CONCRETE PETROGRAPHY – PAST, PRESENT, AND FUTURE

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Abstract

One hundred and twenty-three years ago, French chemist LeChatelier used polarized-light microscopes to study portland cement clinkers. In 1897, Törnebohm used microscopy to determine alite, belite, celite, felite, and an interstitial glassy phase in portland cement clinker. In 1915, Johnson used reflected-light microscopes to study ground and polished sections of concrete. Since the early 1940's, a galaxy of eminent petrographers from the U.S. Bureau of Reclamation have published a series of articles on applications of petrography in concrete and aggregate. In 1954, ASTM standard C 295 on petrography of concrete aggregates was published, which was followed by C 457 in 1960 on hardened air analysis, C 856 in 1977 on concrete petrography, and C 1324 in 2002 on mortar petrography. Bryant and Katharine Mather and R. C. Mielenz played key roles in drafting and developing the ASTM petrography standards on aggregates and concrete. In 1967, Gunner Idorn's doctoral thesis showed the importance of thin-section microscopy in failure investigation of concrete. In 1972, Romer and Dobrolubov introduced fluorescent-light microscopy, which was later advanced by H.N. Walker in the USA and notably by the Danish group of scientists. Since the early 1970's, the scanning electron microscope (SEM) has been used as an essential petrographic tool for materials characterization and failure investigation.

During the past two decades, significant developments have taken place on various techniques and instruments used in petrographic examinations of building materials. Examples include fluorescent-light microscopy and image analysis to determine the microstructure and water-cementitious materials ratio in concrete, rapid determination of air-void parameters in hardened concrete by image analysis, various hardware and software refinements in user-friendly SEM and in elemental analysis of concrete, examination of wet samples and cement hydration in environmental scanning electron microscopes (ESEM), and ultra-high-magnification examinations of cement hydration in transmission electron microscopes (TEM) with modifications for elemental and morphological examinations (AEM and STEM).

Fourier transform infrared microscopy, confocal microscopy, ESEM, cryo-SEM, microstructural modeling for predicting concrete properties, and x-ray microscopy are the areas that are either advancing or still in an experimental stage and expected to provide a variety of additional information. Despite the technological advancements, the basic petrographic methods applied in the past that are known to be effective for their intended purposes should continued to be used in the future.

A link between the research and routine applications of petrography in the cement, aggregate, and concrete industry is essential.

Keywords: **Petrography, Petrographer, Microscopy, Concrete, and Aggregate**

Introduction

Concrete is a continuously changing system – starting from its mixing to its placement. Even after hardening, concrete gains strength for long periods and interacts with the environment to which it is exposed. Strength, durability, and dimensional stability have long been recognized as the three fundamental parameters for a good concrete. Less attention, however, is paid to the microstructure of concrete, which is the detailed anatomy of concrete not only controlling these three parameters but also the overall behavior and performance of concrete. There have been incidences where concrete showed satisfactory project-specified strength and workability during the construction, but failed to provide similar satisfaction in performance or long-term durability. The question remains: Strength or Durability – Which is more important?

Actually, strength and durability are two sides of the same coin – the concrete microstructure. The microstructure of concrete is its entire world seen by using a variety of microscopes, which is indistinct in the unaided human eye. It is the microscopically magnified portion(s) of a macrostructure, which reveals the essential details and locates the source and extent of the problem, if any. Petrography is the science that provides detailed information on the microstructure of concrete and other construction materials.

Conventional Petrography and Concrete Petrography

The root of petrography dates back to the 19th century as a 150-year old science of geology, to the "good old days when men were naturalists and not geologists, and petrologists were unknown" (Johannson, 1938). Petrography (Gr. πέτρα, *petra*, “rock”, and γράφειν, *graphics*, “to write”) comprises the descriptive part of the science of rocks, petrology (Gr. πέτρα, *petra*, “rock”, and λόγοś, *logos*, “discourse”). It deals primarily with the microscopic features of rocks, their relations to the megascopic geological features, and with chemical, mineralogical, and textural compositions of rocks. Conventional petrography comprises the study of the chemical and mineralogical compositions of terrestrial (igneous, sedimentary and metamorphic) rocks, their texture or fabric (i.e., arrangements and interrelationships of minerals in space), classification, and their mutual relationship with the other rock types in the earth. Petrography has also been extended to the study of extraterrestrial rocks such as lunar rocks and meteorite samples.

