassed the many proprietary dry-mix processes, designated as Gunite, Blastcrete, Blocrete, Jetcrete, Guncrete, Spraycrete, Nucrete, etc. [4]. ACI initially used the term "pneumatically placed mortar" to describe the dry-mix process in its Recommended Practice for the Application of Mortar by Pneumatic Pressure (ACI 805–51). However, in the interest of clarification and consistency, the term "shotcrete" was used in the text of this first ACI recommended practice [5]. This standardized the use of the term in the United States, Canada, and all countries basing their shotcrete technology on ACI documents. In contrast, in Europe and elsewhere, the term "sprayed concrete" is more commonly used.

The shotcrete process is a unique construction method using compressed air to pneumatically project mortar or concrete against a surface at high velocity. Shotcrete currently is defined in ASTM Terminology Relating to Concrete and Concrete Aggregates (C 125) as "a mortar or concrete that is projected pneumatically at high velocity onto a surface." It is similarly defined in *Guide to Shotcrete* (ACI 506–95) [6]. The

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* The views expressed by the author do not imply a position on the part of the U.S. Nuclear Waste Technical Review Board (NWTRB) concerning the potential application of shotcrete at the U.S. Department of Energy (DOE) Yucca Mountain Project.

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term encompasses two distinct processes: dry-mixture and wet-mixture shotcrete. ASTM C 125 defines these different processes as follows: "dry-mixture shotcrete, *n*-shotcrete in which most of the mixing water is added at the nozzle"; and "wet-shotcrete, *n*-shotcrete in which most of the ingredients, including water, are mixed prior to introduction into the delivery hose." A more comprehensive description of each process, the plastic and hardened physical properties of the in-place shotcrete, the range and variety of applications, and associated attributes can be found in *Guide to Shotcrete* [6] and other ACI 506 committee documents.

Early References

In 1911, ACI, then known as the National Association of Cement Users (NACU), published what may have been the earliest article on Gunite, a proprietary dry-mix process [7]. An article published in *Cement World* magazine in 1916 described what would later be called the wet-mix process [8]. However, the wet-mix process did not become a fully accepted technology until the early 1950s, when the True Gun, a dual-tank pneumatic device, was introduced. Shortly thereafter, various types of concrete pumps were adapted to the wet-mix shotcrete process and resulted in the acceptance of the wet-mix process as a viable and economical method for many shotcrete applications.

Shotcrete Properties

Shotcrete differs from concrete in manufacture, method of placement, and physical properties, although the final hardened product has in many respects performance characteristics similar to those of concrete. Methods and techniques of shotcrete manufacture and placement are important factors in the shotcrete process and can influence its performance characteristics and properties. Therefore, recognizing and taking into account the effects of shotcrete placement when sampling and testing shotcrete are important. Historically, the emphasis on shotcrete performance was placed primarily on such physical properties and characteristics as compressive, tensile, and flexural strength in addition to bond, permeability, shrinkage, density, and uniformity. Early tests for establishing the compressive strength, bond, and density were carried out by Professor M. O. Fuller. A subsequent series of tests were performed by the University of California and attributed the properties to pneumatic placement. Additional data followed from studies at the University of Toronto, the U.S. Bureau of Standards, the Department of the Navy, and many others before 1939 [9]. The results typically showed that shotcrete compared favorably with concrete.

Early concerns about the durability of shotcrete, reported in the literature [10], were subsequently dispelled, on the basis of evaluation of test results [10–18]. The addition of silica fume in both wet- and dry-mix shotcrete has improved significantly both the strength and the durability of shotcrete [10]. Current evidence consistently demonstrates that good-quality shotcrete, composed of sound materials and properly applied, can produce a very durable material.

ACI and Shotcrete

As previously indicated, ACI has been reporting on shotcrete almost from its inception in 1911, under the auspices of concrete technology. In 1942, ACI formed Committee 805 on Pneumatically Placed Mortar which was retired after completion of the practice (ACI 805–51). In 1957, ACI Committee 506 (ACI 506) was reactivated to revise and update the aforementioned recommended practice for shotcreting. Since that time, ACI 506 has sponsored many ACI symposia and seminars in addition to producing a number of reports and standards including one well-known and popular document, a special publication, *Shotcreting, SP-14* [19], which contains many papers describing the state of the art of shotcrete in the early 1960s. ACI 506 also produced the 1966 Standard Recommended Practice for Shotcreting (ACI 506–66) [20]; the 1977 Specification for Materials, Proportioning and Application of Shotcrete (ACI 506.2–77) [21]; the 1982 Guide to the Certification of Shotcrete Nozzlemen (506.3R-82) [22]; and the 1984 State of the Art Report on Fiber Reinforced Shotcrete (ACI 506.2–77) [23]. *Guide to Shotcrete* (ACI 506R-85) [6], published in 1985, replaced the out-of-date Standard Recommended Practice for Shotcreting (ACI 506–66) [19]. Currently nozzleman certification is under the auspices of ACI committee C 660 as outlined in the ACI publication *CP-60*.

ASTM and Shotcrete

ASTM Committee C09 has produced ten shotcrete standards since 1988 [24]. Chemical Admixtures Subcommittee C 09.23 published two shotcrete standards in 1989, ASTM Practice for Preparing and Testing Specimens from Shotcrete Test Panels (C 1140) and ASTM Specification for Admixtures for Shotcrete (C 1141). In 1984, Subcommittee C09.42 published ASTM Test Method for Flexural Toughness and First-Crack Strength of Fiber-Reinforced Concrete (Using Beam with Third–Point Loading) (C1018), which addressed the toughness of thin concrete overlays or shotcrete linings. In July of 1989, the Fiber-Reinforced Concrete Subcommittee C09.42 published ASTM Specification of Fiber-Reinforced Concrete and Shotcrete (C 1116). In June 1989, ASTM Test Method for Time of Setting of Shotcrete Mixtures by Penetration Resistance (C 1117) was published and was subsequently withdrawn in December of 2002. Still earlier, in November 1988, ASTM published ASTM Test Method for Time of Setting of Portland-Cement Pastes Containing Accelerating Admixtures for Shotcrete by Use of Gillmore Needles (C 1102). This standard was discontinued in January of 2002 and was replaced by Test Method for The Laboratory Determination of the Time of Setting of Hydraulic-Cement Mortars Containing Additives for Shotcrete by Use of Gillmore Needles (C 1398). In February 1998, Subcommittee C0.46 published ASTM Practice for Sampling for Shotcrete (C 1385). In 1999, Subcommittee C09.46 published ASTM Specification for Materials for Shotcrete (C 1436) and in 2000 ASTM Specification for Packaged, Pre-blended, Dry, Combined Materials for Use in Wet or Dry Shotcrete Application (C 1480). In 2003, Fiber-Reinforced Concrete Subcommittee C09.42 published ASTM Test Method for Flexural Toughness of Fiber Reinforced Concrete (Using Centrally Loaded Round Panel) (C 1550). Most recently, Subcommittee C0.46 published ASTM Test Method for Obtaining and Testing Drilled Cores of Shotcrete (C 1604/C 1604M).

When shotcrete is used to provide structural support it is often combined with other reinforcing elements. In addition to concrete standards, ACI 506 references ASTM standards for reinforcing bars, welded wire fabric, and prestressed wire [6]. This group of standards comes under the jurisdiction of ASTM Committee A1 on Ferrous Metals and cannot be revised or rewritten for shotcrete use by a subcommittee of Committee C09. These standards should be reviewed carefully for suitability before being specified by a project.

Refractory Shotcrete

Refractory shotcrete differs from conventional shotcrete in several ways. The primary difference is that refractory shotcrete may be exposed to service temperatures of up to 3400°F (1870°C). The physical properties, such as compressive strength, modulus of rupture, thermal conductivity, thermal expansion, and density, vary with temperature [25]. Because of the effects of thermal gradients that result from firing the refractory shotcrete, these properties may vary throughout the refractory lining section. The design of the refractory shotcrete lining must account for such factors as thermal cycling, thermal shock, chemical attack, abrasion, and erosion. To counter these service conditions, refractory shotcrete uses a wide range of materials. Typically, the 24-h strength of refractory shotcrete is similar to the 28-day strength. Rapid setting of most refractory shotcrete dictates the need for both special application and finishing procedures.

Standards for refractory shotcrete are produced by ASTM Committee C8 on Refractories. Subcommittee C08.09 on Monolithic Refractories describes refractory shotcrete as "cold nozzle-mix gunning" and has produced ASTM Practice for Preparing Refractory Concrete Specimens by Cold Gunning (C 903). In addition, Subcommittee C08.08 on Basic Refractories has also produced ASTM Practice for Preparing Test Specimens from Basic Refractory Gunning Products by Pressing (C 973). An article on the application of prepackaged refractory castables indicates that these two standards are inadequate and several new and coherent standards are needed for refractory shotcrete using a common set of terminology and nomenclature [26].

Fiber-Reinforced Shotcrete

Fiber-reinforced shotcrete using steel fibers was first reported in 1971 in experimental work performed at Battelle Memorial Institute under the direction of D. R. Lankhard [27]. The process and application of fiber-reinforced shotcrete are described in the committee report on fiber-reinforced shotcrete (ACI 506.1R-98) [28]. The report further states that fiber-reinforced shotcrete and conventional or plain shotcrete are essentially the same, the principal difference being the material used and associated properties. In 1998, the report [28] further indicated that design information related to fiber-reinforced shotcrete was limited. Research and application of fiber-reinforced shotcrete over the last two decades have led to a significant increase in the number, type, and materials used as fiber reinforcement. The technology has grown to include steel, natural materials, and synthetic materials, such as polypropylene fibers [29,30]. Aside from conventional shotcrete applications, current fiber-reinforced shotcrete applications include tunnel- and mine-opening support, remediation of concrete structures, and slope stabilization [31–35].

The design of fiber-reinforced shotcrete is alternatively based on common usage, trial and error, empirical observation, rigorous calculation (numerical models), and comparative experimental simulation [33–35]. Typically, the term "design" refers to the process of determining the thickness and the amount of reinforcement. The aim in design is to ensure that flexural, shearing, and torsional demands on the fiber-reinforced shotcrete, for both static and dynamic loading, are satisfied by thickness, reinforcement, and material strength. These aspects may not be considered specifically in terms of a systematic design but may be arrived at as a result of trial and error or empirical observational methods and supplemental testing [35].

Papworth [32] makes an important observation on the basis of laboratory and large-scale testing that fiber type, materials, and shotcrete process have a considerable effect on the performance of fiber-reinforced shotcrete. Thus, there is a need to establish the performance of new fibers, types of fiber, additives, batching, and application methods. In the case of tunnel- and mine-opening support, the design of fiber-reinforced shotcrete linings can be summarized as follows:

- Shotcrete process imposes constraints on the fiber-reinforced shotcrete (FRS) system. There are practical limits imposed on the fiber type and fiber content used because of the application process of shotcrete, i.e., wet-process or dry-process shotcrete.
- Performance of FRS is strongly influenced by specific fiber type(s) and fiber performance.
- FRS systems also are largely dependent on the stiffness of the matrix (cementitious materials) and fiber content.
- The fiber losses typically incurred during the application of the FRS are 20 % for wet-mixture and up to 40 % for dry-mixture shotcrete. These losses need to be considered and compensated for.
- Where crack widths are limited to 1–5 mm, steel and synthetic, fiber-reinforced shotcrete provides significant 40–80 mm deformations.
- Early strength and stiffness can be important in ground support applications.
- There is potentially significant uncertainty in determining the ductility requirements due to tunnel displacements.
- Laboratory-based tests (e.g., standard beams) may not always provide realistic representative data on loading deformation.
- As a minimum, FRS performance criteria should be based on load, displacement, and energy considerations.

Although standards for shotcrete are developed by ASTM Subcommittee C09.46, Subcommittee C09.42 has developed standards for fiber-reinforced concrete and shotcrete, including ASTM Specification for Fiber-reinforced Concrete and Shotcrete (C 1116) and ASTM Test Method for Flexural Toughness of Fiber Reinforced Concrete (Using Centrally Loaded Round Panel) (C 1550). The need for additional standards for fiber-reinforced shotcrete has been recognized, and both subcommittees are expected to participate in their development.

History of Shotcrete

Early Developments

The shotcrete process had its origins around 1911 with the invention of the cement gun, a device that provided the mechanism for projecting cement mortar and plaster onto a surface [36–42]. Around 1908, Carl E. Akeley [36] began his experiments on sprayed cement mortar which culminated in the issuance of U.S. patents in February and May 1911. He eventually sold his patent rights to the Cement Gun Company, which immediately set about improving and promoting the viability of Gunite as a construction process through considerable experimentation and research. The post-World War II period saw a significant upsurge in the use of shotcrete and the introduction of new types of delivery equipment and improved methods of shotcrete application.

Shotcrete Applications

Early engineering literature between 1911 and 1918 describes a wide range of shotcrete applications that includes exterior applications over wood frames; fireproofing of steel structural members; rock surfacing and stabilization; repair of concrete bridges, dams, and sea walls; rust-preventative linings for large steel water pipelines; coating of brick, block, stone, pile, and wood structures; partition walls; boat building; relining of reservoirs, canals, and aqueducts; tunnel linings and relinings; abrasive-resistant linings; refractory linings in the manufacture of steel; coke ovens and furnaces; coal bunkers; and, in general, a replacement for hand-placed mortars and cement plaster. The majority of the Gunite applications developed in the first ten years of its existence are still in use today [36–40]. ACI classifies shotcrete as conventional, refractory, or special [6]. Conventional shotcrete is applied in a range of thicknesses for structural and nonstructural applications; linings and coatings; concrete, masonry, and steel repair; and strengthening and reinforcing of all types of structures.

Shotcrete Systems

Shotcrete Equipment

The term shotcrete encompasses two distinct processes, specifically dry-mixture and wet-mixture shotcrete. A comprehensive description of each process, the constitutive materials, the plastic and hardened physical properties of in-place shotcrete, the range and variety of applications, associated attributes, and primary and ancillary equipment can be found in the *Guide to Shotcrete* [6] and other ACI 506 documents. The description and operating characteristics of equipment for dry-mixture and wet-mixture shotcrete equipment are generally understood and readily available in both commercial and technical literature [6]. Producing high-quality shotcrete that will consistently achieve specified design or performance requirements requires that batching and mixing conform to ASTM Specification for Concrete Made by Volumetric Batching and Continuous Mixing (C 685/C 685M). Dry-mixture shotcrete is usually proportioned and mixed in the field by using portable proportioning and mixing equipment, or, alternatively, both wet- and dry-mixture shotcrete materials may be delivered to the job site by truck from a ready-mix plant conforming to ASTM Specification for Ready-Mixed Concrete (C 94/C 94M).

In some cases, because of logistical, material-handling constraints or lack of available bulk materials, blended prepackaged materials conforming to ASTM Specification for Packaged, Pre-Blended, Dry, Combined Materials for Use in Wet or Dry Shotcrete Application (C 1480) and ASTM Specification for Fiber-Reinforced Concrete and Shotcrete (C 1116) may be used.

Shotcrete Nozzles

The water ring in the body of the nozzle is used in the dry-mixture shotcrete process to introduce water and liquid admixtures to the mix when used. In contrast, in the wet-mixture shotcrete process, the nozzle is used primarily for introducing compressed air at the nozzle to increase the velocity of the ejected material and for introducing liquid quick-set accelerators to the shotcrete. Nozzle manipulation systems and equipment in wide use today include remote-controlled and semiautomatic nozzle booms that are operated in conditions where manual shotcrete application is difficult or is prevented by concerns for personnel safety.

