

APPLICATIONS OF LIGHT MICROSCOPY IN CONCRETE RESEARCH

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SYNOPSIS

The applications of light microscopy in concrete research are outgrowths of its applications in petrology, mineralogy, and chemistry, but there is more quantitative emphasis in its use in concrete research than is common in petrology. Metallographic and combined petrographic and metallographic techniques are used to study portland cement. Normal petrographic methods are used to study aggregates and cement-aggregate reactions. Air content and bubble spacing in concrete are investigated by linear traverse and point-count techniques. The use of light microscopy in making comparative studies of the microstructure of concrete is described.

SUBJECTS FOR MICROSCOPY IN CONCRETE RESEARCH

The major ingredients of concrete are the cement, the aggregates, and the mixing water with which the cement reacts by hydrolysis and hydration to form the hardened cement paste which envelops and bonds the aggregates together. Microscopy is used in concrete research in the investigation of portland cement and other cementitious or pozzolanic materials used as supplements or partial substitutes for portland cement, in the investigation of natural and artificial aggregates, and in the investigation of hydrated cements, mortars, and concretes. The polarizing microscope was the first type to be used; at present, polarizing, metallographic, and stereoscopic microscopes are all employed.

In the general field of concrete research the first extensive use of microscopy was made in the investigation of portland cement, a specialized area in high-temperature physical chemistry, that is closely related to experimental petrology, and to studies of ceramics, refractories, slags, and aluminous cements. The polarizing microscope was the standard tool used in the identification of phases and compounds formed in such high-temperature phase research. It is a most important tool, particularly before X-ray methods became available as alternate techniques, for the identification of specific crystalline compounds. It was usually possible to confirm the results of microscopy by some other chemical or physical test. This situation tended apparently to make the microscopists cautious and specific in their interpretations; aware that their results needed to be related to other tests of the same materials; concerned with confirming what they saw by other means; and interested in technique and in quantitative methods.

The use of microscopy in the examination of natural aggregates developed later and might be regarded as an offshoot of descriptive petrography and related to the petrography of building stone and roadstone. In this field, the microscopist's results have been less subject to external confirmation, interpretations have been less cautious and more generalized, and interest in

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quantitative methods, specific information, and refinements in technique have been more limited than in the microscopy which is more closely connected with high temperature plus physical chemistry.

The microscopy of the individual constituents of portland cement, their hydration products, and ground cement developed along with the microscopy of unground portland-cement clinker. There were difficulties in obtaining crystals of convenient size, and even greater difficulties in the preparation of thin sections of neat cement, mortar, and concrete, because the hydration products may become altered during specimen preparation. The first great problem to be overcome is the problem of preparation, since no satisfactory interpretation can be produced without assurance that the sample examined correctly represents the mortar or concrete and not some alteration product developed during preparation.

Up to 1940 it was generally assumed that aggregates were normally chemically inert toward cement paste. Most of the problems of concrete which had been regarded as connected with aggregate were blamed on properties of the aggregates such as softness or high absorption. But in 1940 reports of what was termed cement-aggregate reaction or alkali-aggregate reaction began to accumulate, and it presently became clear that some aggregates were not inert under some circumstances.

A review of microscopy in concrete research begins logically and historically with microscopy applied to the investigation of portland cement. Attention to the microscopy of aggregates and concretes developed later and at about the same time. The microscopy of aggregates developed rapidly in some respects, but except for the petrography of alkali-aggregate reaction, the microscopy of concretes was largely neglected. It seems reasonable to consider aggregates after cement, to discuss alkali-aggregate reaction as a separate topic, and to leave for the last the most complex and neglected problem, the microscopy of concretes.

PORTLAND CEMENT

Le Chatelier's investigations of portland cement began to appear in 1882 (1); the date may be taken as a starting point in the use of the microscope in portland cement technology. Bogue (2) summarized progress from that time to the early 1930's in a way that indicates the importance of microscopy in the development of research in portland cement:

“There cannot be said to have been any precise knowledge of the constitution of clinker prior to the microscopic examinations of Le Chatelier and Tornebohm (3). These examinations were substantially in agreement with respect to the classification of phases, denoted by Tornebohm, *alite*, *belite*, *celite*, *felite* and an *isotropic residue*, but the chemical nature of the phases has remained a matter of controversy up to recent years.....

“Alite was considered to be $3\text{CaO}\cdot\text{SiO}_2$ by Le Chatelier in 1884 and this view has been confirmed by the investigations of Rankin and Wright (4) who demonstrated the existence of the compound, Bates and Klein (5) who showed its presence in clinker, Hansen and Brownmiller (6) who identified its presence in clinker by X-ray, Guttman and Gille (7) who separated the alite from clinker, and many others. But in 1931, conceding the evidence of

the X-ray means, Guttman (8) closed the book with the proclamation 'At last the fifty year old question "What is Alite" is answered.' A year later Janecke (9) withdrew his entry, $8\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$, and a half century after the original announcement of Le Chatelier, the alite of clinker is generally acknowledged to consist 'essentially' of tricalcium silicate.*

"Modern progress is the result chiefly of advances in the technique of systematic study. Little could have been accomplished without the generalizations provided by an application of the phase rule within the various systems encountered in the complex of cement clinker. But the successful application requires tools for the positive identification of the phases that are produced by the heat treatments. The microscope was used by Le Chatelier and Tornebohm, but improvements and new developments in its use have opened up vastly greater possibilities in the resolution and identification of the clinker constituents. The X-ray was first applied in cement research in 1927 (10), but already it has become indispensable in the recognition of the cement compounds and in the still more basic understanding of the atomic rearrangements that occur in the cement kiln."

The techniques employed in the classic investigations of the constitution of cement were immersion and thin-section methods. While immersion mounts were most useful for the identification of constituents, and thin sections for the display of textures and structures, neither was particularly suited for the determination of quantitative relations among clinker constituents.