Due to the increasing awareness on the importance of microstructure in controlling the mechanical properties and durability of concrete, since the late twentieth century, the science of petrography has became a centerpiece of concrete research and a routine method for investigating concrete deterioration. Concrete is a man-made rock, consisting essentially of aggregates dispersed in a hydrated cement paste. Aggregates are mostly natural rocks, either crushed or collected as gravel from rivers or as slag from volcanic rocks and then processed (i.e., washed and graded) for use in concrete. The paste is the main adhesive component and is the product of reactions between cement and water. Cements used in concrete are hydraulic cements, which indicates they not only harden by reacting with water, but also form a water-resistant product. Portland cement, the most commonly used type of hydraulic cement (calcium-silicate cement), is manufactured by high temperature (~1450°C) burning of a properly proportioned mixture of naturally occurring calcium carbonate (e.g., limestone) and clay-rich (e.g., shale) rocks in a cement kiln, and then grinding the pellet-like reaction products (clinkers) with gypsum, a set controlling component. Carbonate rocks provide calcium oxide, whereas the clay-rich rocks provide oxides of mainly silica with some alumina, iron, and alkalis. The reaction product is, therefore, principally calcium silicate with some minor phases of calcium aluminate and ferrite. The alumina, iron, and alkalis in clay provide a substantial beneficial reduction in the temperature of formation calcium silicates in the kiln. In high alumina cement concrete, the cementitious component is produced by high-temperature (~1450°C) calcining a mixture of bauxite and limestone that are low in silica. Blended cements are manufactured by mixing portland cement with other industrial waste products such as fly ash, blast furnace slag, or silica fume or with a natural pozzolan such as volcanic ash. The raw materials of other specialty cements are also the natural rocks that are processed, proportioned, and calcined in special ways.

Since all these basic ingredients of concrete come mostly from natural rocks either directly or after processing, and concrete itself is a man-made rock, petrography (the descriptive science of natural rocks) has a tremendous impetus in concrete technology. The wedding of petrography, first with cement, and then with concrete was inevitable, and has resulted an integrated science of cement and concrete petrography. The birth of this discipline dates back to the late-nineteenth century when LeChatelier studied cement clinkers under microscopes. It was the beginning of a new era for the application of petrography to the cementitious materials, outside the conventional realm of natural rocks. Since then, cement and concrete petrography has gone through various stages of development, some of which are listed in Table 1. This article is a review of the origin, evolution, present state, and the future of concrete petrography as well as the importance of petrography in the construction industry.

Table 1: Stages of development of the science of concrete petrography

Year	*Events*
1882	The first reported usage of the microscope to study Portland Cement - LeChatelier used polarizing-light microscopes to study cement clinkers and showed that tricalcium silicate was the principal constituent in Portland Cement.