Materials

When Gunite appeared in 1911, the basic materials used in the process were portland cement and well-graded fine concrete sand, with the occasional addition of lime. The constituent materials currently used in shotcrete are in some respects the same but they currently include some additions and refinements. Specifically, lime is typically not used, while fly-ash and silica fume are in common use. The constitutive materials currently used in shotcrete are prescribed in ASTM Specification for Materials for Shotcrete (C 1436). The following is a discussion of the component materials typically used to produce shotcrete.

Binders

The primary binder used in shotcrete has been portland cement that conforms to ASTM Specification for Portland Cement (C 150). Blended hydraulic cements conforming to ASTM Specification for Blended Hydraulic Cements (C 595) or ASTM Performance Specification for Hydraulic Cements (C 1157) may be used. Also, portland cement may be blended with supplemental cementitious materials. For some refractory applications, portland cement can be used, but the predominant binders are calcium aluminate cements with varying purities and alumina contents. There are no ASTM specifications for calcium aluminate cement; information on a variety of refractory cements can be found in ACI 547R, *Refractory Concrete, SP-57* [41].

Supplementary Cementitious Materials

Supplementary cementitious materials such as fly ashes, natural pozzolans, slag, or silica fume may be used in shotcrete. Fly ashes and natural pozzolans should conform to ASTM Specification for Fly Ash and Raw and Calcined Natural Pozzolans for Use in Cement (C 618). Slag should conform to ASTM Specification for Ground Granulated Blast-Furnace Slag for Use in Concrete (C 989). Silica fume should conform to ASTM Specification for Silica Fume for Use in Hydraulic Cement Concrete and Mortar (C 1240). Typical shotcrete applications that utilize these supplemental cementitious materials are described in the *Guide to Shotcrete* (ACI 506–95) [6].

Aggregates

Aggregates used to produce shotcrete are typically required to conform to ASTM Specification for Materials for Shotcrete (C 1436). Shotcrete also may be made from normal, light, or heavyweight aggregates, depending on the application. Normal-weight aggregates should conform to the ASTM Specification for Concrete Aggregates (C 33). Lightweight aggregates should conform to ASTM Specification for Lightweight Aggregates for Structural Concrete (C 330). Heavyweight aggregates should conform to ASTM Specification for Aggregates for Radiation-Shielding Concrete (C 637). Combined grading limits for shotcrete aggregates should comply with grading #1 or #2 identified in Table 1 in ASTM Specification for Materials for Shotcrete (C 1436). Guidance in the selection of grading #1 or #2 is provided in the *Guide to Shotcrete* (ACI 506–95) [6].

Water

Water used for mixing and curing shotcrete in both wet and dry processes should be potable—that is, free from substances that could be injurious to shotcrete. Mixing water should not increase the chloride ion content of the shotcrete. If it does, the total chloride ion content of the water, cement, aggregates, and admixtures should not exceed the values outlined in Table 4.5.4 of Building Code Requirements for Reinforced Concrete (ACI 318–99) [42].

Admixtures

Admixtures for both wet-mixture and dry-mixture shotcrete should meet the requirements of ASTM C 1141. Quick-setting accelerators (quicksets) that induce both early initial and final set in dry- and wet-mix shotcrete are commonly used to apply relatively thick overhead layers of material to tunnel linings and for similar applications. Presently, ASTM C 1398 does not specifically define the time of set for a portland cement mortar containing and accelerating admixture. Because some of the quickset accelerators cause significant reductions in ultimate shotcrete strength, decreased freeze-thaw resistance, and possible reduction in other durability properties, they should be evaluated using samples of accelerated shotcrete under the provisions of ASTM C 1140. Air-entraining admixtures are not typically used in the dry-mix shotcrete process; however, in wet-mix shotcrete, they should meet the requirements of ASTM Specification for Air-Entraining Admixtures for Concrete (C 260). General guidance in the application of chemical and mineral admixtures in wet-mixture and dry-mixture shotcrete can be found in the *Guide to Shotcrete* (ACI 506–95) [6].

Polymers

Polymers, such as latexes and acrylics, have been used as additives to produce a polymer-modified shotcrete. These materials are reported to increase flexural and tensile strengths in addition to reducing permeability and absorption. Currently, ASTM Specification for Admixtures for Shotcrete (C 1141) references the ASTM Specification for Latex and Powder Polymer Modifiers for Hydraulic Cement Concrete and Mortars (C 1438). It should be noted that latex-modified shotcretes are highly susceptible to plastic-shrinkage-induced cracking.

Reinforcing

Reinforcing bars used in shotcrete should conform to ASTM Specification for Deformed and Plain Billet-Steel Bars for Concrete Reinforcement (A 615), ASTM Specification for Rail-Steel Deformed and Plain Bars for Concrete Reinforcement (A 616), ASTM Specification for Axle-Steel Deformed and Plain Bars for Concrete Reinforcement (A 617), ASTM Specification for Low-Alloy Steel Deformed Bars for Concrete Reinforcement (A 706), or ASTM Specification for Zinc-Coated (Galvanized) Bars for Concrete Reinforcement (A 767). Welded wire fabric for shotcrete should conform to ASTM Specification for Steel Welded Wire, Fabric, Plain, for Concrete Reinforcement (A 185) or ASTM Specification for Welded Deformed Steel Wire Fabric for Concrete Reinforcement (A 497) and may be uncoated or coated with zinc or epoxy. When fibers of any type are used, they should conform to the requirement of ASTM Specification of Fiber-Reinforced Concrete and Shotcrete (C 1116). An ASTM standard for anchors used in shotcrete has not yet been developed.

Miscellaneous

Bonding agents usually are not required or typically recommended in normal shotcrete applications. However, if epoxies are used as bonding agents, they should comply with ASTM Specification for Epoxy-Resin-Base Bonding Systems for Concrete (C 881/C 881M). Bonding compounds not properly applied or allowed to harden before shotcrete application potentially can act as bond-breakers resulting in sagging, sloughing, and fall-out of shotcrete. Curing materials for shotcrete should conform to ASTM Specification for Sheet Materials for Curing Concrete (C 171) and ASTM Specification for Liquid Membrane-Forming Compounds for Curing Concrete (C 309). If shotcrete is applied in multiple layers, curing compounds are not recommended and should not be used, so that effective bonding between subsequent layers of shotcrete can be effectively achieved.

Proportioning Shotcrete Mixtures

Prescriptive Versus Performance Specifications

Although the application of shotcrete is very much technology based, it also is a specialized area of activity for most owners and their engineers, who may not use shotcrete extensively. Within the industry, many shotcreting firms have considerable experience. Those in the best position to maintain expertise are those that regularly perform the work. Many have specialized knowledge and equipment and experienced and skilled personnel. Shotcrete specifications should not be so limited or stringent that well-qualified contractors will be prevented from using their innovations and special experience. Specifications should be well thought out and directed to the requirements of the project. They can be prescriptive or performance based, but they must clearly define the intent of the work.

Because the final product depends on strict control of the contractor's operations, prescriptive specifications must be detailed, thorough, and technically correct in every respect. The efforts needed for administration and inspection of work performed under prescriptive requirements typically are much greater than those for work that is based on performance specifications.

Performance specifications require only that a stated result be achieved, leaving the means and methods to the contractor. This encourages contractors to use their greatest ability to achieve the required benefit. All responsibility is assigned to the contractor, which makes administration much easier and greatly reduces the required inspection and testing efforts. This approach requires a clear statement of the required improvements and must designate the means to be used for verification, including, where appropriate, testing of shotcrete. The debate over prescriptive versus performance-based specifications, however, is expected to continue for some time [6].

Selection and Proportioning of Shotcrete Materials

The selection and proportioning of shotcrete mixtures depends on many factors, including the choice of shotcrete process, type of delivery equipment, performance-related requirements, location and environmental considerations, project size, and thickness and volume of shotcrete to be placed in a given time frame. An analysis of a project considering these factors will usually determine which of the two processes, wet- or dry-mixture shotcrete, is most feasible and what type of specification, performance or prescriptive, is most appropriate. Performance specifications usually are preferred because they allow the contractor, with some limits, the choice of materials, proportions, equipment, and methods for achieving the desired result. Prescriptive specifications are more detailed, can impose restrictions, and may constrain the construction approach to a significant extent to prevent the potential for confusing or conflicting requirements.

Typical performance requirements may include but are not limited to the following:

1. Cement-binder type
2. Maximum water/cementitious material ratio
3. Aggregate grading
4. Compressive strength (minimum at specified age)
5. Bond strength
6. Density or absorption
7. Air content (wet-mixture only)
8. Chloride ion content
9. Use of admixtures (Initial/final setting time)

In most applications, the requirements (1–3) may be necessary, the inclusion of the remainder depending on the project-specific shotcrete application. In addition, under special circumstances, the toughness may be specified, as described in the ASTM Test Method for Flexural Toughness and First-Crack Strength of Fiber-Reinforced Concrete (Using Beam with Third-Point Loading) (C 1018). The designer may also want to prescribe the shotcrete process, type of batching, mixing, and shotcrete placing equipment.

Typical prescriptive requirements may include but are not limited to the following:

1. Cement type and amount
2. Aggregate source, type, and grading
3. Permitted admixtures and dosage
4. Supplemental cementitious materials, type and content
5. Mixture proportions or limitations
6. Slump (wet mixture only)
7. Air content (wet mixture only)
8. Water-cement ratio (wet mixture only)
9. Chloride ion content
10. Initial and final setting times
11. Compressive strength (minimum)

Proportioning: General

The basic approach to proportioning shotcrete mixes is consistent with normal concrete practice, especially for the wet-mixture process. However, the shotcrete process can create conditions that require some adjustments in approach. The rebound of larger aggregate particles during shotcrete application usually results in changed in-place physical properties, including higher cement factors, lower coarse-to-fine aggregate ratios, lower water-cement ratios, and a higher specific surface for the aggregate remaining in place. These factors must be considered because of the increased potential for shrinkage and the development of fine surface cracks in the shotcrete. The most practical means of confirming the potential performance of a mixture design is preconstruction or field-testing as outlined in Section 6.4 of the ACI *Guide to Shotcrete* [6]. This approach effectively captures not only the contribution of the constituent materials but also the equipment and the performance capability of the nozzlemen, thus providing an indication of the performance of the total shotcrete system and process. As previously implied, when properties other than strength, such as durability, are determined to be key performance requirements, the shotcrete mixture can be adjusted to include silica fume and fly ash.

Proportioning of Wet-Mixture Shotcrete

Proportioning for wet-mixture shotcrete follows the procedures of ACI 211.1–91 [43] and ACI 211.2–98 [44]. However, the maximum allowable size of the coarse aggregate depends on the material hose size, and the quantity of coarse aggregate used decreases as the size of the aggregate increases. In many wet-mix applications, the sand-aggregate ratio may be as high as 65–75 %. Meeting the air-content requirements of the hardened shotcrete can be a problem because entrained air is lost through the pump and significant amounts of air are lost on exit from the nozzle. If 4 % air entrainment is required in the shotcrete, somewhere between 8 and 10 % may be required at the pump hopper. Maintaining optimal slump is an important factor in wet-mix shotcrete, 50–75 mm (2–3 in.) or less normally being required for consistently uniform placement. Higher slumps in the 100–150 mm (4–6 in.) range often are used with accelerated shotcrete. The use of water-reducers is usually beneficial in helping to achieve the specified properties of wet-mixture shotcrete.

Proportioning of Dry-Mixture Shotcrete

In current practice proportioning for dry-mixture shotcrete usually is less complicated because most mixture designs use very little coarse aggregate, the maximum aggregate size being between 6 mm (1/4 in.) and 9 mm (3/8in.). ASTM Specification for Concrete Aggregates (C 33) contains fine aggregate that can be adapted readily to dry-mixture shotcrete application with minor changes [6]. In most performance specifications, the key design requirement is a compressive strength with a default 28-day strength of 34.5 MPa (5000 psi). Preconstruction testing to confirm the mixture proportions is a suitable alternative if there is a lack of previous data. Generally, preconstruction testing is most appropriate for medium-to-large projects, in which the related testing costs are a small proportion of the overall project budget. It should be emphasized that preconstruction testing may not be necessary on small projects—especially if the performance of the shotcrete mix has been demonstrated on previous similar projects.

Batching and Mixing

General

A considerable quantity of shotcrete that is batched and mixed at project sites is produced using mobile equipment, and stockpiled materials are used as required in the ASTM Specification for Materials for Shotcrete (C 1436). Alternatively, when there are limited lay-down areas, or access or logistical constraints at a project site, bagged preblended materials manufactured according to the requirements in ASTM Specification for Packaged, Pre-blended, Dry, Combined Materials for use in Wet or Dry Shotcrete Application (C 1480) are frequently used. Confirmation of the quality and consistency of shotcrete materials used in these products is achieved by sampling as required in the ASTM Practice for Sampling for Shotcrete (C 1385). These packages contain the cement, aggregates, dry admixtures, and other additives. They usually are available in sealed bags of 22.7–45.4 kg (50–100 lb), in bulk bags of 454–1820 kg (1000–4000 lb), or in other large containers. The individual constituents of the shotcrete mixture should meet ASTM and ACI requirements for shotcrete materials. For proper dry-mixture shotcrete application, the contents of the prepackaged materials should be effectively predampened to facilitate uniform and consistent mixing immediately before field application.

Another traditional source of shotcrete at project sites is that batched at a central ready-mix concrete plant and delivered by ready-mix trucks. Ready-mixed shotcrete can be delivered to the project site as wet-mixture or dry-mixture shotcrete. Time limits on delivery and discharge usually are imposed to prevent significant prehydration losses. If significant prehydration losses are expected, provisions can be made to retard the shotcrete mixture using admixtures meeting the requirements of ASTM Specification for Admixtures for Shotcrete (C 1141).

Batching

Batching of shotcrete materials typically is by weight or volume. Batching by weight is generally the preferred method. On-site mixers use volumetric or manual batching; ready-mix plants use weight batching for cement and aggregates and volumetric batching for water and admixtures. Pozzolans usually are introduced by weight but occasionally by volume, depending on the capabilities of the plant. In the dry-mix process, water is added at the nozzle by the nozzleman at a rate that depends on the appearance of the shotcrete. On projects using volumetric batching, sand bulking and variability in moisture content are potential sources of error in batch proportions. The moisture content and bulk density of the sand should therefore be confirmed when these problems are encountered.

Specifications for weight batching are found in ASTM Specification for Ready Mix Concrete (C 94/C 94M) and for volumetric batching in ASTM C 685/C 685M. For producing fiber-reinforced shotcrete, additional information on the required batching tolerances can be found in ASTM C 1116.

As previously indicated, batch mixture proportions can be achieved manually by using cement in 42.6-kg (94-lb) bags and weight-calibrated containers for aggregates and pozzolans. For dry-mixture shotcrete, standard or quickset accelerators can be added in nonliquid (dry) form during mixing or at the nozzle in liquid form. In the wet-mix process, all admixtures are added during mixing except for conventional accelerators and quick-setting accelerators, which are added at the nozzle in liquid form.