In 1934, Tavasci (11) demonstrated that the constituents could be distinguished in properly prepared etched polished sections examined under vertical illumination. Insley (12) extended the application of this metallographic technique, improving the etching methods, and developing polished thin sections (13) which permitted accurate correlation of observations made in transmitted and reflected light. He also adapted the linear traverse technique, using the Wentworth integrating stage (14) to make quantitative estimates of the constituents in polished sections (15). Micrometric procedures are difficult to apply to thin sections of clinker because the thickness of the section is greater than the grain diameters of some of the constituents. Due to the resulting overlap, it is particularly hard to distinguish the interstitial constituent which form the matrix between the cement silicates. On polished sections the boundaries are distinct and measurements can be made and reproduced more precisely.

Quantitative determinations of constituents both in laboratory and commercial clinker have been made in a number of laboratories in this country and abroad. The original interest at the Bureau of Standards was connected with studies aimed at the production of low-heat cement (12). The Portland Cement Association Fellowship used this approach to determine the effects of heat treatment and cooling rate on the structure of clinker (16). The Corps of Engineers included micrometric analyses of clinkers among the procedures in a research program intended to develop an improved specification for cement (17). Users of the method have tried persistently to improve it

* X-ray investigations recently published indicate some disagreements about crystal structure. See H. O'Daniel and E. Hellner, "Structure of $3\text{CaO}\cdot\text{SiO}_2$," *Neues Jahrb, Mineral. Geol., A*, pp. 108-111 (1950); J. W. Jeffrey, "Crystal Structure of Tricalcium Silicate," *Acta Crystallographica*, V. 5, pp. 26-35 (1952); and "The Tricalcium Silicate Phase," paper No. 2, Third International Symposium on Chemistry of Cement, London, 1952.

(16, 2), particularly by making variation in etching reagents and techniques with the aim of obtaining a clearer discrimination among the interstitial constituents. When the purpose is to determine the variation in composition with clinker-pebble size, whole pebbles are mounted and a cross-section polished and etched. If the purpose is to determine composition micrometrically, samples of the whole clinker, crushed to pass No. 8 sieve and be retained on No. 16, are quartered, cast in Bakelite plugs, and a section of plug is polished and etched.

A recent publication by Brown (18) contains an interesting account of modifications in technique, observations of habit and association of constituents in clinkers, further discussion of the influence of cooling conditions on structure and texture, and comparison of micrometric results with chemical and physical tests. Nine entities could be recognized and measured in one integration on a Wentworth stage with the arm extended to carry nine micrometer screws. The constituents* measured were C_3S , C_2S , C_3A , C_4AF , free CaO, free MgO, dark prismatic interstitial material, glass, and undifferentiated. Dark prismatic is interpreted as essentially C_3A crystallized under particular conditions. The most satisfactory etching technique used distilled water at pH 6.8 to 7.0 applied by pouring it on a velveteen-covered lap just ahead of the face of the specimen. The etched surfaces produced were free of the dark film often formed when the specimen is dipped in the etching reagent, and permitted separation and measurement of eight of the phases in one integration instead of the two or three necessary when several etches are used.

AGGREGATE

The description and classification of rocks for use as concrete aggregate is the petrography of aggregates. The methods employed should depend on the complexity of the sample and the economic and engineering importance of the intended use; thus the amount and kind of microcopy in a petrographic examination should be adjusted to the information needed. Proper field and laboratory sampling, sufficient sample size, and comparable calculation of results from sample to sample are vital if petrographic examinations of aggregates are to yield satisfactory information proportionate to the expense incurred in making the examination. While microscopy of some sort is most important in almost every petrographic approach to aggregates, many other aspects must be considered (19).

The petrographic investigation of building stone and roadstone has produced information still useful in the interpretation of some aspects of the behavior of concrete aggregates. Lovegrove's experiments (20) indicated that loss by attrition in the Deval machine and resistance to fracture by impact were related to texture, mineral composition, and extent of alternation of the rocks tested. Dense unaltered igneous rocks with microgranitic, micrographic, and ophitic textures, which are all fine-grained and highly interlocked, showed the least loss by attrition; the greatest resistance to fracture by impact was found in quartz-rich rocks with fine grained textures. Lord (21) reported that deep-seated igneous rocks with granitic granular textures were harder but less tough than their fine-grained near-surface equivalents. The rocks were classified by examining thin sections. Mineral composition of medium-grained rocks was determined using a reticule divided into 100 squares mounted in the ocular, counting the number of squares subtended by each mineral in the field at 55 X, and averaging 20 such determinations in one section. Fine-grained rocks were examined the same way, using an objective of higher power to give a final magnification of 105 X. Rocks of

* The common abbreviated symbols for cement constituents use C = CaO, S = SiO₂, A = Al₂O₃, F = Fe₂O₃.

variable or coarse grain size were examined at 30 X with an ocular grid of 25 squares; two or more thin sections were used in each determination. The Wentworth stage (14) or the Chayes point-count stage (22) now permit similar and probably superior estimates of mineral composition to be made in less time with less fatigue.

The first record of petrographic description and classification of concrete aggregate located by the author is C.W. Tomlinson's 1915 account (23) of a most interesting method that he developed under the direction of M.O. Withey and A.M. Winchell for use in Withey's survey of the concrete aggregates of Wisconsin. The procedure consisted of separating a sample of sand weighing about 300 g on four sieves, separating each sieve-fraction into four specific-gravity classes by heavy liquids, examining and counting each class in each size-range using a hand-lens or microscope, and computing a weighted average composition for the sample from the weights and counted compositions of the separates. One of the reasons given for separating the sand into sieve-fraction is that "the resulting information is of value, since the composition of sand differs in the several sizes according to definite principles"(23) — an early statement concerning an empirical law still overlooked by many sedimentary petrologists but likely to be impressed on anyone who makes a long series of petrographic examination of natural sand.