1897	Tornebohm extended the use of polarizing-light microscopes in cement clinkers and identified various mineral constituents in clinkers - alite, belite, celite, and an apparent isotropic residual phase.
1915	By using reflected-light microscopes N. C. Johnson used ground and polished sections of concrete to study various deteriorations. His attempt to prepare thin sections of concrete showed loss of information during sample preparation (commercial epoxy resin, which is needed to hold the fabric of concrete during sectioning, was not available until 1946).
1934	Tavasci first showed that the main clinker phases in Portland Cement can be quantitatively distinguished in a polished section using etching techniques.
1935	R.J. Holden used petrography to determine that the first reported evidence of alkali-aggregate reaction in concrete in the United States (the expansion and cracking of concrete in the Buck hydroelectric plant on the New River, Virginia within 10 years of construction) was caused by a chemical reaction between the cement and the phyllite rock used as aggregates.
1935-36	Parker and Hurst (1935) and Brown and Carlson (1936) examined thin sections of hydrated cements with difficulty.
1941	Foundation of the Petrography Laboratory at the US Bureau of Reclamation that has subsequently published numerous pioneering papers on the applications of petrography in studying concrete and concrete aggregates.
1952	Katharine Mather's publication on the application of light microscopy in concrete research.
1954	Publication of ASTM C 295 "Standard Guide for Petrographic Examination of Aggregates for Concrete", which was drafted by Katharine and Bryant Mather.
1960	Publication of ASTM C 457 "Microscopical Determination of Air-Void Content and Parameters of the Air-Void System in Hardened Concrete".
1962	Publication of R.C. Mielenz's pioneering paper on "Petrography Applied to Portland-Cement Concrete," which is a classic article in concrete petrography.
1966	Katharine Mather's publication on "Petrographic Examination of Hardened Concrete" set the stage for future standardization of this science by the ASTM.
1967	Application of SEM in studying microstructure of hydrated cement paste by S. Chatterji and J.W, Jeffery (published in Nature v 209, p. 1233).
1972	Application of fluorescence microscopy in concrete by B. Romer and G. Dubrolubov
1977	Publication of ASTM C 856 "Standard Practice for Petrographic Examination of Hardened Concrete", which was drafted by Katharine and Bryant Mather.
1978	Establishment of the International Cement Microscopy Association (ICMA) - a leading organization devoted to the application of microscopy in cement and concrete.
1979	Application of fluorescent light microscopy in concrete by Christensen et al. and Walker and Marshall.
1986	Publication of D. H. Campbell's classic book on "Microscopical Examination and Interpretation of Portland Cement and Clinker"; also Dr. S. Diamond's classic paper on "The Microstructure of Cement Paste in Concrete" summarizes the application of SEM in studying concrete microstructure.
1990	Extension of fluorescent -reflected light microscopy in a polished concrete section by Mayfield.
1989	Dr. Scrivener's publication on "The Microstructure of Concrete" summarizes the importance of backscatter electron imaging in studying the stages of development and overall microstructure of hardened cement paste and concrete.
1990, 1994	ASTM symposiums on the application of petrography in cement, aggregates, and concretes; and subsequent publications of ASTM STP 1061 (1990), and ASTM STP 1215 (1994).
2002	Publication of ASTM C 1324 "Standard Test Method for Examination and Analysis of Hardened Masonry Mortar". The standard was drafted by Bernard Erlin (on petrography) and William Hime (on chemical analysis).

Applications of petrography in the construction industry

Petrography has the following broad applications in the construction industry:

(a) Characterization, evaluation, and quality assurance of concrete and concrete-making materials (i.e. clinkers, cements, and aggregates);
(b) Diagnosing evidences of improper construction practices (e.g., improper proportioning, mixing, placement, finishing or curing operations) in hardened concrete that could lead to premature failure;
(c) In situ observation of materials, microstructural characteristics, and defects in a deteriorated concrete at a high magnification and resolution, where all concrete constituents preserve their original composition, texture and distribution;
(d) Identification, location, abundance and chemical composition of deleterious constituents and products or evidences of adverse reactions that are responsible for the concrete deterioration;
(e) Evaluation of the durability of concrete to various external deleterious chemical agents (acid, alkali, sulfate, chloride, seawater) and physical forces (frost, fire);
(f) Evaluation of repair materials, the depth of the deteriorated original material to be replaced, effectiveness of a repair strategy, and diagnosing failure of a repair scheme.

The major applications of petrography are in characterization, quality assurance, evaluation, and failure investigation of construction materials. Since proper diagnosis of a problem is the key to the formulation of a successful repair scheme, petrography is gaining a significant acceptance in the concrete repair industry (Jana, 2005). Table 2 shows various properties of concrete that can be determined from field investigation, chemical analysis, physical testing, non-destructive testing, and petrography. Although each method has its own merit and is commonly recommended for a specific purpose, the wealth of information obtained from petrography is overwhelming.