Mixing

Currently, ASTM has only one specification that is applicable to batching and mixing of dry-mixture shotcrete, ASTM C 685/C 685M. However, the *Guide to Shotcrete* (ACI 506R-95) [6] provides excellent supplementary information on good mixing practice. Uniform and consistent mixing is essential for producing high quality dry-mixture shotcrete and for minimizing the occurrence of sand pockets and laminations in the applied material. For wet-mixture shotcrete, mixing requirements should conform to either ASTM C 685/C 685M or ASTM C 94/C 94M depending on the mode of mixing. Additional information on mixing pumped concretes is available in ACI 304.2R-96 [45]. Again, uniform production and consistency in batching and application are essential in the production of high-quality shotcrete.

Miscellaneous

Curing of in-place shotcrete requires the same attention as for concrete to fully develop the strength and durability of the shotcrete. Because shotcrete typically is applied in relatively thin sections ranging from 50 to 150 mm (2 to 6 in.), using curing procedures as specified in ACI 506.2 is important. Ponding or continuous sprinkling provide the best curing conditions, but if these procedures are not practical, curing compounds that comply with ASTM C 309 or sheet materials meeting the requirements of ASTM C 171 can be used. As previously indicated, in order to ensure that bonding is effectively achieved where multiple layers of shotcrete are applied, curing compound should not be used between the applied layers.

Quality Assurance and Testing

Quality Assurance

A project quality assurance program normally should be commensurate with the size and complexity of the shotcrete project. On small projects, quality assurance will typically be minimal. On medium-to-large or complex projects, the quality assurance program would be expected to be much more comprehensive and would include such elements as preconstruction testing, prototype tests or mock-ups, prequalification of shotcrete materials, and certification of the nozzlemen–essentially covering all aspects important to the performance of the shotcrete system. Preconstruction testing as described in the Specification for Shotcrete (ACI 506.2–95) [46] uses the ASTM Standard Practice for Preparing and Testing Specimens from Shotcrete Test Panels (C 1140) and ASTM Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete (C 42/C 42M) for strength determination. Core specimens are typically provided for acceptance testing and construction control and for assessing the condition, quality, and uniformity of the shotcrete in accordance with the ASTM Standard Practice for Examination and Sampling of Hardened Concrete in Constructions (C 823).

Extraction of core samples should be based on a sampling plan that indicates the number of samples and their locations. Specimens obtained for this purpose also can be used to verify the thickness of shotcrete and aid in the visual assessment of shotcrete quality, workmanship, defects, shotcrete-to-substrate bond, and the envelopment of any reinforcement used in the shotcrete. Evaluation of shotcrete properties is discussed in ACI Guide for the Evaluation of Shotcrete 506.4R-94 [47]. Acceptance criteria for shotcrete core strengths usually is established by the specifier of tests.

Testing

Subcommittee C09.46 has adapted ASTM Test Method for Obtaining and Testing Drilled Cores and Sawed Beams of Concrete (C 42/C 42M) and developed ASTM Test Method for Obtaining and Testing Drilled Cores of Shotcrete (C 1604/C 1604M) to account for the differences between concrete and shotcrete that have been previously discussed. As with concrete, compressive strength is considered a good general indicator of the quality of hardened in-place shotcrete. However, depending on the type of structure or application, flexural strength, toughness in addition to bond, permeability, shrinkage, density, absorption, and uniformity also may be important performance parameters. Sampling and testing of hardened shotcrete during construction or on a post-construction basis should be based on a minimum but sufficient number of representative samples. The tests used in a project quality assurance program should provide data and information that, when evaluated and assessed, directly relate to the performance of the shotcrete material or structure and satisfy the purpose of the sampling and evaluation plan.

Nondestructive testing (NDT) methods, typically developed for evaluating concrete structures [48], can be used in evaluating shotcrete. NDT devices and methods such as impact hammers, ASTM Test Method for Rebound Number of Hardened Concrete (C 805); probes, ASTM Test Method for Penetration Resistance of Hardened Concrete (C 803/C 803M); ultrasonic and pulse velocity methods, ASTM Test Method for

Pulse Velocity Through Concrete (C 597); and, where applicable, pullout devices, ASTM Test Method for Pullout Strength of Hardened Concrete (C 900), can be used to determine the uniformity of the in-place shotcrete as described in Guide for the Evaluation of Shotcrete ACI 506.4R-94 [47]. Testing requirements for the plastic state of shotcrete are, in general, similar to those established for ordinary portland cement concrete. Differences between concrete and shotcrete can again arise from the type of application and shotcrete process. Methods for placing ordinary concrete utilize procedures that place concrete in its final position at close to zero velocity with slump varying between 0 and 200 mm (0 and 8 in.). Wet-mixture shotcrete may have a slump at the pump hopper that varies between 38 and 75 mm (1½ and 3 in.), with the in-place material adhering and not sagging or sloughing. Properly placed dry-mixture shotcrete normally has zero in-place slump without subsidence. Current ASTM specifications and test methods are used to qualify the individual materials used in the shotcrete process, except for admixtures, for which ASTM C 1141 replaces ASTM Specification for Chemical Admixtures for Concrete (C 494/C 494M).

Most of the current ASTM test methods, specifications, and recommended practices for concrete and concrete-related materials include four general divisions of concrete technology: materials, manufacture, plastic-state properties, and hardened-state properties. Except for ASTM C 1116, ASTM C 1140, ASTM C 1141, ASTM C 1398, ASTM C 1436, ASTM C 1480, and ASTM C 1550, concrete standards have been adapted in an attempt to account for the processes and practice of shotcrete application.

Summary

Shotcrete has a long and established history as an effective and important tool for a wide range of applications in construction. The use of shotcrete continues to increase, a result largely based on the recognition that as a material, structural system, or component, shotcrete generally has met the expectations of project owners, designers, inspectors, and contractors. In addition, developments in shotcrete technology have resulted in significant improvements in reliability and consistency that have in turn resulted in efficient, effective, and economical construction.

Therefore, it is essential that a comprehensive set of standards for shotcrete be developed to address the current state of shotcrete technology and the testing needs of the shotcrete industry. Such standards will make a significant contribution to the means and methods for the systematic evaluation of shotcrete that will in turn enable industry to confirm the quality and performance of shotcrete. The goal of shotcrete standards development, however, should be achieved in a way that does not impose unnecessary economic burdens on contractors or owners. The cost benefits associated with applying new shotcrete standards for quality assurance or performance confirmation must be seen as relevant and useful by both owners and contractors. Specific areas that currently need to be addressed in terms of standards development are in the plastic and hardened state of shotcrete. These areas represent the biggest challenge in developing new or adapted standards. Those who develop these standards must clearly recognize the shotcrete process, current technology, and the broad range and types of shotcrete application.

Sampling and testing standards are needed to facilitate the shotcrete industry's efforts to evaluate the performance of shotcrete, specifically with regard to the acceptance or rejection of shotcrete. This approach should be based on the use of appropriate sampling and testing methods that implicitly account for the performance of shotcrete as a material, structural element, or component. As emphasized previously, sampling and testing methods must account for both spatial and temporal variability in a way that is representative of the shotcrete structure as a whole. Test parameters should directly relate to key performance or design parameters that constitute the basis for accepting or rejecting both plastic and hardened shotcrete, as indicated in the ASTM Practice for Examination and Sampling of Hardened Concrete in Constructions (C 823).

An approach that could be used for this purpose would be to adapt the guidelines identified in ASTM Standard Practice for Examination and Sampling of Hardened Concrete in Structures (C 823) for shotcrete structures, with sampling and testing on conventional statistics and appropriate geostatistical sampling methods [49].

The current shotcrete subcommittee, C09.46, will continue to review Committee C09 standards and determine which of them are applicable to current shotcrete practice. Where changes are appropriate, Subcommittee C09.46 will continue to develop new standards for shotcrete or to adapt existing concrete standards for shotcrete if necessary.

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Organic Materials for Bonding, Patching, and Sealing Concrete

Raymond J. Schutz[1]

Preface

IN PREPARATION OF THIS CHAPTER, THE CONTENTS of the 4th edition were drawn upon. The current edition will review and update the topics as addressed by the previous authors, introduce new technology, and include up to date references.

The chapter was expanded to include a new standard test method to determine the bond and tensile strength properties of overlays, patches, and adhesives in the field or laboratory.

Introduction

Concrete is one of the most durable materials of construction. However, because concrete is inherently porous and alkaline, it can be attacked both physically and chemically by certain injurious solutions including acids, salts, and water.

Attack of concrete by water or injurious solutions is especially severe where the concrete is exposed to alternate cycles of wetting and drying or freezing and thawing, or both.

Since such attack can only occur in a wet environment, coatings quite often are applied to concrete to prevent ingress of the solution. There is no frost damage to concrete if it is dry [1], and all chemical attack requires the presence of water [2].

Absorption and subsequent chemical or physical attack can also be reduced by polymer impregnation [3] or by inclusion of the polymer in the plastic-concrete mixture in the form of a latex or unreacted premixed epoxy resin and curing agent [4].

Epoxy resins have been used to bond overlays and patches and to bond a variety of materials, such as wood, masonry, metal, and plastics, to concrete.

Organic materials that have been used for sealing of concrete include epoxy resins, silicones, bitumens, linseed oil, oil-based paints, acrylics, urethanes, polyvinyls, styrene butadienes, chlorinated rubber, polyesters, and vinylesters.

Bonding and Patching Materials

Organic materials in common use for bonding and patching of concrete fall into three general groups: (1) bonding admixtures, (2) adhesives, and (3) resinous mortars.

Admixtures

Admixtures that are used to improve the adhesion of plastic mortar or concrete to hardened concrete are classified by the American Concrete Institute (ACI) as bonding admixtures. Mortars and concrete prepared with these materials are classified by ASTM as polymer modified mortar and concrete and are covered in detail in Chapter 52.

Latex Adhesives

ASTM Specification for Latex Agents for Bonding Fresh to Hardened Concrete (C 1059) provides for the classification of latex bonding agents according to use: Type I (redispersable) for use in interior work not subject to immersion in water or high humidity, and Type II (non-redispersable) for use in areas subject to high humidity or immersion in water, also suitable for use in other areas.

Type I latices are generally based on polyvinyl acetate, and Type II latices are generally based on styrene butadiene or acrylic polymers.

There are two test methods available for determining the bond strength of latex systems used as adhesives: ASTM C 1042 Test Method for Bond Strength of Latex Systems Used with Concrete by Slant Shear and ASTM C 1404 Bond Strength of Adhesive Systems Used with Concrete as Measured by Direct Tension.

ASTM Specification Latex Agents for Bonding Fresh to Hardened Concrete (C 1059) requires a minimum bond strength as tested by C 1042 of 2.8 MPa (400 psi) when tested dry for Type I systems and 8.6 I/1Pa (1250 psi) for Type II systems after immersion.

There is no ASTM adhesive specification associated with C 1404. This test method is usually used only to determine the bond strength of polymer modified mortar or concrete.

Type I systems are applied neatly either by brush, spray, or roller. The fresh mortar or concrete can be applied at any time after application of the Type I latex. Since Type I systems are redispersable, the latex may be allowed to dry before application of the fresh mortar or concrete.

Type II latices are mixed with cement or cement and sand and are applied as a slurry or grout. Since Type II latices are non-redispersable, application of the fresh mortar or concrete must be accomplished before the slurry dries. The importance of slurry application of Type II systems is illustrated in Fig. 1 [5]. When applied neatly, the systems do not

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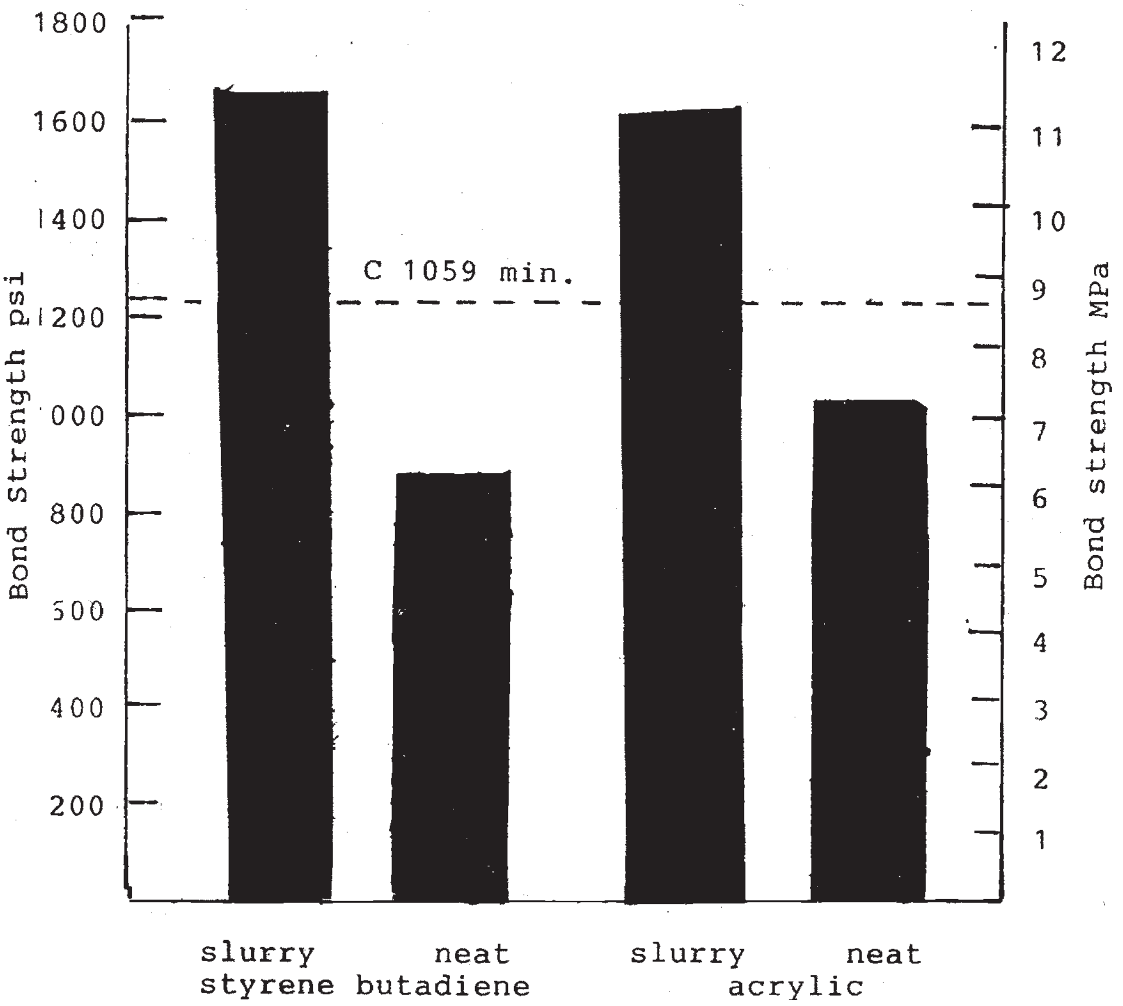

Fig. 1—Example of bond strengths obtained using Type II latices applied as a slurry and neat ASTM test method C 1042.

meet the requirements of C 1059. When applied as a slurry, they exceed the minimum requirements.

Epoxy Resins as Adhesive, Patching, and Overlaying Materials

Epoxy resins are the most widely used organic systems for bonding, patching, and overlaying concrete. Suitable systems are available for use as adhesives (for bonding new plastic mortar or concrete to existing hardened concrete) and also as binders (for the manufacture of resinous mortar and concrete patches or overlays). For these purposes, low molecular-weight liquid epoxy resins of the bisphenol A type (reaction products of the bisphenol A and epichlorohydrin) are generally used. These resins are mixed with a curing agent immediately prior to use. These curing agents are generally of the chemical groups, such as amines, polyamines, or polyamides.