G.F. Loughlin (24) made one of the earliest investigations of a failure of concrete ascribed to the properties of constituents in the aggregate. The concrete was cast stone made with aggregate manufactured from altered anorthosite. Loughlin inspected the deteriorated concrete in place, visited the quarry, examined thin sections of the aggregate and thin sections of unaffected and deteriorated cast stone made from it. Considerable portions of the quarry sample and the sand in the deteriorated concrete had altered to a zeolite, laumontite, which characteristically loses water and falls to pieces when it is exposed to air. The sand in the unaffected concrete was relatively free of that kind of alteration.

Loughlin next reviewed the qualifications of different kinds of natural stone for concrete aggregate (25), writing from the viewpoint "of the geologist interested in the correct use of stone but inexperienced in the mixing and handling of concrete." Perhaps his most valuable specific emphasis was on the potentialities for trouble from clay distributed in aggregate so as to be accessible to water. Almost all of the rest of Loughlin's review seems to be based on the literature dealing with building stone and roadstone and on considerations of the behavior of rocks exposed to natural weathering. There seemed to be no recognition of the importance of particle surface or of the differences in bond which particle surface can cause. The solubility of calcite was considered without mention that the cement matrix of concrete may contain more easily soluble materials. But these omissions were almost inevitable considering the stated viewpoint, and the paper probably represented as much of a contribution as could be made without benefit of detailed examinations of concrete and without the realization that performance of a particular aggregate in a particular concrete is a question not of the properties of the aggregate a such, but of the interrelated properties of the aggregate and the matrix in the circumstances of the exposure.

The interest aroused by this review caused several engineers to send Mr. Loughlin samples of limestones with poor service records or suspicious test results from Pennsylvania, Illinois, Iowa, and Kansas. The results of the examination of these samples became the subject of a paper published the next year (26). Service records and determinations of absorption and loss in the sodium sulfate test were available for most of the samples. Hand specimens and thin sections of the rocks were examined and the amount of material insoluble in hydrochloric acid was determined. The mineral

composition of the insoluble residues was investigated microscopically by C.S. Ross of the United States Geological Survey. All of the limestones contained montmorillonitic clays. Variations in amount of clay, size and distribution of clay pellets, absorption, and distribution of permeable bedding planes and fractures appeared to explain the differences between beds in the same quarry and from quarry to quarry. Small differences in amounts and arrangement of clay accounted for differences in test results and in service. It is interesting to recall that this well-documented account of troubles due to swelling clays in limestones was based on diagnosis made with the light microscope, supplemented in one instance by chemical analysis.

Wuerpel and Rexford (27) investigated the correlation of absorption, bulk specific gravity, and loss in the magnesium sulfate test of the chert in 12 samples of gravel. All of the chert examined was composed of cryptocrystalline quartz rather than chalcedony or opal. They found that loss in the magnesium sulfate test increased with absorption and with decrease in bulk specific gravity. Using thin-section, polished-section, and powder-mount techniques, they concluded that as bulk specific gravity decreased, microscopically visible pores increased except in some porous cherts which had some of their pores filled with hydrous iron oxide which increased the bulk specific gravity. But these filled pores did not decrease the absorption or loss in magnesium sulfate by a comparable amount.

The most extensive effort in recent years to relate mineral composition, texture, and amount and kind of alteration of rocks to behavior in service and tests was made by the Knights (28) in their book, "Road Aggregates." While their major emphasis is on aggregates for bituminous mixtures and stone for sett paving, many of the relations between textures and alterations and physical behavior are illuminating when applied to the interpretation of freezing-and-thawing test results.

ALKALI-AGGREGATE REACTION

In 1940, Stanton (29) reported instances of failure by expansion and cracking in concrete pavements in California which had been observed from 1923 on, at first near the coast from Monterey to Los Angeles County and later near the San Joaquin River. The failure of a section of pavement north of Bradley, California, in 1938 was correlated with the use of sand from one source and cement containing over 1 percent total alkali calculated as Na_2O . The laboratories of the California Division of Highways made extensive engineering tests of the sand and of other California aggregates, and developed a test procedure to determine reactivity. The procedure consisted of making mortar bars of the aggregate and a high-alkali cement, storing the bars in a sealed container over water, and measuring changes in their length at intervals up to one year.

The first rock demonstrated to cause expansion in this test was described as "siliceous magnesian limestone" (30) or opaline dolomitic limestone from the Monterey group of California. Its mineral composition was determined microscopically, and samples of each mineral were made up in mortar bars for test by the method outlined above. Only the bars containing opal expanded (31). Further microscopic examinations of other California sands demonstrated that all except one of the sands which had been used in concrete that had shown this type of expansion and cracking contain opal in small amounts, as opaline chert, opaline shale, or opalized wood.

Reports of similar failures accumulated soon after Stanton's paper appeared. Meinser (32) described the cracking, expansion, and exudates found at Parker Dam two years after its completion in 1938.

Careful examination of cores drilled from the dam indicated that the gel exudates were usually associated with andesitic pebbles. Some andesite and rhyolite pebbles were found with dark outer shells regarded as differing from those produced by weathering, in that the matrix of the rock in the shells was soft but feldspar and mica phenocrysts were hard and fresh. A small amount of secondary opal was found in some of the andesite but not in all of it. The aggregate had been tested by the usual methods and by petrographic examination and had been regarded as satisfactory material. The reactive ingredient in the andesite had not been identified; feldspar was suspected. In discussion, Insley (32) pointed out that amorphous silica, as opal, had been demonstrated as reactive in California, and andesite was a rock which often contained amorphous silica as volcanic glass.