Table 2: Advantages of Petrographic Examination of Concrete (modified from ACI Committee Report 364.1R)

Properties of Hardened concrete	*Field Evaluations*	*Nondestructive testing*	*Physical Testing*	*Chemical Testing*	*Petrography*
Admixtures – Their compositions and various effects in concrete				•	•
Aggregate Quality				•	•
Air Content in hardened concrete					•
Alkali-aggregate reaction	•				•
Abrasion resistance of concrete floors			•		•
Carbonation					•
Cement Content				•	•
Chloride Content				•	
Coating-concrete-subgrade adhesion or bonding character	•	•			•

Consolidation	•	•			•
Compressive strength		•	•		
Corrosive contaminant				•	•
Contaminated materials				•	•
Cracking	•	•	•		•
Delaminations	•	•	•		•
Discoloration	•			•	•
Degree of cement hydration				•	•
Efflorescence, coating failure, discoloration	•			•	•
Fire attack					•
Frost attack					•
Freeze-thaw durability			•		•
Honeycomb; Popouts, Segregation, Bleeding	•				•
Mixing evaluation					•
Mixture proportions					•
Mortar composition and quality evaluation				•	•
Paste quality and bonding					•
Permeability			•		•
Rebar corrosion	•	•		•	•
Soundness		•		•	•
Scaling, spalling, dusting, and other surface defects	•	•			•
Surface hardness		•			
Sulfate resistance			•	•	•
Uniformity	•	•			•
Water/cement ratio				•	•

The following paragraphs list applications of petrography in various construction materials.

Portland cement concrete and mortar

In portland cement concrete and mortar, petrography has several significant applications:

(a) Petrography determines the type of the aggregates used – the lithology, size, shape, grading, distribution, potential alkali-aggregate reactivity, unsoundness, deleterious constituents, cracking, alterations, reaction rims, coatings, overall quality, and the effects of aggregates in various fresh and hardened concrete properties.

(b) The physical properties (color, hardness, density, porosity, water absorption), textural properties (variation of color, hardness, porosities, microstructural arrangements of crystalline products) and compositional/mineralogical properties of paste including the cement hydration products.

(c) The air-void system of concrete including the volume (air content), fineness (specific surface), closeness (void-spacing factor), distribution (profile) of air voids, and the volumetric proportions of aggregate, paste, and air.
(d) The type, location, composition, abundance, and significance of various secondary deposits in the concrete.
(e) The depth and pattern of various macro and microcracks in the concrete.
(f) The depth of carbonation and other atmospheric alterations in the concrete.
(g) The nature of the interfaces between the paste and other fillers such as aggregates, reinforcing steels, fibers, etc. and the role of these interfaces in improving or degrading the overall strength and quality of the concrete.
(h) Investigation of various concrete surface distresses such as scaling, spalling, cracking, corrosion, discoloration, delamination, blistering, debonding, etc.
(i) Evidence and extent of various external chemical attacks in concrete from the exposure to acidic or sulfate environments.
(j) Evidence and extent of fire attacks in concrete.
(k) Evidence of freezing and frost attack in concrete in plastic or hardened state;
(l) Concrete deterioration due to reversible salt hydration reactions and salt crystallization pressures.
(m) The presence of various pozzolanic and cementitious materials and their effects in improving the overall microstructure (and thereby the strength and durability) of concrete.
(n) The water-cementitious materials ratio and cementitious materials contents in the concrete and any deviations of these proportions from the mix design.
(o) Reasons for improper setting of concrete (accelerated or delayed setting compared to the expected rate).
(p) Reasons for lower-than the designed strength of concrete.

Based on all the above information, petrography evaluates the overall quality of concrete and diagnoses the causes of improper performance, premature failure, evidence of expansion or shrinkage and cracking due to restrained volume instability, and long-term deterioration of concrete.

Other portland cement products

Petrography is also used in quality assurance, microstructural examinations, and failure investigation of other portland cement based products including:

(a) Specialty concretes such as high strength concrete, high performance concrete, lightweight concrete, fiber-reinforced concrete, and polymer-modified concrete.