The epoxy resins cure by cross-linking; therefore, curing shrinkage is much less with these than with the polyester or vinylester systems that cure by polymerization. The cured-resin systems will have tensile strengths between 10 and 20 times that of good concrete and will be resistant to alkalies, mild acids, solvents, and oils.

ASTM Specification for Epoxy-Resin-Base Bonding Systems for Crete (C 881) classifies epoxy resin systems by type, class, grade, and color.

Seven types are described by this specification according to their proposed use:

1. Type I – For use in non-load-bearing applications for bonding hardened concrete to hardened concrete and other materials, and as a binder in epoxy mortars or epoxy concretes.
2. Type II – For use in non-load-bearing applications for bonding freshly mixed concrete to hardened concrete.
3. Type III – For use in bonding skid-resistant materials to hardened concrete, and as a binder in epoxy mortars or epoxy concretes used on traffic-bearing surfaces or surfaces subject to thermal or mechanical movements.
4. Type IV – For use in load-bearing applications for bonding hardened concrete to hardened concrete and other materials and as a binder for epoxy mortars and concretes.
5. Type V – For use in load-bearing applications for bonding freshly mixed concrete to hardened concrete.
6. Type VI – For bonding and sealing segmental precast elements with internal tendons and for span-by-span erection when temporary post tensioning is applied.

7. Type VII — For use as a non-stress-carrying sealer for segmental precast elements when temporary post tensioning is not applied as in span-by-span erection.

Three grades of systems are defined according to their flow characteristics and are distinguished by the viscosity and consistency requirements:

Grade 1 — low viscosity,
Grade 2 — medium viscosity,
Grade 3 — non-sagging consistency.

Classes A, B, and C are defined for Types 1 through 5, and Classes D, E, and F are defined for Types 6 and 7, according to the range of temperatures for which they are suitable.

1. Class A — For use below 4.5°C (40°F), the lowest allowable temperature to be defined by the manufacturer of the product.
2. Class B — For use between 4.5 and 15.5°C (40 and 60°F).
3. Class C — For use above 15.5°C (60°F), the highest allowable temperature to be defined by the manufacturer of the product.
4. Class D — For use between 4.5 and 18.0°C (40 and 65°F).
5. Class E — For use between 15.5 and 26.5°C (60 and 80°F).
6. Class F — For use between 24.0 and 32.0°C (75 and 90°F).

The temperature in question is usually that of the surface of the hardened concrete to which the bonding system is to be applied. This temperature may be considerably different from that of the air or the applied material. Where unusual curing rates are desired, it is possible to use a class of bonding agent at a temperature other than that for which it is normally intended. For example, a Class A system will cure rapidly at room temperature.

Two ASTM test methods are available to determine the suitability of epoxy resins for use with concrete:

1. ASTM Test Method for Bond Strength of Epoxy-Resin Systems Used with Concrete (C 882) is a test method that determines the bond strength by using the epoxy system to bond together two equal sections of a 76.2 by 152A-rom (3 × 6 in.) portland-cement mortar cylinder, each section of which has a diagonally cast bonding area at a 30° angle from vertical. After suitable curing of the bonding agent, the test is performed by determining the compressive strength of the composite cylinder.
2. ASTM Test Method for Thermal Compatibility Between Concrete and an Epoxy-Resin Overlay (C 884). Although developed for epoxy-resin systems, this test method may be used to determine the thermal compatibility of any resinous mortar and concrete. In this test method, a layer of epoxy-sand mortar is applied to a slab of cured and dried concrete. After the epoxy has cured, the sample is subjected to five cycles of temperature change between 25°C (77°F) and −21.1°C (−6°F). Crack lines between the crete and the epoxy mortar constitute failure of the test.

Epoxy-resin systems conforming to ASTM C 881 Types I, II, IV, and V have relatively high elastic moduli, since a structural bond is required in most adhesive applications. In the thin glue lines usually used, the differences in linear coefficient of expansion between the epoxy resin and the concrete is inconsequential because the total tensile strength of the thin glue line (1 mm or less) is less than the tensile strength of the concrete. In thin glue lines, creep of the epoxy resin is not a problem because the effective modulus of elasticity as a material in a thin glue line will be extremely high [6].

For applications as an adhesive, the epoxy resin should contain no solvent, since solvent may be entrapped during cure and cause both a rubbery cure and later shrinkage of the adhesive. The epoxy-resin systems may be applied by brush, roller, or spray to properly prepared concrete surfaces. Where reinforcing steel is exposed, the steel should be cleaned of rust, oil, and foreign material and coated with the epoxy-resin adhesive to prevent further corrosion.

ACI 503.1, Standard Specification for Bonding Hardened Concrete, Steel, Wood, Brick and Other Materials to Hardened Concrete with a Multi-Component Epoxy Adhesive, and ACI 503.2, Standard Specification for Bonding Plastic Concrete to Hardened Concrete with a Multi-Component Epoxy Adhesive, cover bonding techniques in detail [7].

Epoxy-resin systems are used quite commonly as the sole binder for concrete patches and overlays. Since such patches and overlays are generally thick, a resin with a low modulus of elasticity, such as those conforming to ASTM C 881, Type III, must be used.

If high-modulus binders are used, the difference in thermal coefficient of expansion between the thick resin mortar and the base concrete will result in failure of the overlay or patch by shearing of the concrete adjacent to the resinous mortar or concrete. When applied in layers 40-mm (1½ -in.) or greater in thickness, it is desirable to include coarse aggregate in the system, both for reasons of economy and thermal compatibility. Figure 2 illustrates the effect of aggregate loading on linear coefficient of expansion of epoxy-mortar systems [7].

The use of epoxy resins as a binder for mortars is covered by ACI 503.4, Standard Specification for Repairing Concrete with Epoxy Mortars [3].

Thin resinous mortars (also referred to as skid-resistant surfacings or resinous overlays) may be economically produced by applying the resin by roller, squeegee, or spray to a horizontal concrete surface and broadcasting fine aggregate in excess onto the uncured resin. After cure, the excess aggregate may be swept off and recovered.

Several applications may be applied if desired. This method eliminates the need for mortar mixers, troweling, and transporting of resinous mortars. These thin overlays have been used for sealing bridge decks and other flatwork for over 20 years and have a good service record.

ACI 503.3, Standard Specification for Producing a Skid-Resistant Surface on Concrete by the Use of a Multi-Component Epoxy System, details this method [7].

Some epoxy-resin systems are water sensitive before cure. Therefore, they can only be used under dry conditions. Systems conforming to ASTM C 881 are able to cure under humid conditions and bond to damp surfaces.

Where large cavities are to be repaired by use of an epoxy-resin concrete, the most economical method is to prepack aggregates into the void and then inject the epoxy resin as a grout from the bottom of the void. This approach displaces air or displaces water in the event the repair is made under water.

Proportioning epoxy-resin mortar or concrete follows the same guidelines as proportioning portland-cement concrete mixtures. However, since the resin systems are sticky and viscous, the sand/aggregate ratio is usually the reverse of that used for portland-cement concrete.

The total of voids in the system should be less than 12 % by volume. Voids in excess of 12 % will result in a permeable mass with resultant poor chemical and freeze/thaw resistance. When

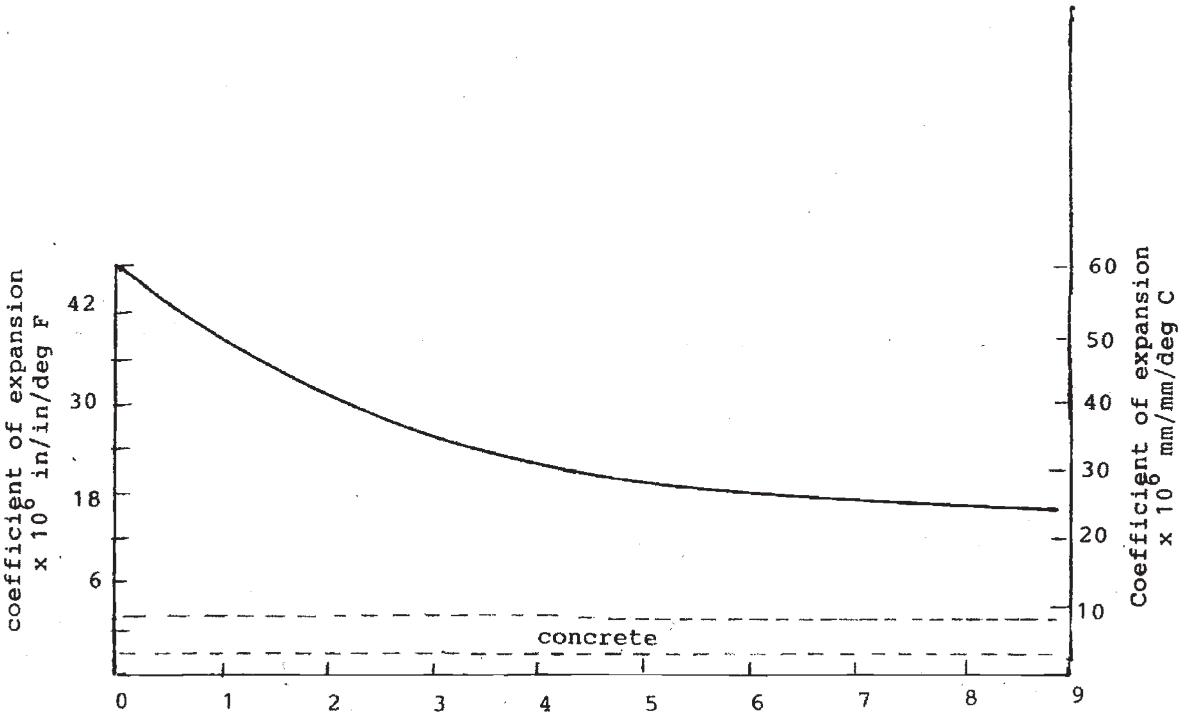

Fig. 2—Effect of sand aggregate-binder ratio on the thermal coefficient of expansion of an epoxy system.

using forms for epoxy-resin mortar or concrete, the forms should be coated with polyethylene rather than oil. Oil may be absorbed into the resin, impairing the cure of the system.

ACI Committee 503 has issued a guide covering the use of epoxy resin in concrete construction [7]. This report should be studied by those planning to use these materials. It stresses the necessity for preparing the concrete surface to be treated before application of the resin system. Three surface conditions must be met if an application is to be successful: (1) the surface must be strong and sound, (2) the surface must be clean (i.e., free of oil, grease, or other contaminants such as residues of curing compounds, waxes, or polishes that may have been applied to the surface of the concrete), and (3) water vapor must not be expelled from the concrete (out-gassing) during application and curing of the system.

It is often desirable to use resins other than epoxies for mortars and overlays. Acrylic, polyester, and vinylester resins have been used successfully. The choice may be dictated by economy, viscosity, cure rate, and specific chemical resistance [8].

The thermal coefficient of expansion of these resins is similar to that of the epoxy resins, and it is recommended that a compatibility test, such as ASTM C 884, be performed prior to use.

In general, the vinylester and the polyester resins have better acid resistance than the epoxies, but, when used for chemical protection, each system should be evaluated or pretested for resistance to that particular chemical exposure and temperature.

Epoxy resins have been used extensively for pressure grouting cracks to restore the structural integrity of a concrete member [7]. For gravity-flow sealing of cracks in concrete decks, the high molecular-weight methacrylate resins are appropriate because of their low viscosity. These resins are available with viscosities lower than 25 cps, whereas the lowest viscosity epoxy resins will be in the range of 100 to 400 cps [9,10].

A test method which has been in use with many variations for over 25 years has now been standardized and published: ASTM C 1583–04, Tensile Strength of Concrete Surfaces and the Bond Strength or Tensile Strength of Concrete Repair and Overlay Materials by Direct Tension (Pull-off Method).

This test method is suitable for both field and laboratory use to determine: the near-surface tensile strength of the concrete surface, the bond strength of a repair or overlay, or the tensile strength of a repair material or overlay or the adhesive used.

The test specimen is formed by drilling a 50 mm (2.0 in.) core into the surface of the substrate and leaving the core attached to the concrete. A steel disk is bonded to the top surface of the test specimen, and a tensile load is applied to the disk. The tensile or bond strength and mode of failure are reported. The modes of failure possible are shown in Fig. 3.

Bituminous Coatings

Bituminous materials include both asphaltic and coal-tar-based materials.

There is considerable difference between asphalts and coal tars. Asphalts are resistant to many mildly corrosive materials but are attacked readily by solvents, oils, and gasoline. Further, unless they are specially formulated, asphaltic coatings may be permeable to water. Asphaltic coatings generally are used to protect foundations, basements, and similar structures from water.

Coal-tar coatings possess a high degree of water resistance and should be considered wherever continuous immersion is encountered. Coal-tar coatings have a tendency to crack and craze when exposed to ultraviolet light or high temperature. The most common use for coal-tar coatings is the protection of underground pipelines. When formulated with epoxy-resin systems, excellent long-term performance has been recorded [9].

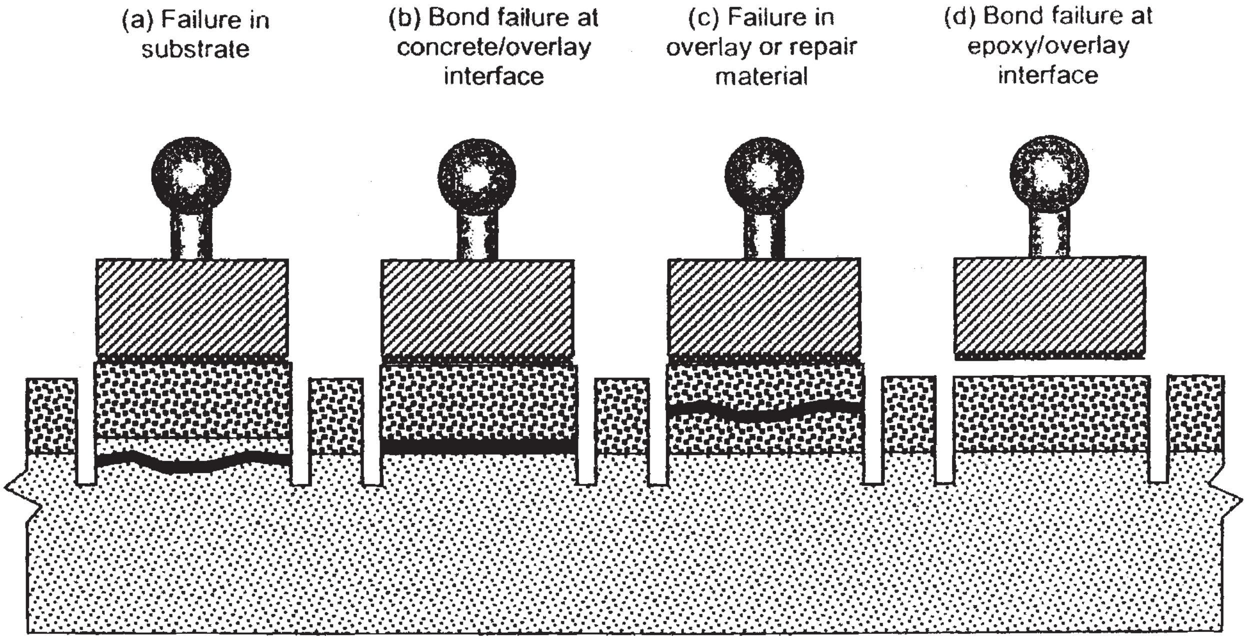

Fig. 3—Schematic of failure modes.