Additional instances of expansion and cracking diagnosed a alkali reaction were recorded from Virginia (33), the State of Washington (34), Nebraska, and Idaho (30). Stanton, Porter, Meder, and Nicol(30) established, by examination of concrete and thin sections, that reaction had affected andesites from Parker Dam, Friant Dam, a traffic circle at Fresno, Calif., and bridges in Washington state; the common factor appeared to be the glassy groundmass of all of the andesites. They synthesized andesite glass and found that mortar bars made with high-alkali cement and the synthetic glass expanded and cracked. Following up indications Stanton had found, Coombs (35) investigated the possibility of detecting aggregate which had reacted by means of ultraviolet light and staining thin sections. Reacted concrete showed more fluorescent rims than unreacted, but the results were not entirely satisfactory. Interpretation of the stained areas in sections was complicated by the presence of clay minerals and hydrous iron oxides which absorbed the dyes, and it was uncertain whether the dye absorption had anything to do with reactivity. Hornibrook, Insley, and Shuman (36) found that tridymite and the natural glasses, obsidian and pitchstone, also produced expansion of mortar bars.

Parsons and Insley (37), in 1944, reported investigations of the effects of alkaline solutions on polished surface of rocks and mineral known to have reacted with alkalis in cement or suspected of possible reactivity. They suggested a test for potential reactivity which consisted of immersing polished specimens of rocks and minerals from the aggregate in a 10 percent NaOH solution maintained at 50 C for 18 to 24 hr; etching produced on minerals or rock except calcite or dolomite would be an indication of potential reactivity. The test was not widely applied.

The identification of reactive materials based on tests in mortar bar and on diagnose of field concrete established a category of materials which had been shown to be reactive. The list now includes opal, chalcedony, tridymite, cristobalite, acid and intermediate volcanic glass, pyrex (38), and silica gel (39). If chalcedony is assumed to consist of a submicroscopic mixture of fibrous low-temperature quartz and interstitial opal (40), all the substances listed above may be described as consisting of or containing "highly siliceous materials which are thermodynamically metastable at ordinary temperatures." (40) That description does not appear to cover the hydromica (38) that is a suggested reactive constituent of the Buck Dam phyllite. Very fine-grained quartz (38) had also been suggested as the reactive constituent at Buck Dam, and its status appears uncertain (40) in terms of reaction at normal temperature.

Hansen (39) advanced a hypothesis to explain the alkali-aggregate reaction, based on experimentation, chemical considerations, and examination of mortars and concretes at low magnification. It may be summarized as follows: SiO₂ as opal or as pure silica gel would react with NaOH or KOH in the solution phase in the cement paste, presumably associating as complex

silicate ions, assumed to be incapable of diffusing through the paste. The cement paste would then act as a semipermeable membrane. It would allow water and the ions and molecules of the alkali hydroxides to pass through it without allowing passage of the silicate ions and would thus form an osmotic pressure cell. The alkali silicate formed on a silica particle would draw solution from the paste and produce a pocket of liquid which would exert hydrostatic pressure. The silicates would occupy greater volume than the original particle and thus increase pressure against the surrounding paste. An additional reaction between alkali-silicate solution and $\text{Ca}(\text{OH})_2$ might occur to form calcium silicate and regenerate the alkali hydroxides for further reactions. The transparent gel-like product of reaction was regarded as the alkali silicate and the white opaque or porcelaneous product as the calcium silicate. The difference in behavior between $\text{Ca}(\text{OH})_2$ and alkali hydroxides with silica was believed to be due to lower solubility of calcium silicates than of alkali silicates.

McConnell, Mielenz, Holland and Green (41) provided an interesting summary of criteria for the recognition of alkali-reaction, with additional information on reaction products in concrete. They reviewed their experience with many proposed tests for reactivity and gave a large accumulation of results obtained in mortar-bar tests. The primary criterion cited as proof of alkali reaction was the presence of gel reaction product in hand specimens or thin sections of the concrete. The gel ranged from colorless and transparent to opaque and white, and in consistency from thin and watery to rubbery to hard and brittle. It was found in voids or cracks, as rims around aggregate particles, or in pore space in aggregate particles. The indices of refraction of the gel varied with moisture content, ranging in desiccated gels from about 1.46 to 1.49. The second criterion given was the presence of reaction rims around aggregate particles. To be applicable, it must be demonstrated that the rims formed after the incorporation of the aggregate in the concrete and that the aggregate was a type capable of reaction. The rims appeared dark in reflected light but more translucent in thin sections than the interiors of the aggregate particles. The precautions necessary in applying this criterion were clearly described.

Some differences of opinion about the location of the osmotic pressure cells hypothesized by Hansen have been recorded. The petrographers of the Bureau of Reclamation (41,42) have considered the sockets around reactive particles or the outer shells of the particles as the location of the semipermeable membrane and have apparently regarded the pressures as caused by increase in volume of the reacting aggregate particles. As supporting evidence, they have published a number of photomicrographs of concrete in which the majority of the fractures appear to originate at aggregate particles and die out in the matrix. Parsons and Insley (42) studied the movement of gels formed by reaction between opal and high-alkali cement in pastes and mortars by microscopic examination of broken surfaces and thin sections. They observed that, in their specimen, the gel which formed around the opal grains moved into surrounding mortar and filled available voids; cracks subsequently developed, apparently originating at the voids and dying out in the matrix. The author has observed some cracks which appear to start from aggregate particles, some which appear to start from voids, and many which did not appear to be related to anything in the plane of the surface or section being examined.

One type of crack which appeared frequently in chert particles in the concrete of Tucaloosa Lock (43) was later found in less marked degree in chert particles in mortar bars; it appears to be a useful indicator of chert particles in which reaction has begun and does not appear to be a normal feature of the Gulf coast chert gravel before they are incorporated in concrete. In the Tucaloosa cores, many of the chert particles on sawed or drilled surfaces showed one crack, of greatest width in the

interior of the particle, often dying away before the outside of the pebble or sand grain was reached, but sometimes passing the boundary to dwindle to nothing in the mortar. Several stages were observed and illustrated (43), including empty fractures confined to the aggregate, empty and partly filled fractures passing into the mortar, and some crack systems passing through several chert pebble, wider in the pebbles and narrower in the mortar. The bond of the surfaces of such pebbles to the mortar is usually extraordinarily good, much better than is to be expected of smooth polished chert, so good that the concrete will frequently break through rather than around the pebble. The shape of the cracks, their usual location inside the pebbles, and the unusually good bond, tempt one to imagine that they could have been formed by a tensile stress moving the whole exterior of the pebble out away from the center.