(b) Portland cement plasters (stucco) – the thickness and composition of the base (scratch, brown) and finish coats, the causes of cracking, spalling, corrosion of metal lath and other deterioration in stucco.
(c) Composition, microstructure, and causes of deterioration of shotcrete.
(d) Various portland cement based proprietary grouting materials marketed for rapid setting, self leveling, shrinkage compensating, and high early strength; repair materials for patching or anchoring; and reasons for their sometime uncontrolled expansion and cracking at the hardened state.
(e) Concrete pipes and the depth of deterioration in the pipes from the external (acid, chloride, carbon dioxide from the atmosphere, or soil in underground prestressed concrete cylinder pipes) or internal environments (from sewage materials in sewer pipes such as hydrogen sulfide gas, chloride, sulfate, and other corrosive agents).
(f) Geothermal grouting materials, cement-based backfill materials and their potential for expansion.
(g) Various other project-specific applications.

Non-portland cementitious materials products

Petrography has significant applications in various non-portland cementitious materials products such as:
(a) Mortars made using hydrated calcitic or dolomitic lime, hydraulic lime, lime putty, or gypsum.
(b) Potential unsoundness of free lime and magnesia.
(c) Gypsum-based plaster, wallboard, and gypsum-based grouts.
(d) Various pozzolanic and cementitious materials such as fly ash silica fume, slag, metakaoline, and natural pozzolans used in combination with portland cement.

Masonry

In the masonry industry, petrography has the following applications:
(a) Petrography determines the overall quality, various physical properties (color, density, freeze-thaw durability, porosity), microstructure, and composition of clay, stone, and concrete masonry units.
(b) Petrography determines the type and composition of jointing mortar.
(c) Petrography diagnoses the evidences of masonry deterioration and mortar failure such as water penetration, cracking, premature stiffening of mortar, over-sanding, and improper combinations of masonry units and mortars.
(d) Petrography determines the type and composition of the original mortar used in historic structures, the depth of replacement of the original mortar needed in a repointing project, and selection of a suitable modern mortar compatible in color, texture, and composition to the ancient mortar.

(e) The nature of the bond between masonry units and mortar can be examined by visual and microscopical examinations.
(f) Physical properties (color, luster, hardness, porosity), mineralogy, lithology, and causes of failures of dimension stones are best assessed by petrography.
(g) Type and composition of architectural cast stones and natural stones; causes of stone failure, and freeze-thaw durability are also evaluated by petrography.

Floor covering

In the flooring (e.g., tile, terrazzo) industry petrography has several applications such as:

(a) Overall quality, and possible reasons for failure or unsatisfactory performances of terrazzo, ceramic tile, quarry tile, vinyl tile, or stone tile.
(b) Failure due to moisture migration and entrapment or moisture condensation.
(c) Composition of setting bed mortars and jointing grouts and their potential unsoundness in causing tile deterioration.
(d) Diagnosing evidences for tile failures due to debonding, cracking, discoloration, efflorescence, and blistering.
(e) Cracking of tile or other floor covering due to moisture-related expansion of the setting bed mortar or jointing grout.
(f) Investigation of floor covering failure due to improper surface preparation, improper preparation of the setting bed, adhesive, and/or the covering material, or improper applications of the coverings.

Methods used in concrete petrography

Detailed descriptions of sampling, various approaches, techniques, and methods used in concrete petrography are beyond the scope of this article and can be found elsewhere (St. John et al., 1998; French, 1994; Mielenz, 1962; Mather, 1952; Walker, 1992; ASTM C 295, C 457, C 823, C 856, and C 1324). Following is a brief description of various approaches, techniques, and methods used in the author's laboratory during petrographic examinations:

(a) Field reconnaissance and photographic documentation of the concrete structure to get a first-hand impression of the concern that initiates subsequent petrographic examination.
(b) Detailed visual examinations of the samples in the laboratory; photographic documentation; a detailed description of the nature of the sample, its dimension, weight, color, texture, integrity, exposed surfaces, embedded items, visual distress such as cracking, discoloration (if any), corrosion, and any other relevant features possible to note.
(c) Sample preparation for microscopical examinations, including sectioning, lapping or fine grinding, thin sectioning, polishing, oil immersion mount preparation, coating, staining, and etching for specific purposes.