Bituminous coatings may be used with reinforcing fabric and tape to increase their resistance to impact on back-filling and handling. Such reinforcing also increases their thickness as well as their impermeability and service life.

Synthetic-Resin Coatings

Synthetic-resin coatings other than latex-based coatings that are successfully used for sealing concrete are generally applied as solvent solutions and include: coumaroneindene, styrene-acrylic, chlorinated rubber, chlorosulfonated polyethylene, polyurethane, neoprene, and epoxy resins.

The most commonly used coatings in this group are chlorinated-rubber, styrene-acrylate, polyurethane, and epoxy-resin-based. For protection for interior use, where aggressive solutions such as mild concentrations of inorganic acids and low concentrations of organic acids and caustics are encountered, epoxy-resin-based coatings have proven very successful. These may be either polyamine, polyamide, amine-adduct, or acrylic-amine systems [11]. They may be applied as solvent solution, water-based, or 100 % solids systems.

Because these epoxy-resin systems will tend to chalk and yellow, they are not suitable as exterior architectural coatings. The acrylic-amine epoxy-resin systems exhibit better resistance to ultraviolet radiation. However, they are not quite as chemically resistant as the epoxy-resin systems.

The polyurethane-based coatings exhibit extremely good scuff and abrasion resistance and form the basis for many coatings for concrete floors. They are often used in conjunction with an epoxy-resin-based prime coat.

The aromatic polyurethanes exhibit good chemical resistance but tend to yellow on outdoor exposure. The aliphatic polyurethanes have excellent ultraviolet resistance and are the choice for protection of exterior concrete.

Sealing and Curing Compounds

Compounds conforming to ASTM Specification C 1315 Liquid Membrane-Forming Compounds Having Special Properties for Curing and Sealing Concrete can be applied to fresh concrete as a curing compound and will perform as a sealer or may be applied to hardened concrete as a sealer. Chapter 43 covers the use of these compounds as curing mediums. These compounds are the most widely used sealers. They are usually based on chlorinated rubber, styrene acrylate, and styrene-butadiene. They do not harden or dust proof the concrete as such but do provide a protective resinous coating, which will resist the abrasion and dusting caused by foot and wheel traffic.

Although C 1315 is a performance specification, a minimum of 25 % vehicle solids is specified in order to provide a dry film thickness of 0.025 mm (1 mil) at the specified coverage rate. This thickness is considered a minimum to achieve the desired characteristics.

The specification covers two types, Type I (clear or translucent) and Type II (white pigmented). There are three classes based on their resistance to yellowing or darkening on exposure to UV radiation: Class A (essentially non-yellowing), Class B (moderate color change), and Class C (no restriction on color change, for use where color changes are acceptable).

These materials are often used to prevent dusting of concrete, which can affect the adhesion of covering materials such as ceramic tile or vinyl tiles and floor coverings. Tests are included in the specification for bond of commercial adhesives.

Tests are also included for resistance to acids and alkalies. These coatings, when subject to forklift or other vehicular traffic, may exhibit the phenomenon of "rubber-burn," that is, permanent markings or stains from rubber tires. All organic coatings may exhibit this phenomenon to some degree. The two-component polyurethane or epoxy-resin-based coatings have better resistance to rubber-burn than single-component coatings. They may, however, also exhibit staining, depending on the composition of the tires.

Latex Coatings

Latex coatings are relatively insensitive to dampness and alkalinity and have become the most widely used coatings for architectural interior and exterior concrete surfaces. Two types of latices in common use are acrylic and polyvinyl acetate latices. While both are suitable for interior concrete coatings, the acrylic gives the best results in exterior exposure due to its superior ultraviolet resistance. Since these latices are water borne, surface dampness is not a problem. When heavily filled or pigmented, they form permeable coatings, and the presence of vapor pressure does not result in failure.

Sealers

Materials commonly termed "sealers" or "penetrating sealers" are used to reduce the water absorption and chloride ion penetration of concrete. Most of these materials do not change the appearance of the concrete significantly. Sealers do not actually seal the concrete but reduce water inflow. Penetration is also minimal, 3 mm (1/8 in.) being reported as a general maximum [12]. Many act as effective chloride ion shields.

Silanes

Many materials are marketed for this application. The alkoxysilanes, oligomeric alkoxysilanes, and the polysiloxy silanes do not form films but react within the concrete pores to form a hydrophobic layer. As a class, these materials have shown good performance in laboratory tests and field applications and form effective chloride shields [13]. The sealers based on urethanes, epoxies, and acrylics form semipermeable films.

One extensive test program [12] indicated that an alklylalkoxy silane, a methyl methacrylate, an amine-cured epoxy, a moisture-cured polyurethane, and a linseed oil-based sealer performed well in simulated exposure tests.

Other tests [10] indicate that the performance of these sealers is highly dependent on the characteristics of the concrete treated. These tests also indicate that although there are trends in the performance of a generic type, being of a particular generic type does not ensure the effectiveness of a sealer. Therefore, it is recommended that pretesting should be carried out with the candidate sealers and the concrete in question. Such a test method is described by Marusin [13]. The test consists of determining the depth of sealer penetration and weight gain and chloride ion profile after 21 days of ponding with a salt solution.

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Packaged, Dry, Cementitious Mixtures

Dennison Fiala[1]

Preface

PACKAGED, DRY, COMBINED MATERIALS FOR MORtar and Concrete was covered in the three previous editions of ASTM special technical publications, *ASTM STP 169A, ASTM STP 169B,* and *ASTM STP 169C.* Professor A. W. Brust, the Department of Civil Engineering, Washington University, St. Louis, Missouri, was the original author of this chapter for *STP 169A.* A. C. Carter, Manager of Quality Assurance, Texas Industries, Inc., Arlington, Texas, updated the chapter in *ASTM STP 169B,* and Owen Brown, Consultant, Savannah, GA updated the chapter for *ASTM STP 169C.*

Early History

In 1935 packaged concrete mixes first appeared on the market and were soon an ever-growing industry covering a variety of packaged cement based products both for the homeowner and the contractor. Concrete mix was the first product produced and today represents the largest selling product.

The first producers of packaged, dry, combined materials for mortar and concrete either introduced the ingredients separately into the package or mixed them in small increments and placed them in the package. Processes used weigh or volume measurements, or a combination of both to proportion the ingredients of the mix.

Today, high-capacity production lines control proportions. Automated process lines help keep uniformity and quality at a high level of control. Some production lines are able to produce up to 40 bags/s.

The first publication of ASTM tentative specification for packaged, dry, combined materials for mortar and concrete, ASTM C 387, was for information only and was published in the *ASTM Bulletin* in April 1955. The tentative specification was accepted by the Administrative Committee on Standards in 1956. The compressive strength minimums for mortar for unit masonry and for normal-strength concrete were the same as those required by the current ASTM C 387. Compressive strength minimums for concrete mortar (sand topping mix) and other concretes were slightly lower than for the same mixes today (see Table 1 from ASTM C 387-99)

Initially, package sizes were generally larger than demanded by the market today. Products were available from hardware stores, building supply yards, lumber yards, and from the producing locations. These locations were accustomed to handling 42.6 kg (94 lb) bags of portland cement.

Hazardous Considerations

ASTM addresses the hazards of a standard or method with a generic caveat. Packagers provide their industrial customers with a detailed hazardous warning covering the ingredients in the product. This is accomplished with a Material Safety Data Sheet (MSDS) that must comply with Federal Regulation 29 CFR 1910.1200.

Portland cement is known to produce a high pH when in contact or mixed with water. The Occupational Health and Safety Administration (OSHA) and the American Conference of Governmental Industrial Hygienists (ACGIH) have both listed it as a hazardous material with specific permissible exposure limits listed for workers in contact with this material.

Portland cement is classified as a nuisance dust, and exposure can affect the skin, eyes, and mucous membranes. The MSDS for products containing portland cement must address these issues, classify them by acute and chronic exposure, and provide other information including, but not limited to, emergency first aid procedures, fire and explosion hazard data, precautions for safe handling and use, control measures, and disposal of spills.

Silica sand was concluded to be, in 1987, a potential carcinogen by The International Agency for Research on Cancer (LARC). The MSDS for products containing silica sand therefore contain a hazardous statement warning not only of the danger of the inhalation of dust from silica sand and its effect in respiratory disease, including silicosis, pneumoconiosis, and pulmonary fibrosis, but now must also warn of the possibility of cancer.

Warning statements are required on the MSDS and other areas including package wording as well as accompanying data sheets.

Current Specifications

The Products

Although the packaged industry began with mortar and concrete, the list of specifications covering packaged materials continues to grow. The most important step in standardization

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TABLE 1—C 387 Physical Requirements

Kind of Material	Water Retention	Min. Compressive Strength		
		3 days	7 days	28 days
Concrete				
High-Early Strength	–	17.0 (2480)	24.0 (3840)	
Normal Strength				
Normal Weight	–		17.0 (2470)	24.0 (3480)
Lightweight	–			
using normal wt. sand[A]			17.0 (2470)	24.0 (3480)
Lightweight	–		17.0 (2470)	24.0 (3480)
High Strength Mortar	–		20.0 (2900)	35.0 (5075)
Mortar for Unit Masonry				
Type M	75			17.0 (2470)
Type S	75			12.0 (1740)
Type N	75			5.0 (725)

[A] Lightweight concrete using normal weight sand may contain some portion of lightweight fines

of these materials is the requirement that the ingredients used to produce the packaged materials, such as the cements, aggregates, admixtures, etc., must conform with the applicable ASTM standard for that ingredient.

Furthermore, standardized ASTM tests are required to verify the performance of the packaged materials so that the homeowner is afforded the same protection of performance as the contractor or highway engineer who also may use prepackaged products.

ASTM C 387 Packaged, Dry, Combined Materials for Mortar and Concrete

The specification currently covers four types of concrete–incorporating both normal-weight and lightweight aggregates and four types of mortar. The required compressive strength of normal strength concrete is the same regardless of whether lightweight aggregates are used. High early-strength concrete is included in the specification, as are a high-strength mortar and three kinds of mortar for unit masonry. The identification of the mortars for unit masonry are the same as those found in ASTM Specification for Mortar for Unit Masonry (C 270) that is under the jurisdiction of ASTM Committee C 12 and specifies the same performance levels for the Types N, S, and M mortars as ASTM C 270.

The ingredients used for producing these mortars and concretes must comply with the applicable ASTM specification for ingredient compliance. Since it is essential that all ingredients be dry before packaging, a section on aggregate preparation limits the moisture content of dried aggregates to 0.1 % by weight. Each package must produce at least the minimum physical requirements required for that product. Sampling and testing procedures require that standard ASTM methods be utilized.

Packaging and marking include requirements that specify the proper product identification, yield, and amount of water recommended for mixing. Container construction requirements are established to ensure adequate protection against breakage and water absorption by the ingredients in the package. The rejection of products is addressed and may be made by the user for several reasons.

For many years, package sizes were specified, and products had to be produced in certain sizes. This is not the case now, as products can be produced in any size package.

Recently, the governing subcommittee decided to revise two paragraphs on mixing mortar to comply with the property values of C 270.

ASTM C 928 Packaged, Dry, Rapid Hardening Cementitious Materials for Concrete

This specification was first issued in 1980, in response to State Highway Departments' requests to standardize the performance of concrete repair materials being offered in the marketplace. The responsibility for developing this specification was presented to Subcommittee C09.03.17 because the materials would be packaged for distribution.

The principle requirement is a 3-h compressive strength test. Testing cubes for 3-h strength was first addressed in this specification.

The allowable length increase after 28 days in water is 0.15 %, and the allowable decrease in length or shrinkage in air after 28 days is −0.15 %. These length change requirements are based upon an initial length measurement at 3 h age.

Generally, the requirements of this specification are similar to those in C 387. As modern products have ever-faster setting times and early strength requirements, the specification has kept pace since it was issued.

Ingredients used in producing these repair materials are not referenced to ASTM specifications, since many of the cements used for these products are not currently addressed in ASTM documents. This specification therefore uses performance criteria to accept compliance rather than address the constituents of the product by prescription. Testing to verify performance and compliance to the specifications are methods of testing that are ASTM standards, which have been examined and approved by the consensus process. Packaging and marking instructions are part of the specification, and reasons for rejection of products are clearly defined.

In a recent attempt to have the specification invoked by State Highway Departments, a bond strength requirement

TABLE 2—ASTM C 928-99 Performance Requirements

Compressive Strength, minimum, psi (MPa)	**3 h**	**1 day**	**7 days**	**28 days**
Rapid hardening	500 (3.4)	2000 (13.8)	4000 (27.6)	*b*
Very rapid hardening	1000 (6.9)	3000 (20.7)	4000 (27.6)	*b*
Length change, maximum				
Allowable increase, after 28 days in water, based on length at 3 h, %				+0.15
Allowable decrease, after 28 days in air, based on length at 3 h, %				−0.15
Consistency of concrete and mortar:			**Concrete slump min, in. (mm)**	**Flow of mortar min, %**
Rapid hardening, consistency at 15 min after addition of the mixing liquid			3 (76)	100
Very rapid hardening, consistency at 5 min after addition of the mixing liquid			3 (76)	100
Scaling resistance to deicing chemicals after 25 cycles:				
Concrete specimens, max visual rating 2.5				
Mortar specimens, max 1.0 lb/ft^2 (5.0 kg/m^2) of scaled material[c]				

[a] It is recognized that the characteristics and qualities of hardened repair material other than those mentioned in Table 3 might need consideration when certain kinds of concrete repairs are to be made. For the more severe use and exposures that require a higher level of performance, the user is advised to consult with individuals skilled in dealing with such matters.

[b] The strength at 28 days shall be not less than the strength at seven days.

[c] A 10-in. (254-mm) square spalled to an average depth of ⅛ in. (3.17 mm) for 100 % of its surface would have about 2.0 lb/ft^2 (10 kg/m^2) of scaled material.

was added to the specification. It incorporates a minimum requirement for bond strength when tested in accordance with ASTM Test Method for Bond Strength of Epoxy-Resin Systems Used with Concrete (C 882). Both rapid hardening and very rapid hardening materials for concrete repair must exhibit a minimum bond strength of 6.8 MPa (1000 psi) at one day and 10.2 MPa (1500 psi) at seven days when tested by this method. This is another example of the continuing review and improvement of ASTM specifications by the subcommittee and committee structure.

ASTM C 1107 Packaged, Dry, Hydraulic–Cement Grout (Nonshrink)

This specification was first issued in 1989 and was subsequently adopted by the Corps of Engineers under CRD-C 621-89a. The specification currently covers three grades of nonshrink grout, as follows:

- Grade A pre-hardening volume-adjusting,
- Grade B post-hardening volume-adjusting, and
- Grade C combination volume-adjusting.

Performance of the grout in the early volume change (C 827) or height change (C 1090) tests classifies the grout by grade.

In addition to the requirement that the various grades of nonshrink grout must comply with specific minimum and maximum height changes, all grades are required to attain minimum compressive strengths at specified ages.

The minimum physical requirements include compressive strength limits. Nonshrink grouts may be placed at differing consistencies but are required to meet the same minimum performance criteria. This also applies to temperature of placement and maximum working time.