Although an enormous amount of work has been done since 1940 in the attempt to control alkali-aggregate reaction, to identify and characterize reactive constituents, and to explain the chemistry and mechanics of the reaction, a great deal remains to be done. The evidence obtained by microscopic examination of reacted concrete has been useful but is in its nature indirect; frequently it has been ambiguous, particularly regarding the mechanics of the expansion and cracking.

Fear of the possibility of alkali reaction has been an active influence in increasing the use of petrographic examination in acceptance testing of concrete aggregate. The same fear has increased the care and detail with which the examinations are conducted. The particular value of petrography in this respect is that if an adequate sample is studied with proper care by a competent petrographer, it can indicate definitely whether reactive constituents are present, and if found, their nature, amount, and distribution with respect to particle size.

CONCRETE

Microstructure

Any microscopist working on cement hydrates, mortar, and concrete soon discovers that the literature is widely scattered, hard to locate, and apparently scanty. Much information of value to the microscopist is found in publications with titles which do not so indicate. Many of the publications of the Portland Cement Association Fellowship and the Bureau of Standards may be overlooked on that account.

In 1915 Rankin and Wright's paper (4) on the system $\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$, Tomlinson's method (23) for mineralogical analyses of sand, and two of N. C. Johnson's papers (44) on the microstructure of concrete appeared. Johnson had accumulated many specimens of concrete exposed to fresh and salt water and had examined polished surfaces at magnifications up to 160 X. Apparently he was not interested in the composition or structure of portland cement or in the optical identification of hydration or alteration products. As a result of this disregard for the necessary basis of interpretation he misinterpreted some of the phenomena he illustrated; some of the misinterpretations were discussed at the time by Messrs. Bates and Spackman.

Some of the background needed was already available (45). Considerable additional information on the nature and optical properties of the hydration products of pure compounds in cement (5, 46, 47) and on crystalline substances common in hydrated cement and concrete (48, 49) became available in American journals before 1935. Brown and Carlson's studies (50) of hydrated cements in thin section are of great value because they describe and illustrate a wide range of variations in texture of

hydrates, variations related to water-cement ratio, fineness, curing conditions, and admixtures, with information about tensile or compressive strength of several of the groups and observations of the behavior of the specimens during preparation. These photomicrographs and descriptions permit comparisons of textural differences in specimens of recorded composition and age, cured under laboratory conditions. Additional information on the effects of increased curing time on texture was provided by Ward (51). While several interesting and valuable microscopic and chemical investigations of deteriorated concrete that had been subjected to chemical and physical attack or chemical attack (52, 53, 54) have appeared, the work by Brown and Carlson and the work by Ward seem to provide the only published basis for extrapolation about undeteriorated hydrated cement or mortar in concrete in thin sections.

It has seemed to the author that the texture and microstructure of concrete examined in thin section should provide information as useful in the interpretation of the history of the concrete as is the information provided by microscopic petrography in the interpretation of rocks — if the considerable problems of preparation of thin sections could be overcome, if a wide enough range of comparative material could be assembled, and if the interpretation was made cautiously and checked against all the other kinds of information that might be obtained about the concrete.

Thin sections of satisfactory quality can be prepared by cooking the blank in a synthetic resin until the surface to be ground is impregnated, grinding the surface with progressively finer abrasives in some fluid other than water (kerosene or isopropyl alcohol, depending on the solubility of the resin), mounting the ground surface on a slide, allowing the mounted blank to cool slowly so that thermal shock to the resin is avoided, and grinding the mounted blank to satisfactory thickness. Thickness can be judged by the birefringence of the calcium hydroxide, which should show a maximum interference color about first-order yellow between crossed nicols. If calcium hydroxide is absent (as it may be with thoroughly hydrated concretes containing an efficient pozzolan or in advanced cases of alkali reaction or leaching), unhydrated C_3S should show dark gray, or quartz in the aggregate, pale to medium gray interference colors.

Such thin sections, of the order of 10 to 20 μ thick, are desirable because the unhydrated cement grains, minute aggregate particles, calcium hydroxide, and alteration products have a grain size so small that they overlap and interfere in a thicker section, and the gel ground mass is often fairly dark and opaque even in a section of less than standard thickness.

The constituents which can be recognized in thin sections of undeteriorated portland-cement concrete are aggregate particles, unhydrated cement, calcium hydroxide, and the amorphous gel groundmass. The most common secondary alteration product is calcium carbonate which, in concrete which has been exposed to air but not to excessive leaching or chemical attack, is usually confined to thin zone near the outer surface of the structure and thin zones along cracks. High-sulfate calcium sulfoaluminate is another common alteration product which may be found in small quantities in air voids in normal concrete several years old, particularly if the concrete was made with Type I or Type III portland cement. A thin section of dense adequately consolidated concrete, under 10 years old, taken from a location inside the structure and away from cracks, will probably contain only aggregate, unhydrated cement, calcium hydroxide, and gel groundmass — a restricted group of constituents. The variations which can be observed from concrete to concrete are variations in amount of each constituent, in size of constituents, particularly hydroxide crystals and crystal groups and gel areas, and variations in arrangement of constituents. The close attention paid to the

arrangement, crystal size, and abundance of calcium hydroxide is because it is crystalline, anisotropic, recognizable, and presumably capable of yielding information indirectly about the state of the gel and whether the concrete has been subject to leaching and chemical attack.