(d) Examinations of oil immersion mounts and thin sections in a petrographic microscope.
(e) Examinations of fluorescence dye-mixed epoxy impregnated thin sections of concrete in a reflected and/or transmitted fluorescent-light microscope.
(f) Examinations of an “as received”, saw-cut, freshly fractured, ground, polished, stained, or etched sections in reflected light-microscopes (in a stereomicroscope at a magnification up to 70X or in a metallurgical microscope at a magnification of up to 1000X).
(g) Examination of the air-void system of the concrete in a stereomicroscope by the conventional modified point count or linear traverse methods of ASTM C 457, or by the automated image analysis procedure.
(h) Photomicrographic documentation of various features observed in light optical microscopes by digital cameras and image analysis softwares.
(i) Examinations of an “as received” sample, saw-cut surface, freshly fractured section, polished section, or polished thin-section in a scanning electron microscope that is equipped with a backscatter electron and secondary electron detectors and an energy-dispersive x-ray spectrometer (SEM-EDS). Such examinations reveal the morphology, microstructure, and chemical composition of concrete and its components at high magnifications.
(j) X-ray diffraction analysis for rapid identifications, qualitative and quantitative determinations of various crystalline materials.
(k) Supplemental chemical analysis (e.g., cements, bulk oxide, chloride, sulfate, organic analyses) and other physical tests (e.g., absorption, density, strength, permeability) apart from microscopical examinations that are needed for the detailed investigation.

Concrete Petrography: The Glorious Past

As mentioned before, the use of petrography in cement and concrete dates back to the late-nineteenth century, when LeChatelier first studied clinker particles in a polarized-light microscope and determined that tricalcium silicate is the principal constituent of portland cement. In 1824, Joseph Aspdin patented portland cement. In 1887, LeChatelier identified the following components in portland cement – abundant, clear, colorless tricalcium silicate; turbid yellowish crystals of dicalcium silicate; a dark brown interstitial calcium alumino-ferrite; and another material that he inferred as tricalcium aluminate. In 1897, Törnebohm, a Swedish investigator, extended the use of polarizing-light microscopes in cement clinkers and identified various mineral constituents in clinkers - alite, belite, celite, felite, and an apparently isotropic (glassy) residual phase. In 1903-1905, Richardson demonstrated the use of a polarized-light microscope in the prediction of cement quality from clinker examinations. In 1915, Rankin and Wright established the optical properties of principal phases in commercial cements. In 1928, Guttmann and Gille tabulated the basic optical properties of clinker phases and the common hydration products. In 1934, Tavasci first showed that the main clinker phases in portland cement can be quantitatively distinguished in polished section using etching techniques. In 1936, Insley showed that alite is C_3S, belite and felite are two different habits of C_2S, and celite is C_4AF. Insley and McMurdie (1938) described the later developments in etching techniques and the use of polished thin sections in clinker examinations. Since 1940's, Levi S. Brown at the Portland Cement Association provided significant contributions on the application of microscopy in examination of clinker, cement, and concrete. The early and later studies on clinker microscopy were reviewed by Mather (1952) and Campbell (1986), respectively.

In 1915, Johnson studied ground and polished sections of concrete in reflected light microscopes to study various deteriorations. His attempt to prepare thin sections of concrete showed loss of information during sample preparation (commercial epoxy resin which is needed to hold the fabric of concrete during sectioning was not available until 1946). Parker and Hurst (1935) and Brown and Carlson (1936) studied thin-sections of hydrated cement pastes with difficulty. With the advent of commercial epoxy resin that was necessary to improve the overall integrity of concrete, thin sectioning of concrete was possible; and it set the stage for the subsequent rapid development of concrete petrography.