The specification is somewhat cumbersome as compressive strengths, early volume change (C 827), and hardened grout height change must be run on several grout preparations:

1. Freshly mixed grout using highest water/solids ratio (suggested by manufacturer)
   @ Minimum temperature (specified by manufacturer)
2. Grout retained in mixer for maximum working time (supplied by manufacturer)
   @ Minimum temperature (specified by manufacturer)
3. Freshly mixed grout using highest water/solids ratio (suggested by manufacturer)
   @ Maximum temperature (specified by manufacturer)
4. Grout retained in mixer for maximum working time (supplied by manufacturer)
   @ Maximum temperature (specified by manufacturer)

The governing subcommittee has had several proposals over the years for simplifying the specification, but none have been approved. This effort is still underway.

Use of the Products

By Whom

One of the major decisions for the original production of mortar and concrete in packaged units was the desire on the part of the manufacturers to provide these materials in smaller quantities to the do-it-yourself (DIY), homeowner market.

Prior to the availability of packaged mixes, the homeowner would have to buy a minimum of one 42.6-kg (94-lb) bag of cement, and enough bulk sand or sand and gravel for his project. This was not a very convenient method for those who lived in city or urban areas. Ready-mixed concrete then, as

TABLE 3—ASTM C 1107-99 (Table I Performance Requirements)

Compressive Strength, minimum	psi	(MPa)
1 day[a]	1000	(6.9)
3 day	2500	(17.2)
7 day	3500	(24.1)
28 day	5000	(34.5)

	Grade Classification		
	A Prehardening Volume Controlled Type	B Post-Hardening Volume Controlled Type	C Combination Volume Controlled Type
Early-age height change			
Maximum % at final set	+4.0	NA[b]	+4.0
Minimum % at final set	0.0	NA	0.0
Height change of moist cured hardened grout at 1, 3, 14, and 28 Days			
Maximum, %	NA	+0.3	+0.3
Minimum, %	0.0	0.0	0.0

[a] When required, the purchaser must so specify in the purchase contract.

[b] NA = not available.

today, served those who purchased a minimum of one or more cubic meters (cubic yards).

Today the DIY market is served with a variety of package sizes of mortar and concrete from ∼4.5- to 40.8-kg (10- to 90-lb) bags. The sizing of packages permits the family to purchase and carry mortar and concrete home for those DIY projects.

Although the DIY market was the primary purpose for the packaging of mortar and concrete, other user purposes have been served. State highway departments are the largest users of ASTM C 928 rapid and very rapid hardening concrete repair materials. These packaged mortars and concretes are particularly effective in highway repair work where small patches can be made and highways reopened in a matter of a few hours.

Various service companies, such as telephone, natural gas, water, and others, are finding that packaged mortars and concrete are convenient for their repair jobs as they install their much-needed utilities in homes and industry.

Contractors, both small and large, are also learning that the packaged materials are essential to their use in both repair and construction. This is particularly true in the larger metropolitan areas, where space limitations make it more difficult to deposit bulk materials for job-mixed concretes and mortars. Environmental considerations and disposal of excess or waste materials add to the contractor's problem and lead him toward the prepackaged concept.

Applications

The applications for prepackaged materials are many, and the list continues to grow. Both DIY homeowners and contractors are constructing slabs on grade for such uses as driveways, sidewalks, and patios and are using the concretes covered by ASTM C 387. Prepackaged concrete also finds uses in the repair of existing concrete. Concrete mix is also used for other construction and repair needs.

High-strength mortar or sand mix (sand topping) is used where thin layers (less than 50.8 mm (2 in.)) are required. Sand mix is frequently employed for overlays of deteriorated slabs. These applications require the use of a bonding media to ensure that the new topping will not separate from the base slab. In many cases, the overlay may be as thin as 6.35 mm (¼ in.) or even less. High-strength mortar also serves as a mortar to lay brick, especially when the brick is to serve as a walkway, patio surface, or driveway.

Mortar for unit masonry is used for the typical laying of brick and block for various DIY projects, such as the construction of parting walls, retaining walls, and barbecue pits. While most prepackaged mortar for unit masonry is not used on major construction sites, small contractors do use the product for small building projects. Prepackaged mortar for unit masonry is applicable for tuckpointing, where mortar deterioration is evident on existing brick and block buildings.

Rapid hardening and very rapid hardening repair products, covered by ASTM C 928, have their largest application with the highway engineer, where the urgency to patch and reopen highways quickly justifies the use of these more costly materials. The potential for accidents resulting from closed highway lanes is sufficient to make the additional cost of materials trivial.

Because many of these repair products have demonstrated their capabilities in highway repair, they are becoming increasingly popular for use in other concrete repair projects with contractors. These include floors of garages, warehouses, and manufacturing plants needing fast repair in areas that are quickly reusable for vehicular traffic.

Nonshrink grout covered by ASTM C 1107 is the least likely of the prepackaged products to find application by the homeowner or do-it-yourselfer. Contractors will set building columns and heavy machinery by using material that meets ASTM C 1107 in its many consistencies. Its uses are established by the need to ensure positive and complete contact by a supporting member.

Availability

The concrete and mortars covered by ASTM C 387 are available in most, if not all, retail home centers, lumberyards, and hardware stores. These products together with the repair products covered by ASTM C 928 and the nonshrink grouts covered by ASTM C 1107 can be found in most construction supply houses. Most producers of these products do not offer the products or sell from their manufacturing facilities. Arrangements may be made with construction supply houses to make deliveries of full truckload quantities directly to large construction projects.

Quality Control

The term quality control has many meanings. For the producer, quality control is the maintenance of the performance of a product that begins with the raw materials used to produce that product. All manufacturers maintain some form of quality control program in order to ensure that the product they make meets the requirements of the user. The producer is interested in maintaining a uniform production, since this makes his process run more efficiently. Therefore, the uniformity of the raw materials he uses is vital to his efficiency.

For the user, quality control provides the assurance that the product meets the applicable specification. In addition, the user also hopes that uniformity of performance is maintained by the producer.

Both producer and user have the same goals that can only be realized through adequate specifications of the materials used to produce the end product.

Materials

The cementitious components, with few exceptions, are listed within the specifications covered in this chapter by reference to existing ASTM documents. These components are essentially cements covered either by ASTM Specification for Portland Cement (C 150) or ASTM Specification for Blended Hydraulic Cements (C 595), or ASTM C 1157 Performance Specification for Hydraulic Cement. Mortars for unit masonry produced in accordance with ASTM C 387 utilize masonry cements under ASTM Specification for Masonry Cement (C 91) as well as Portland-lime varieties proportioned in accordance with ASTM C 270 where the hydrated lime meets ASTM Specification for Hydrated Lime for Masonry Purposes (C 207) requirements.

The cementitious components permitted in the production of products to meet ASTM C 387 are required to meet existing ASTM specifications. This results from the fact that these specifications are basically prescription specifications. This is not the case with ASTM C 928 and C 1107, prepackaged mortars, where the specifications are written to performance criteria.

The proprietary components used in ASTM C 928 and C 1107 are not covered by ASTM specifications. In many instances, the cementitious components represent special formulations or combination of ingredients that have limited - potential in the area of mortars and concrete and therefore have not been considered for coverage by ASTM specifications.

Aggregate quality is specified in ASTM C 387 by reference to ASTM Specification for Concrete Aggregates (C 33) and ASTM Specification for Aggregate for Masonry Mortar (C 144). These specifications provide for grading and for other aggregate qualities. Aggregates for ASTM C 887, C 928, and C 1107 are not specifically referenced but are left to the discretion of the manufacturer through the use of performance criteria.

The dryness of aggregates is covered in ASTM C 387 under the section, Preparation of Aggregates. In this section, aggregates shall have a moisture content of less than 0.1 % by weight. There are no similar provisions in the other specifications.

Packaging

At one time, package sizes were part of the ASTM specifications, but currently they are not. The construction requirements of packages are included in the specifications, in order to ensure that the ingredients have a reasonable useful life. This is frequently referred to as the “shelf life” of the material. Container construction is specified in terms of water-vapor transmission of the package, determined in accordance with ASTM Test Methods for Water Vapor Transmission of Materials (E 96).

Physical Requirements

Each of the prepackaged mortar and concrete specifications has a table of physical requirements. These tables have been included in this chapter for reference. In general, the physical requirements involve strength minimums to be met at various test ages. Other characteristics are included and vary by product.

Significance of Specifications

Specifications are written because of a need to standardize the performance of products between what the manufacturer visualizes and what the user demands. For this reason, it is frequently difficult to develop a specification with a minimum of options.

The prepackaged mortar and concrete specifications contain a list of physical requirements, usually with minimum values. There are no other limits to which the manufacturer must adhere. Occasionally, the producer is required to produce a mortar or concrete to some higher guaranteed performance.

Rejection of Product

Prepackaged mortar and concrete specifications incorporate provisions for the user to reject the product failing to meet requirements of the specification.

The most obvious reason for product rejection is that the product fails to meet some or all of the physical requirements of the specification.

In addition, the user also may reject a shipment if the package weights vary from the printed weight by more than a specified amount, or if the average weight of a given number of packages is less than that printed on the bag. All broken packages also may be rejected.

Standards for Producer and Consumer

The entire standardization process in ASTM ensures that the development of specifications is unbiased and, therefore, the standards for prepackaged mortars and concrete provide protection for both producer and consumer.

Future Needs of Prepackaged Product Specifications

Concrete Products

There are many additional prepackaged concrete products available on the market that should be applicants for coverage by ASTM specifications.

Fast-setting concrete mix provides both the DIY and contractor user with a prepackaged concrete with fast-setting characteristics. This type of concrete permits rapid utilization of the finished slab or footer and is particularly adaptable for that weekend project or for the small-contractor one-day project.

A fiber-reinforced concrete mix aids the homeowner in producing a finished slab with a minimum of drying shrinkage cracks. Because of the general tendency for the homeowner to use too much water in placing his concrete, this product will help the average user produce a better crack-resistant slab.

Mortar Products

There are many prepackaged mortar products that are designed to provide specific performance characteristics for the user that are being considered by ASTM.

Rapid Concrete Mortar

This specification effort has been underway for some time and is currently before C9.

Shotcrete

Prepackaged shotcrete, frequently called Gunite when the material is to be dry-applied, has been available to users for many years. Both fiber and non-fiber reinforced, modified and unmodified, mixes have been offered to meet specific project requirements. These materials generally utilize fine aggregate conforming to ASTM C 33 and can utilize coarse aggregate meeting the same specification, if desired.

Shotcrete materials are frequently used in underground coal mines to cover and reinforce the ribs and roof to prevent sloughing. They also are employed in major tunnel projects as temporary support during construction of tunnel linings. Shotcrete is also a reinforcing technique used to repair deteriorated poured-in-place concrete structures. Subcommittee C09.03.20 on Shotcrete is preparing a specification for this class of materials. It is anticipated that a prepackaged version, under the jurisdiction of Subcommittee C09.03.17, will follow.

Self-Leveling Flooring Materials

A new class of floor resurfacing products has emerged from Europe for use in both the construction and reconstruction markets. These products need no troweling or secondary placement operations. Many of these materials are pumped in-place and self-level much as the spreading of water. These products are offered in normal or fast-setting varieties, depending on the need to place the finished floor in service

A specification effort is presently underway in Subcommittee C 09.43.

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Self-Consolidating Concrete (SCC)

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Preface

SELF-CONSOLIDATING CONCRETE (SCC) IS BEING discussed in this technical publication for the first time. Being a relatively new material, the industry as well as the scientific community is still learning. Therefore, this document presents a condensed state-of-the-art description that, in time, will need further updating.

Definition

First named High Performance Concrete (HPC) in Japan in the late 80s, the name changed to Self-Compacting Concrete [1] to avoid confusion with the more widely accepted definition of HPC, which is concrete based on the use of low water/cementitious ratio leading to higher strength and durability. Since then, this terminology has been accepted worldwide. However, in the United States, in 2002 a proposal was made to adapt the terminology to make it compatible with ACI terminology. The ACI 116R-90 "Cement and Concrete Terminology" document states that the concrete reaches its final compactness through the operation of *consolidation* and not *compaction*. Therefore, in 2002 a new ASTM terminology was adopted: Self-Consolidating Concrete. In this document, the acronym SCC will be used indifferently of the terminology.

Various definitions have been used in recent years to describe SCC [2]. Most of them share the following common points:

- SCC is fluid enough to fill the forms without internal/external energy;
- SCC remains homogeneous during and after placement;
- SCC is able to flow through dense reinforcement, if needed.

However, the ASTM Subcommittee C09.47 on SCC as well as the American Concrete Institute (ACI) has yet to agree on a common definition.

History

In 1983, Professor Okamura from the University of Tokyo started to investigate the growing durability problems related to concrete structures in Japan. One of his finding was that a major cause of the poor durability performance of these structures was the improper consolidation of the fresh concrete due to unskilled labor on the jobsite. In 1986 he proposed the concept of a high durability concrete requiring no consolidation to achieve full compaction—SCC was born. In the following years, the concept was refined to permit the use of local raw materials, and guidelines for the use of SCC were published in Japanese. It should be noted that concrete requiring no consolidation energy was used previously in the late 70s and 80s, either to increase placing rate or to allow placing in hard-to-reach or highly reinforced sections [3].

Okamura and his team presented the first publication on SCC in 1989 at the Second East-Asia and Pacific Conference on Structural Engineering and Construction (EASEC-2) [4]. Then various publications were presented in the first half of the 90s, which attracted researchers from other countries. As a result, Sweden, the Netherlands, Korea, Thailand, and Canada started their own investigations in the mid-90s in an effort to evaluate the potential benefits SCC can bring to the industry [5–9]. Recommendations and guidelines for the use of SCC were developed through cooperative work in Europe by the late 90s [10–12].

Large construction companies as well as Precast/Prestressed Concrete Products companies also started to take a serious look at this technology, not for its increased durability potential, but mainly for logistic reasons. The early results established that SCC could be poured in a shorter time and required less post-demolding operations than normal concrete.

By the end of the year 2000, the use of Self-Consolidating Concrete (SCC) started to spread throughout the United States. The Precast/Prestressed industry quickly realized the benefits of this technology. The acceleration of the pouring process coupled with the reduction of labor was an obvious motivation for the producers of structural concrete elements. The architectural segment of the industry also saw the aesthetic advantage in using a concrete that easily flows even through restricted areas of the form without vibration, reducing the risks of surface defaults and homogeneity.

The Precast industry is driving the SCC technology implementation in the USA. The Precast/Prestressed Concrete Institute (PCI) has been very active in 2002 with the creation of a *Fast Team* whose task was to draw recommendations on the use of SCC in Precast/Prestressed operations by early 2003 [13].

The Ready-Mix Concrete industry is also experimenting with SCC. According to a recent National Ready-Mix Concrete Association (NRMCA) survey, 22 out of 23 respondents indicated they were either testing or occasionally producing SCC.

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Fig. 1—Akashi-Kaikyo Bridge.

The applications are multiple, ranging from structural columns and walls to floors, tilt-up, and architectural panels. However, the estimated 2003 SCC production is still under 100 000 m^3.

Applications

SCC was first used in 1991 for the construction of bridge towers in Japan [14]. However, the first large-scale project using SCC has been the Akashi-Kaikyo Bridge, completed in 1998 (Fig. 1).