The four pairs of photomicrographs are reproduced as examples of the difference in texture and in size of calcium hydroxided crystals and gel areas which may be found among undeteriorated mass concretes. They were selected as representative after comparisons of five sections from each of four cores, each core taken from exterior concrete of a different dam. Photographic data and information about the concretes is shown in Table I. The concrete of cores A and B (Figs. 1 and 2) contained limestone coarse and fine aggregates, while C and D (Figs. 3 and 4) contained granodiorite and diorite coarse and fine aggregates, respectively. One conspicuous difference between the two sets of pairs is the difference in crystal size of the calcium hydroxide between the concretes with limestone aggregate and the concretes with siliceous aggregate. Those with limestone aggregate have relatively small hydroxide crystals and those with siliceous aggregate have relatively large ones. This general relation appears to obtain in the comparisons made so far between undeteriorated concretes with limestone aggregate and concretes with siliceous aggregate; it exists between concretes of the same age and cement factor made with the same cement and what is intended to be the same gradation of fine aggregate. Another difference which appears to be consistent between the concretes with limestone aggregate and the concretes with siliceous aggregate is a difference in amount and particle size distribution of material finer than the No. 200 sieve in the aggregate. The reported amounts of aggregate finer than the No. 200 sieve were larger in A and B than in C and D; the concretes presumably contain what is reported plus some additional amount which is either that adhering to coarse aggregate and not allowed for or that produced between the point where samples were taken for sieve analysis and the final location of the concrete, or both.

The additional unreported fines can be expected to amount to more in limestone aggregates than in siliceous aggregates, because of the differences in hardness between the minerals in the rocks. In the crushing of brittle crystalline solids, the amount of new surface formed per unit of energy input increases as the hardness on Moh's scale decreases (55). Calcite has a hardness of 3; quartz has a hardness of 7; the range of hardness in the feldspars is from 6 to 6.5. The same expenditure of energy yields about 1.8 times as much new surface on calcite as on quartz and feldspar (55). Assuming that the limestones and the granodiorite and diorite were all similar in original grain size and interlock and were subjected to the same amount of accidental impact during batching and mixing, the amount of new surface produced on the limestones should be about 1.8 times that produced on the diorite and granodiorite, and the fines should be proportional. As a matter of fact, the limestones were of finer grain; the degree of interlock was hard to compare between limestones and the diorite and granodiorite. Comparing these sections and all the other fairly comparable sets that could be obtained, it did appear that material finer than No. 200 was likely to be of different particle size distribution, generally smaller in the limestone aggregates but varying from one limestone to another.

The difference in crystal size of the hydroxide and the difference in abundance and size of the aggregate passing the No. 200 sieve implies another point that the photomicrographs illustrate — there are larger areas of gel uninterrupted by crystalline particles in the concrete with siliceous aggregates. This last point seems to be correlated with the tendency to crack in the cores from which the sections were made; when sawed surfaces of the concretes were examined at 10 X after several

months exposure to air, B had more cracks per 100 sq in. of surface than A, and A and B both had more than either C or D.

Air Content

As the practice of entraining air in concrete has become more widespread particularly in the last ten years, there has been increased interest in means of determining air content of plastic and hardened concrete. Verbeck (56) described a method in which polished specimens with pigment rubbed into the voids were mounted on a microscope equipped with a camera lucida. Magnification in the microscope was 50 X to 90 X; magnification in the camera-lucida image was greater and was determined in order to calculate the average void area. The camera-lucida image of a field of known dimensions was traced, using separate colors to distinguish voids from aggregate particles. Planimeter measurements were made of the total area of air voids and of aggregate particles, and the number of air voids was counted. These measurements, with the dimensions of the traced area and the final magnification in the tracing, were used to calculate air content, average void area, and void concentration. The measurement of four fields per section and two sections per specimen was recommended to obtain representative results. The results obtained were in good agreement with determinations of the air content of the plastic concrete.

In discussion of Verbeck's paper, Rexford (56) briefly described four modifications of the linear traverse technique: one using 5 X to 10 X magnification of a ground surface on a fairly large specimen such as a 6-in. diameter core or cylinder; another using photographic enlargement of a fairly large specimen, with traverses measured on the print; the third using photomicrographs of thin or polished sections taken at a magnification to make the smallest voids visible, measuring traverses on the print; and the fourth using thin sections and a Wentworth integrating stage.

Brown and Pierson (57) have described a large motor-driven integrating stage working on the principle of the recording micrometer stage with two motions, and a carriage accommodating specimens up to 6 by 10 in. The specimens used are sawed ground slices of concrete. The traverses are measured during observation of the specimen with a stereoscopic microscope at magnification of 30 X to 40 X in oblique illumination. One micrometer is used to measure air and the other to measure coarse aggregate and mortar. Brown and Pierson found that with this equipment, measurement of a traverse length of 100 in. on a 6 by 8-in. specimen of concrete with aggregate of 1 1/2 in. maximum size would determine the air content of the sample with a standard error of 0.4 percent. If a counter is added to record the number of voids per traverse, it is possible to calculate the bubble spacing factor, which Powers (58) has shown to be important to the frost resistance of concrete. A similar integrating stage has been constructed and used by the Missouri State Highway department (57).

An alternate approach of comparable precision uses a point-counter stage of the type described by Chayes (22, 57). In the linear traverse method, the lengths of intercept of each constituent are measured on equally spaced traverses and the lengths for each constituent accumulated. In the point-count method, each traverse is broken down to a series of equally spaced points. The constituent found at each point in the grid is identified and recorded on a tabulating counter with one key assigned to each constituent. The accumulated totals show the number of times each constituent was encountered at an intersection of the grid. The principle of the point-count can be used in the determination of proportions of constituents in any heterogeneous solid; it is much quicker and less fatiguing to the operator than a micrometer stage, and fairly extensive tests of its precision on thin sections of granites have already been made (59, 60). The petrographers of the

Waterways Experiment Station (57) and Mr. Willis of the Missouri State Highway department (61) have found that determinations of air content of concrete by point count and linear traverse are comparable and comparably precise. So far it has not been adapted to determinations of bubble spacing.