Petrographers at the US Bureau of Reclamation – Since the early twentieth century a galaxy of notable authorities starting from W.Y. Holland, R.C. Mielenz, Duncan McConnell and many other petrographers from the petrography laboratory of the U.S. Bureau of Reclamation have published a series of pioneering papers on the techniques and applications of petrography in evaluating sulfate soundness and alkali-silica reactivity of aggregates, concrete, alkali-aggregate reactions in concrete, air-void system in concrete, freeze-thaw deterioration of aggregates and concrete, and pozzolanic and cementitious materials in concrete. Since 1934, petrography has been used for routine examination of concrete aggregates. Petrographic examinations received its biggest impetus in the early 1940's immediately after the discovery of alkali-aggregate reactions by Stanton Walker. In 1941, a petrographic laboratory was formed including Roger Rhoades, Duncan McConnell, Richard Mielenz, Bill Holland, Robert Wilson and Herbert Winchester. A series of publications appeared from the US Bureau of Reclamation on petrography applied to aggregates and concrete with reference to alkali-aggregate reactivity (see DePuy, 1990 for a review).

Although it is impossible to acknowledge the contributions of so many notable petrographers in a single article, following is a list of the individuals whose publications and contributions have greatly inspired the present author.

Richard Mielenz – The present state of concrete petrography owes much to the efforts and publications of Dr. Richard Mielenz who has been actively involved in petrography of aggregates and concrete. Since 1940s, Dr. Mielenz has published a series of pioneering articles. His works and publications involved applications of petrography in detailed examinations of concrete aggregates, alkali-silica reactions, concrete deteriorations from various external chemical and physical agents, chemical reactivity of aggregates, petrographic techniques, origin and measurements of air-void systems in air-entrained concrete, and pozzolanic and cementitious materials (see DePuy 1990 for a review of list of publications).

Bryant and Katharine Mather – The late husband and wife team made many significant contributions in the application of petrography for studying various aspects of concrete and aggregates. The ASTM C 295 and C 856 procedures for petrographic examinations of aggregates and hardened concrete, respectively, were originally drafted by Katharine and Bryant Mather. A recent commemorative publication by ACI combines their many significant contributions in a book entitled "Investigating Concrete: Selected Works of Bryant and Katharine Mather".

Bernard Erlin – Since the late 1950s, Mr. Erlin has played a leading role in popularizing the application of petrography in the construction industry in the USA. Prior to starting his own petrographic services, the author was fortunate to work closely with Mr. Erlin for five years and jointly publish several articles. Mr. Erlin is one of the world's first full-time, hands-on concrete petrographers.

Gunner Idorn and other petrographers in Denmark – In Europe, Dr. Gunner Idorn, a notable authority in concrete petrography, has popularized the usefulness of thin-section petrography, and stressed the importance of petrography in failure investigation of building materials. Some eminent petrographers who either directly worked with Dr. Idorn or in the Danish Technological Institute or similarly contributed significant useful research in concrete petrography are S. Chatterji, A. D. Jensen, P. Christensen, Niels Thawlow, and U. H. Jakobsen.

Parallel to the petrographic publications of the US Bureau of Reclamations, petrographers at the research and development laboratories of the Portland Cement Association and its subsidiary Construction Technology Laboratories (L.S. Brown, Bernard Erlin, H. Hadley, Donald H. Campbell, and David Stark) published many important books and articles. Mr. Stark did pioneering research on the application of petrography in various concrete deterioration processes specifically, alkali-aggregate reaction and sulfate attack in concrete. In 1986, Dr. Campbell published a classic book on the application of microscopy in cements and clinkers.

D. A. St. John and other petrographers in New Zealand have done significant petrographic works on: alkali-aggregate reactions in concerts structures in New Zealand, concrete sewer and stormwater pipe deterioration, large area thin section production, fluorescent microscopy, geothermal cement grouts, DSP mortars, and unsoundness of concrete due to the presence of argillite aggregates. In 1998, D.A. St. John, A. B. Poole, and Ian Sims published the first full-length textbook on concrete petrography.