Over 290 000 m^3 of SCC was used for the construction of the anchorages of the bridge. The concrete was mixed on-site and pumped into the forms through a pipe system. It is estimated that the construction time was shortened by 20 %, from 2.5 to 2 years because of the use of SCC. A second application, in 1998, was for the construction of the walls of a natural gas tank for the Osaka Gas Company where approximately 12 000 m^3 of SCC was used. Again, the construction period was shortened from 22 to 18 months while reducing the number of workers from 150 to 50. However, the use of SCC in Japan is still marginal with less than 1 % of penetration in both Ready Mixed Concrete (RMC) and Precast/Prestressed [15].

In Europe, SCC is mainly used in Precast/Prestressed operations. The applications are various and cover most of the wet-cast industry. In the U.S., SCC is mainly used in precast for structural (Figs. 2 and 3), architectural elements (Fig. 4), and utilities (Fig. 5).

SCC can replace conventional plastic concrete in almost any Precast/Prestressed application. It has even been reported used where the SCC had to maintain a 30 % slope [16]. However, because the raw material cost to produce SCC is higher than for normal concrete, the successful use of SCC for a given application is mainly driven by the cost per unit. Therefore, it is difficult to establish precise trends as each plant has different constraints; e.g., SCC can be advantageous for the production of beams in Plant A but not in Plant B.

Fig. 3—Bridge.

It can still be anticipated that, in the future, concrete elements or buildings will be designed considering SCC from the start with shapes, textures, and structures that would be impossible to achieve with normal concrete.

Advantages

As previously mentioned, the obvious advantage of SCC comes from its ease of placement resulting in shorter pouring time. This advantage alone can justify its use for many applications. But there are other advantages.

Surface Quality

SCC's high deformability allows for a better-molded surface quality (Fig. 6). If placed adequately (see section on placement), SCC elements have very few bug holes. Moreover, when leakages occur in the forms under vibration, the concrete will lose enough paste locally to change its surface quality (Fig. 6. dotted line).

As a result, architectural precast concrete producers can significantly reduce the time allocated for patching surface defects after demolding the elements. For architects it also opens new dimensions for shape and textures.

Environmental Benefits

The use of SCC can have a significant impact on the working environment [17]. These are the recommended standards from the Netherlands. Table 1 summarizes the various benefits.

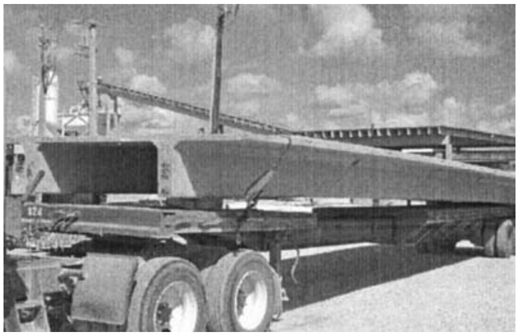

Fig. 2—Double T.

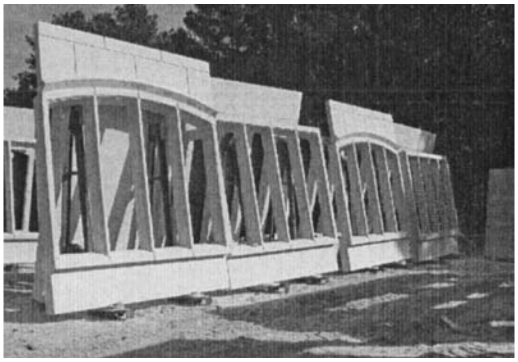

Fig. 4—Architectural panels.

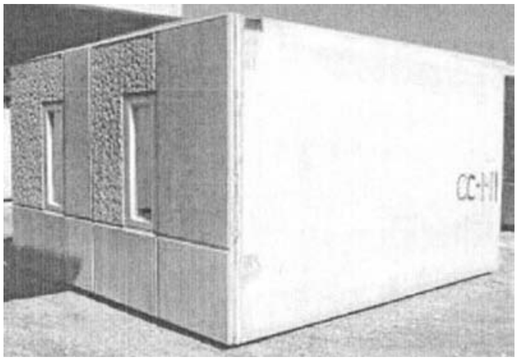

Fig. 5—Jail cell.

A safer and more comfortable working environment can also attract better skilled labor and reduce employee turnover.

Other advantages can also be expected from SCC from an engineering standpoint. These benefits will be reviewed in the *Hardened Properties* section.

Materials

SCC can be produced with any materials used for the production of conventional concrete. However, specific raw material characteristics can help achieve better performances. The raw material selection is an important part of the mix design process for SCC, since it will significantly influence the stability as well as the cost of the mix, which are two key elements in the successful use of SCC.

Cementitious Materials

Hydraulic cement meeting the requirements of ASTM Specification for Hydraulic Portland Cement (C150), Specification for Hydraulic Blended Cements (C 595), or Performance Specification for Hydraulic Cements (C 1157) can be used. Engineering and durability concerns must be considered when specifying cement type. Supplementary cementitious materials such as ground granulated slag, fly ash, or silica fume can also be beneficial to increase the stability of the mix and/or reduce its cost. The quest for a well-graded combination of cementitious materials can help reduce the paste content requirement and, therefore, make the SCC mix more cost efficient. Where the use of ground limestone is permitted, it can help produce highly stable and flowable mixes with a reduced impact on cost. However, the addition of ground limestone may result in increased drying shrinkage values and should be considered during the mixture design and qualification process.

Aggregates

The aggregate should be selected based on their shape, size, and complementarities. Rounded or cubical shaped coarse aggregates are easier to use than angular aggregates as they facilitate flow. There is no limit to the maximum aggregate size. However, the larger the aggregates, the higher will be the requirements in viscosity in order to keep the mix homogeneous as it flows through obstacles.

Natural as well as manufactured sand can be used for SCC. Unlike for conventional concrete, a high proportion of fine particles (passing #200 sieve), as long as they are not deleterious, can be beneficial, as it can increase the stability of the mix and reduce the need for additional cementitious materials. Aggregates meeting ASTM C 33 requirements are appropriate for producing SCC.

Admixtures

SCC can be produced with the same admixtures used for normal concrete. Chemical admixtures meeting the requirements of ASTM Specification for Chemical Admixtures in Concrete (C 494) and air-entraining admixtures meeting the requirements of ASTM Specification for Air-Entraining Admixtures for Concrete (C 260) may be used. To achieve the high fluidity it is recommended to use a High Range Water Reducer (HRWR). Various HRWR will have different effects on the mix stability and, consequently, trials should be made to select the right one for the set of materials available at the plant. The usual air entraining admixtures can also be used to generate the adequate air-void system. When needed, a Viscosity Modifying Admixture (VMA) can also be used to improve the stability of an SCC mixture by increasing its viscosity. Other admixtures such as set accelerators, retarders, corrosion inhibitors, and shrinkage reducing admixtures, just to name a few, can be used with SCC.

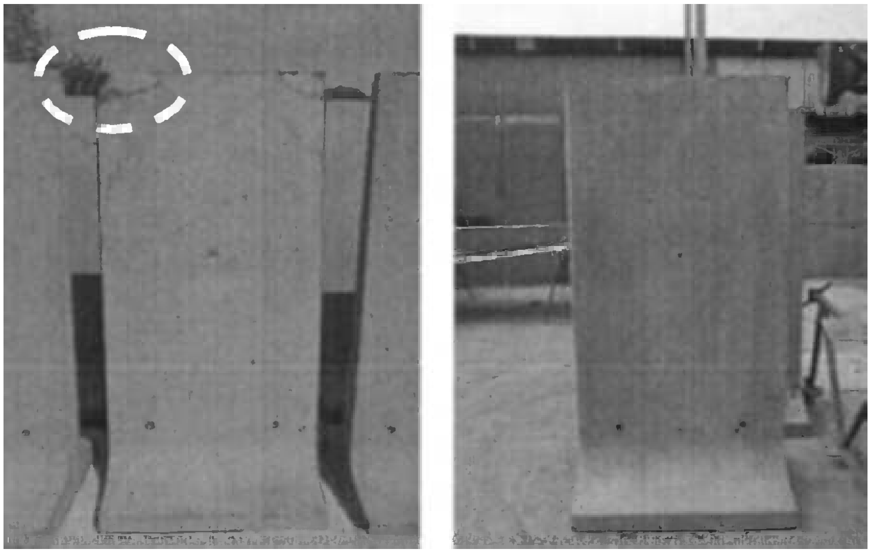

Fig. 6—Surface quality-normal concrete (left) and SCC (right).

TABLE 1—Benefits of SCC

(Recommended Standards from the Netherlands)

	Normal Concrete	SCC	Recommended Limits
Noise Protection	93 dB	< 80 dB	Protective measure required if > 80 dB
Vibrations	0.75 to 4 m/s^2	0 m/s^2	Protective measure required if > 0.25 m/s^2
Quartzite dust	Not measured	0.01 mg/m^3	Max 0.075 mg/m^3
Dust	3–4 mg/m^3	0.2 mg/m^3	Industry goal is < 1 mg/m^3
Savings due to elimination of vibration		Energy Consumption: − 10 % Forms cost: − 20 % Maintenance Cost: − 10 % Illness time: − 10 %	

Mix Proportioning

Various concepts exist for the design of SCC. However, the goal is always to achieve an adequate level of fluidity, stability, and passing ability for the application at the lowest cost. This balance is not easy to achieve and guidance from an experienced SCC technologist may be required. ACI is responsible for mixture proportioning and ACI 211 is developing guidelines.

This section will briefly cover the basic elements of SCC mix design. As for any concrete, the expected performances should first be established (fresh and hardened concrete properties). Then, there are four steps to consider in the mix design methodology:

Step 1: Designing the Paste

The paste is the driving force of strength development and durability. It is important then to take great care when designing it. The paste design involves the determination of the w/c and supplementary cementitious materials replacement ratio and should be based on structural and durability needs. SCC obeys the same relationship as regular concrete with respect to strength and durability and should, therefore, be designed using the ACI and PCI guidelines or project specifications. These documents will guide you in the right selection of cement type, supplementary cementitious materials, and water/cementitious ratio.

Step 2: Paste Content Optimization

The paste content selection is a critical part of the mix proportioning process. The paste is the phase that provides the plasticity/flowability to the concrete. The optimum paste volume selection is a complex process. The concrete starts to behave as a plastic material when all the voids between the aggregates are filled (Fig. 7).

A minimum volume of paste (filling paste) is then required. Because of the high level of fluidity required to achieve full compaction without external energy, SCC requires additional paste volume. This complementary paste volume (fluidity paste) reduces the aggregates interaction as the concrete flows by increasing the distance between the aggregates. The greater the distance between the aggregates, the higher will be the SCC fluidity. ACI Committee Self-Consolidating Concrete is preparing a report that includes placement guidance. However, another factor comes into play to influence the concrete fluidity: the paste fluidity. For a given paste volume, the higher the paste fluidity, the higher the concrete fluidity will be. However, as the paste fluidity is increased by the addition of superplasticizer, its ability to keep the aggregate in suspension (yield stress) decreases. Consequently, concrete fluidity is managed through a well-balanced combination of dry aggregate combination void volume, fluidity paste volume, and paste fluidity level.

Step 3: Aggregate Proportioning

As seen in Step 2, aggregate proportioning will have a direct effect on the filling paste need and, indirectly, on the SCC raw material cost. It is then important to optimize the aggregates proportioning towards the highest possible packing density. For a given set of aggregates, the highest packing density is usually obtained with a high fraction of the largest coarse aggregate. However, when flowing through reinforcement, the largest aggregates often have a tendency to restrain SCC flow by blocking gaps between rebars (Fig. 8).

Consequently, aggregates proportioning should be made in order to reach the right balance between packing density and flowing ability through reinforcement.

Fig. 7—Paste content optimization.

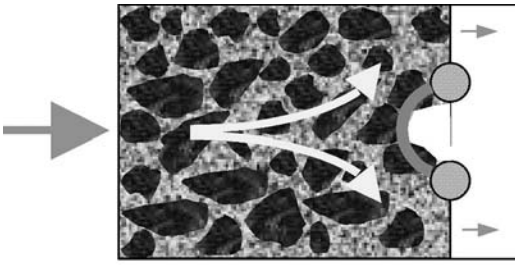

Fig. 8—Aggregate blocking.

Step 4: Admixtures Dosage Adjustments

As with any other type of concrete, the dosage in admixtures required to obtain the desired level fluidity, air content, retardation, or acceleration will have to be optimized experimentally. The reason is that every cement reacts differently with a given set of admixtures. Therefore, it is almost impossible to anticipate the admixture dosages without running test batches.

Batching and Mixing

Concrete batching and mixing systems should meet the same requirements as those for producing conventional concrete. The amount of free water in a SCC mixture significantly influences the concrete's performance [18]. Therefore, excellent control of aggregate moistures is critical during batching operations. Some guidelines require that in-line aggregate moisture probes be able to detect changes in moisture content of 0.5 % [12].

A specific batching sequence of materials may be necessary based on the mixture proportions, materials, and admixtures being used. This sequence should be established during the mixture qualification stage [12,19].

Some SCC mixtures may require longer mixing time to produce a homogenous mixture [20]. However, SCC has been mixed in precast plants using only a 60-s mixing time. The optimum mixing time for an SCC mixture will be influenced by the plastic viscosity of the mixture. A higher viscosity mixture will require longer mixing times than a lower viscosity mixture to reach a steady state in fluidity.

Transporting

Traditional concrete transporting equipment, including agitator trucks, buckets, etc., is used with SCC. Some equipment, such as buckets, may require attention to reduce or eliminate any tendency for leaking. With mixing trucks special attention should be paid to load size if the terrain is hilly due to the fluid nature of SCC and a tendency to spill [21].

If transport is made with nonagitating equipment, special care should be taken to ensure that the SCC mixture being used is stable. This is particularly important if the concrete is to be delivered across an area that will cause jostling and, therefore, impart energy into the concrete. In the absence of agitation the mixture, if not designed properly, may have a tendency to segregate [22].

The workability retention of the mixture being used should be evaluated to correspond with the maximum haul time possible for the project under consideration.

SCC can be discharged and placed much faster than conventional concrete; therefore, precise coordination between delivery and placement is critical. This should be discussed and a delivery/placement plan be agreed upon prior to the start of the job.

Placement

SCC can be placed by most methods used for conventional concrete. If the SCC is to be pumped an appropriately stable mixture is critical to ensure that the paste is not forced past the aggregate resulting in blocked pump lines. Depending upon the viscosity of the SCC mixture being used, pumping pressures may be higher or lower than conventional concrete. The energy imparted to the concrete during placement should be considered during the mixture development stage. Table 2 shows the relative energy imparted to the concrete depending upon the placement equipment/technique used [23]. Each placement technique is given a relative rating in four categories:

- Discharge Rate–The volume of concrete being discharged over a set time frame.
- Discharge Type–Is the energy being supplied during placement intermittent or continuous?
- Single Discharge Volume–How much concrete will be placed prior to the first stop in placement?
- Relative Energy Delivered–This is the overall rating determined as a combination of all of the three aforementioned categories.

All of the four categories, except for discharge type, are given a relative rating of high, medium, or low. Discharge type is either continuous or discontinuous. The relative ratings assume constant drop heights, etc., which would also certainly affect the amount of energy delivered to the concrete.