CONCLUSION

From the days of Le Chatelier to the present, a number of ingenious and enterprising students have contributed ideas and information to the microscopy of concrete and its constituents. Much remains to be done in terms of observation and description of microstructure, relation of microstructure to larger structures, and detailed interpretation of the phenomena observed.

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DISCUSSION

A Member—How long does it take to make a particle constituent count by both methods?

Mrs. Katharine Mather (author)—It depends on the number of constituents that you want to deal with. For instance, if there are four constituents to be counted in a thin section of granite, an experienced operator using the point-count method ought to be able to cover one thin section, which is an area of about $\frac{3}{4}$ sq in., in 20 min. If for some reason you have a more complex system and a count of all the different constituents is desired, the time will increase somewhat. The time, using linear traverse techniques, would be two or three times as great.

*Mr. A. Borowik**—How can the data of microscopical examination be correlated with those of strength or durability of concrete?

Mrs. Mather—The factor which seems to make the greatest difference in durability of otherwise comparable concretes is air content. Probably the air content of the durable and nondurable concrete should be measured micrometrically.

* Metallurgist, Frankford Arsenal, Philadelphia, Pa. and Associate Professor, Villanova College, Villanova, Pa.

Strength might be indicated by freedom from microfractures, by denseness of texture, by absence of alteration and deterioration products, and by degrees of hydration of the cement—how much unhydrated cement there is left.

Of course, one cannot look at a thin section concrete and deduce the mixture design with accuracy. Usually some difference can be found between two concretes which will at least suggest an explanation of the differences in their behavior.

Mr. C.W. Mason **—How much is done in the study of the fractured surface of the concrete?

Mrs. Mather—The fractured surfaces of concrete are extremely useful in a number of way. They are useful for examination at low magnification to determine gross structure. They are useful when one is examining deteriorated concrete to locate deterioration products for subsequent examination with a polarizing microscope. I do not believe that anybody has done much work with high power—say over 60 X magnification of fractured surfaces.

** Professor of Chemical Microscopy, Cornell University, Ithaca N.Y.

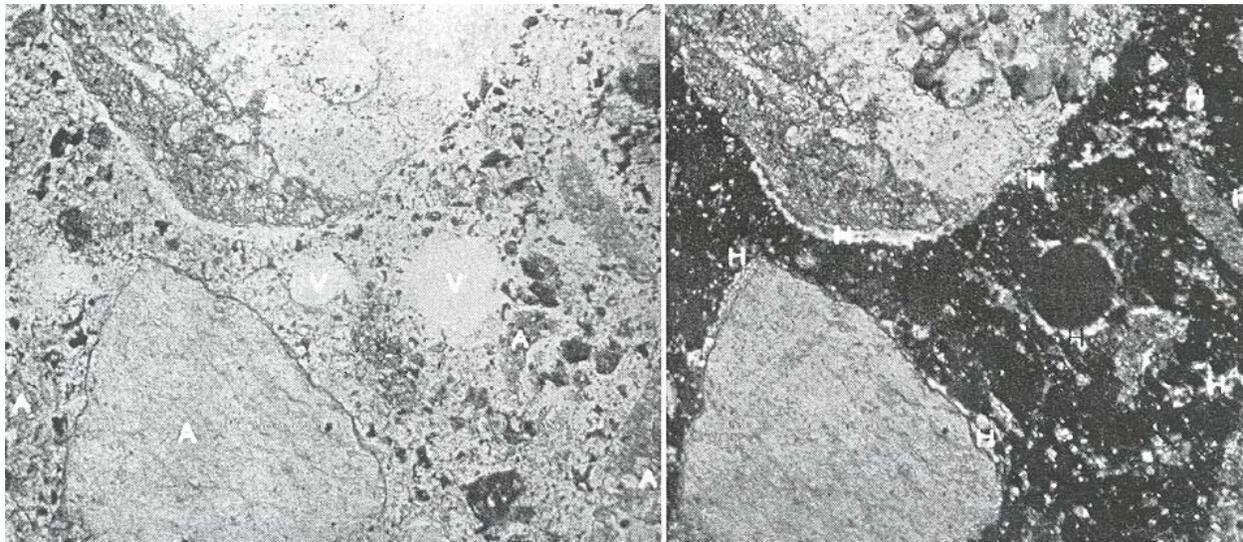
Table I—Data for Photomicrographs

Photographic Data: American Optical Co. 16 mm chromatic objective, 10 X Hyperplane ocular; 40_w Zirconarc lamp at normal setting, Wratten B Filter; Isopan film; exposure time ½ sec for plane light views, 1 ½ for crossed nicols. Magnification 165 X.

Concrete Data:

Dam	Water-Cement Ratio by Weight	Actual Cement Factor, bags per yd ³	Fine Aggregate, percent by Weight of Aggregate	Aggregate Finer than no 200 Sieve, percent of Fine Aggregate	Age of Concrete at Examination, months
A.....	0.51	3.9	26	4.8	8
B.....	0.59	3.8	26	8.1	19
C.....	0.54	3.9	24	3.2	11
D.....	0.56	3.8	26	3.2	5

Note—Type II portland cement was used in all four concretes. The cements came from different mills.