TABLE 2—Relative Comparison of Placement Techniques and Energy Delivered [23]

Placement Technique	Discharge Rate	Discharge Type	Single Discharge Volume	*Relative Energy Delivered
Truck Discharge	High	Continuous	High	High
Pumping	Medium/High	Continuous	Medium	High/Medium
Conveyor	Medium	Continuous	High	High
Buggy	Medium	Discontinuous	Low	Low
Crane and Bucket	High	Discontinuous	Low	Low/Medium
Auger (Tuckerbilt) Discharge	Medium	Continuous	Medium	Medium
Drop Tube	High	Discontinuous	High	High

If a discontinuous placement technique is used, a mixture that retains its fluidity should be developed to eliminate any chance for pour lines or cold joints. The use of SCC can permit greater design flexibility in concrete elements. If elements are intricate in design or if the formwork has multiple corners, a SCC mixture with a moderate viscosity and relatively higher level of fluidity will be necessary.

The Japanese Society of Civil Engineers (JSCE) has suggested a maximum flowing distance of 15 m or less to eliminate the potential for separation of the paste from the aggregates during placement. In addition, the JSCE has suggested a maximum dropping height of 5 m to ensure homogeneity of the in-place concrete [24]. Other examples have been cited in North America where SCC has been successfully placed with free-fall dropping heights greater than 5 m [25].

The method of placement has been shown to influence the surface finish of pieces cast with SCC [21]. Uncontrolled placement can lead to entrapped air voids that can migrate to the formed surfaces. During placement, the discharging concrete should be flowing in the same direction as the concrete in the form. The rate of placement should be such that any entrapped voids are provided the opportunity to escape.

With lower viscosity SCC mixtures some bleeding of the mixture through gaps in formwork is possible. The majority of the time the gaps are small enough so that they become plugged very quickly with mortar and no problems are experienced. SCC mixtures can be developed to overcome this issue.

Some studies have shown that the pressure on formwork of SCC mixtures may be less than that of vibrated concrete and less than full hydrostatic pressure, but it is influenced by many variables including the type of SCC mixture as well as the casting technique and placement rate [26–28]. Further research is underway in this area to investigate the influence of the concrete's rheology on form pressures. If mixtures are pumped from the bottom up (usually for aesthetic reasons), the pressure on the bottom of the form will never be reduced due to fresh concrete continuously being introduced into the bottom of the form [27].

Excessive form oil can be pushed ahead of the flowing concrete and cause staining of the elements. Therefore, the coating of form oil should conform to the minimum required or as recommended by the manufacturer.

Finishing and Curing

All typical techniques for finishing concrete can be employed with SCC. The timing of the finishing procedures becomes critical. Depending upon the fluidity retention characteristics of the mixture, the surface may not hold a broomed or roughened surface immediately after placement and some time delay may be necessary. In addition, because the volume of SCC that can be placed is greater than that of conventional concrete, a larger surface area of concrete will need to be finished at a single time. Therefore, an adequate number of finishers should be dedicated to any job using SCC in flatwork.

Some SCC mixtures will have a significantly reduced tendency to bleed; therefore, appropriate measures should be taken to eliminate the possibility of plastic shrinkage cracking. The measures required are those similar to when one is using silica fume concrete.

With the mixture proportions used in SCC and a new generation of polycarboxylate high range water reducers, it has been found that in some precast operations steam curing can be significantly reduced or eliminated [29].

Hardened Properties

SCC is produced using the same materials used to produce conventional concrete. Therefore, the hardened properties of SCC follow the same general rules as conventional concrete. Some SCC mixtures are produced using an elevated content of cementitious materials as well as an increased sand-to-total-aggregate ratio (s/a). These adjustments to a mixture may result in a relative increase in drying shrinkage and compressive creep and a decrease in the modulus of elasticity (MOE). However, It should be noted that SCC mixtures could be developed to produce acceptable shrinkage, creep, and MOE values [30].

SCC mixtures can be produced to provide good durability properties. Improved durability (due to absence of vibration)

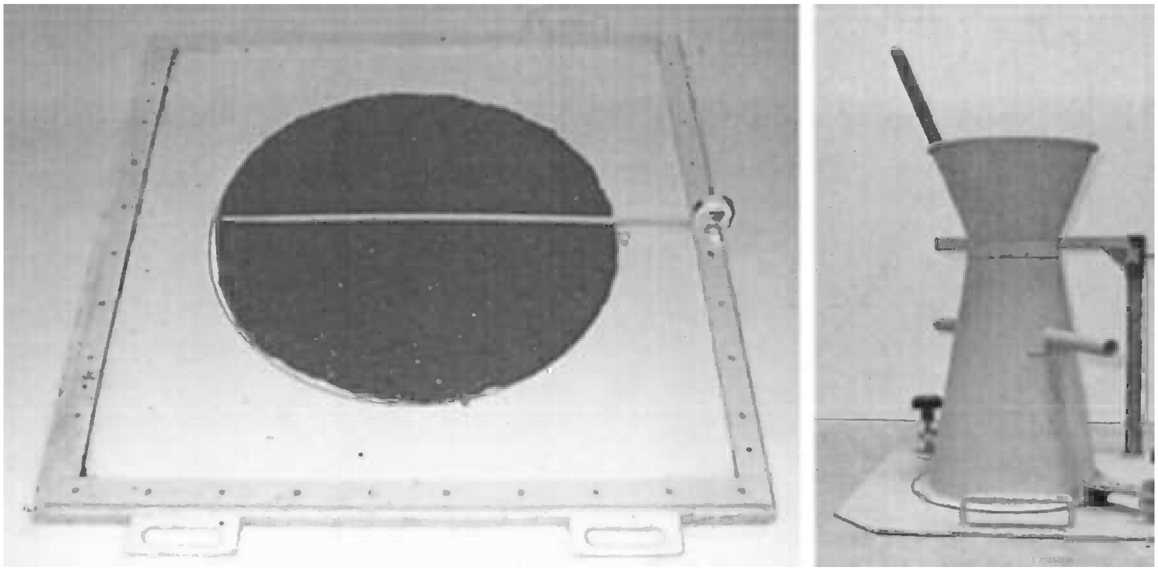

Fig. 9—Slump flow test.

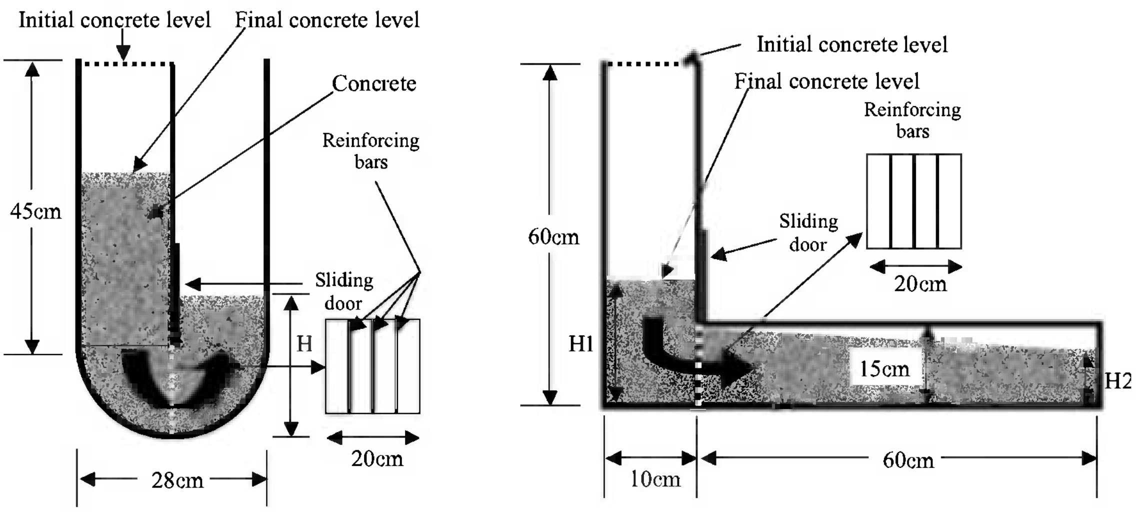

Fig. 10—U-Box and L-Box filling apparatus.

was one of the major reasons for the initial development of SCC. When developing a mixture, it should be noted that the mixture stability will influence the air-void system. If a mixture is unstable it may allow for the coalescing of larger air bubbles resulting in increased spacing factors and decreased specific surface values.

Quality Control

Test methods have been developed worldwide to quantify the SCC characteristics of fluidity, passing ability and stability (segregation resistance). Some methods, such as the column segregation test, are useful for laboratory development and others, such as the slump flow test, are useful for acceptance testing in the field. A few of these methods are briefly presented in the following paragraphs. The dimensions may vary from one country to the other.

Concrete Rheometer

A rheometer is a device that applies a range of shear rates and monitors the force needed to maintain these shear rates in a plastic material. The force is then converted into stress, knowing the flow distribution of the concrete in the device, allowing drawing the stress/shear rate relationship. A few concrete and mortar rheometers are available on the market and have been and are still used for measuring the yield stress, viscosity, and other rheological characteristics of SCC. They are of a tremendous help in the understanding of SCC behavior.

However, this type of equipment is fairly expensive and not easy to use at a job site. Therefore, numerous lighter test methods have been developed for SCC. Neither one of them allows for measuring yield stress or viscosity, but they all simulate more or less real scale casting environments.

Slump Flow Test

This procedure relies on the use of the Abram's cone. The cone is filled in one layer without rodding and the diameter instead of the slump of the concrete sample is measured after the cone has been lifted (Fig. 9). This test is mostly used for evaluating the SCC self-compactibility as it mainly relates to its yield stress.

Monitoring the time it takes for the concrete to reach a slump flow of 500 mm can also be done as an evaluation of SCC viscosity.

L-Box, U-Box

These two tests simulate the casting process by forcing a SCC sample to flow through obstacles under a static pressure. The final height H and H2/H1 for the U-Box and the L-Box, respectively, are recorded. They provide indication on the static and dynamic segregation resistance of a SCC (Fig. 10), as well as its ability to flow through reinforcements. They are frequently used in the field as an acceptance test method.

V-funnel and Orimet

By monitoring the time it takes for the SCC to flow through an orifice under its own weight, these two test methods give an indication of its viscosity. Both tests are used in the field and are sometimes used as acceptance tests. The V-Funnel is presented in Fig. 11.

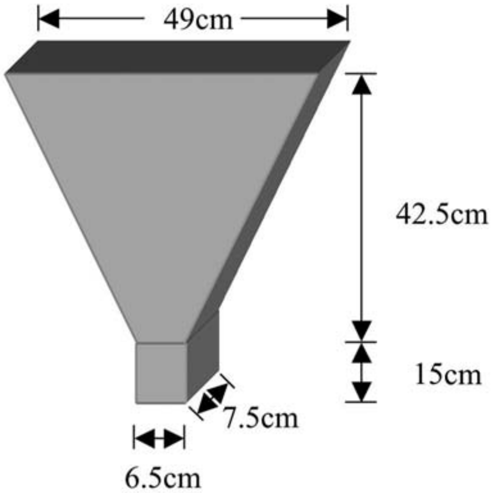

Fig. 11—V-Funnel apparatus.

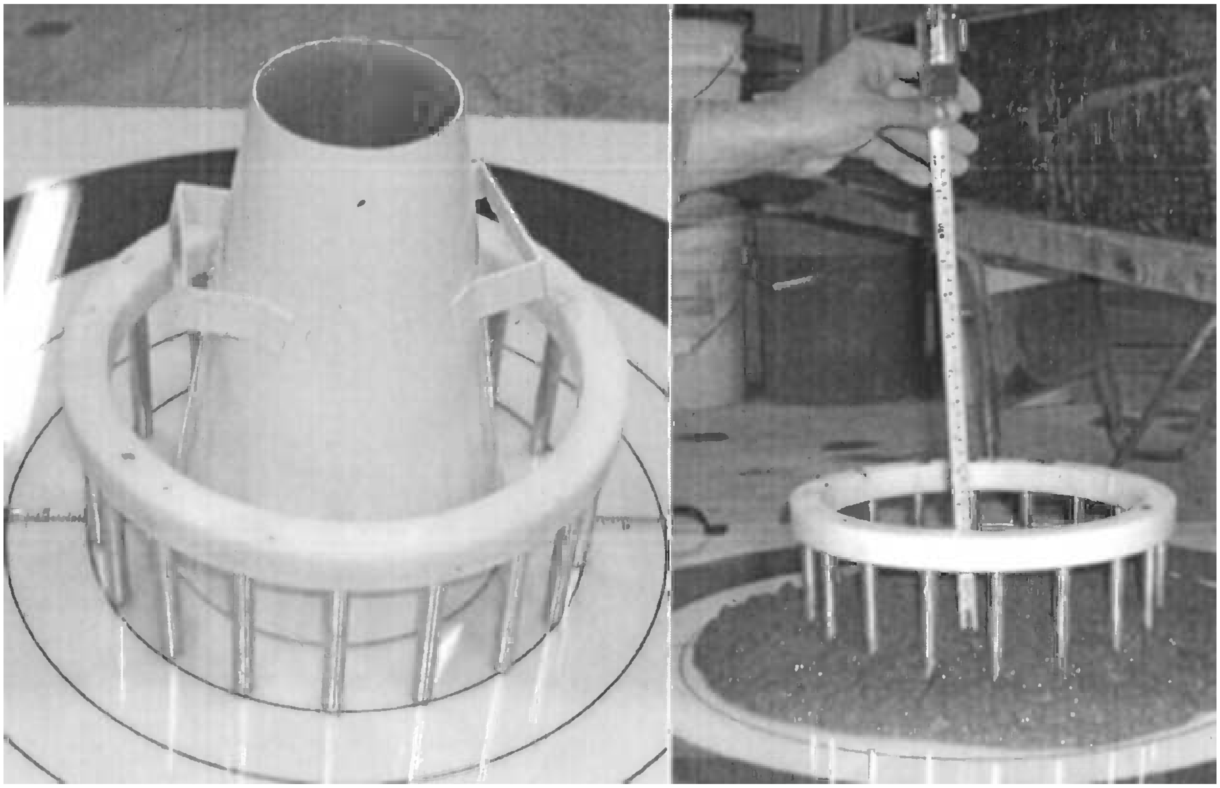

Fig. 12—J-Ring.

J-Ring

This apparatus is used to force the SCC to flow through reinforcement (Fig. 12). It must be used in conjunction with an Abrams cone or the Orimet setup. The concrete is flowing from the inside to the outside of the ring. The size and the spacing between the bars can be adjusted to simulate any reinforcement configuration. The differences between the spread with and without the ring or the height difference between the concrete inside and outside the ring are measured.

German studies showed that with a bar spacing equivalent to 2.5 times the maximum aggregate size, the spread difference with and without the J-Ring must be smaller than 50 mm.

Sieve Stability

This procedure is used to evaluate the resistance to static segregation of a SCC. A sample of concrete is poured over a 5-mm sieve and the amount of mortar passing through the sieve in a 2-min period is measured. The French Civil Engineering Association has published a complete procedure (in French and English) in July 2000 [10].

As mixtures are being qualified in the laboratory, some thought should be given to establishing appropriate quality control criteria. For example, it has been shown that the fluidity level of a given SCC mixture, proportioned with consistent raw materials has a direct impact on the segregation resistance of the mixture [22]. Figure 9 shows how the relationship between the column segregation test and slump flow can be used to set quality control parameters. One can find the point at which the segregation factor exceeds some value (in this example 10 %), the maximum slump flow value then is set at this level minus one inch. In this way the simpler slump flow test can be used rather than the column segregation test for quality control/consistency testing.

Conclusion

SCC is considered a high-performance concrete. It is high performance in the plastic state. This advancement in concrete technology has the potential to change concrete construction in the years to come. Once it becomes a more mainstream technology, structures will be designed and constructed with SCC in mind. This is good news for the concrete industry.

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