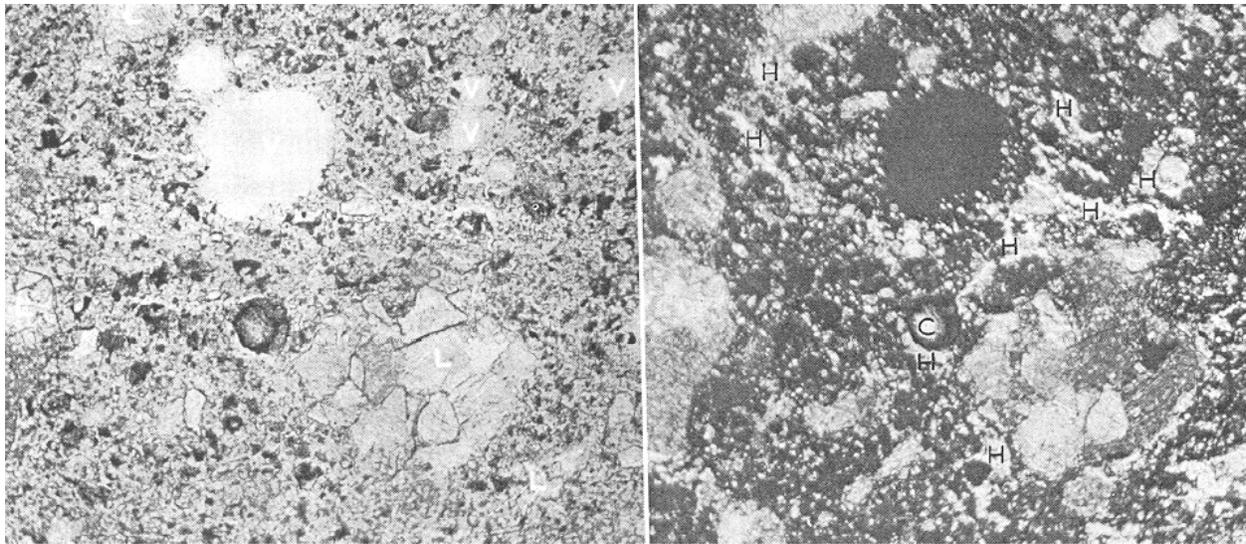


(a) Plane light.

(b) Crossed nicols

Fig. 1 — Mass Concrete Dam A (X165).

In (a) *V* marks voids, *A* aggregate; the abundant small dark irregular grains in the matrix are residual and unhydrated cement. In (b) the larger calcium hydroxide crystals are marked *H*. Hydroxide forms narrow borders on the two largest aggregate grains and an almost complete rim around the right void. The most irregular small light spots in the dark gel background are small hydroxide crystals and the minute light dots, limestone dust.

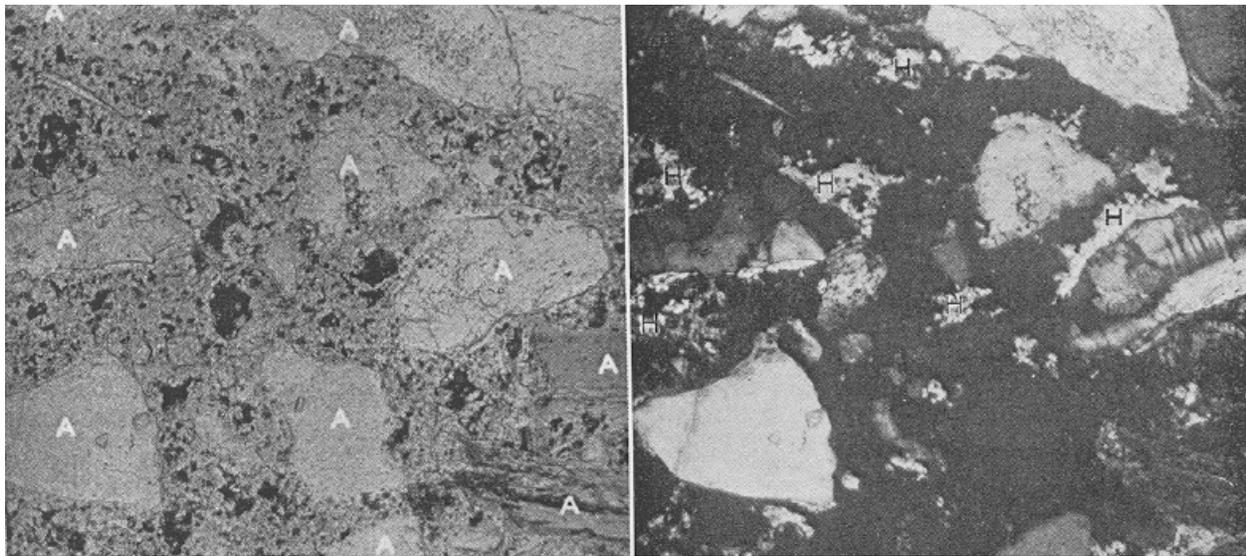


(a) Plane light.

(b) Crossed nicols.

Fig. 2 — Mass Concrete Dam B (X165).

In (a) *V* marks voids; *L*, limestone, and the dark rounded grains in the matrix are unhydrated cement. In (b) the larger hydroxide crystals are marked *H*, and one large unhydrated cement grain with a hydroxide rim, *C*. The dark gel groundmass is almost lost, obscured by the myriad of limestone dust particles and some small hydroxide crystals.



(a) Plane light.

(b) Crossed nicols.

Fig. 3 — Mass Concrete Dam C (X165).

Aggregate is marked *A* in (a) and hydroxide is marked *H* in (b). The crystals of hydroxide are large, irregular in shape, and widely separated in the dark gel background. Compare with Figs. 1 and 2 for the differences between concrete with siliceous aggregate and concrete with limestone aggregate.



(a) Plane light.

(b) Crossed nicols.

Fig. 4 — Mass Concrete Dam D (X165).

Voids are marked V and aggregate A, in (a). The clear hexagonal grain, C, is tricalcium silicate, two dicalcium silicate grains are labeled C in (b). The aggregate grain at the middle upper margin shows cross-hatched as well as lamellar plagioclase twinning, with crossed nicols. Hydroxide crystals are marked H in (b); the three largest near the center of the field surround or partly surround several unhydrated cement grains. The dark gel groundmass is almost free of minute hydroxide crystals and minute aggregate particles.