The author has tried to mention the names of petrographers who have personally or through their pioneering publications contributed significantly to the development of his career in concrete petrography. While mentioning these names the author gratefully acknowledges many other practicing petrographers in various testing and consulting firms both in the USA and abroad whose contributions are not possible to list due to the limited space. All should be acknowledged for providing a better understanding of the behavior and properties of concrete through this fascinating science of geology.

Concrete Petrography: The Exciting Present – Standards, References, and Conferences

The increasing importance of petrography in the concrete industry has resulted in the appearance of a number of standardized test methods, conferences, organizations, and publications in the last 50 years. Following is a list showing standardization and increasing acceptance of this science in the construction industry:

(a) The ASTM International provides the following four standards for microscopical examinations of aggregate, hardened concrete, and mortar (the year of original publication is given in parentheses): (1) ASTM C 295: "Standard Guide for Petrographic Examination of Aggregates for Concrete" (1954); (2) ASTM C 457: "Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete" (1960), (3) ASTM C 856: "Standard Practice for Petrographic Examination of Hardened Concrete" (1977); and (d) ASTM C 1324: "Standard Test Method for Examination and Analysis of Hardened Masonry Mortar" (2002).

(b) The American Society of Testing and Materials has conducted two symposiums on petrography applied to cement, aggregates and concrete, which have resulted in publications of ASTM STP 1061 on "Petrography Applied to Concrete and Concrete Aggregates" and ASTM STP 1215 on "Petrography of Cementitious Materials".

(c) British Standards provide various test methods: BSI 812 (1994) "Method for qualitative and quantitative petrographic examination of aggregates"; BSI 1881 (1988) "Testing Concrete" Part 124, "Methods for Analysis of Hardened Concrete".

(d) A number of Nordtest methods appeared on the applications of microscopy in examinations of hardened concrete, such as NT Build 381 on "Concrete, Hardened: Air void structure and air content" (1991), and NT Build 361 on "Concrete, Hardened: Water-cement ratio" (1991).

(e) In 1978, the International Cement Microscopy Association (ICMA) was founded in the United States, and since 1981 the annual Proceedings of ICMA directly focus on the application of microscopy in the study of clinker, cement and concrete. So far, 26 proceedings have been published from these conferences that contain a wealth of research on applications of various forms of microscopy in investigation of clinker, cement, concrete and other building materials.

(f) Since 1987, nine proceedings have appeared from the Euroseminar on "Microscopy Applied to Building Materials". Similar to the ICMA in the USA, these proceeding are mostly European experiences on the applications of microscopy in cement, concrete, masonry and other building materials.

(g) Proceedings of the International Congress on the Chemistry of Cement and Alkali-Aggregate Reactions include voluminous publications on the application of petrography on various aspects of cement and concrete. The "Cement and Concrete Research" and "Cement and Concrete Composites" journals, both published by the Elsevier are two excellent sources that have many articles on the application of petrography in cement, aggregate, and concrete. "Cement, Aggregate, and Concrete" – a bimonthly publication of ASTM is also a good journal that has many articles on various aspects of aggregate and concrete petrography.

(h) A number of excellent papers and textbooks appeared on the application of microscopy in cement clinker: Microscopy of ceramics and cements by Insley and Frechette (1955); Gille et al., 1965; Hofmanner, 1973, Fundal, 1980, and "The microscopy of unhydrated portland cement" by Barnes and Ghose, 1983; and Campbell, 1986.

(i) Three excellent textbooks on the application of petrography in concrete and aggregates have been published by Dolar-Mantuani (1983), Walker (1992), and St John et al. (1998).

(j) In 2000, the Society of Concrete Petrographers was founded in the USA for a platform to exchange knowledge, ideas, and experiences among fellow practicing petrographers.

(k) Some key papers published on concrete petrography are by: Johnson in 1915 on "The microstructure of concrete"; K. Mather in 1952 on "Applications of light microscopy in concrete research" and in 1956 on "Petrographic Examination of hardened concrete"; L. S. Brown in 1959 on "Petrography of cement and concrete"; Mielenz in 1962 on "Petrography applied to portland-cement concrete", and "Diagnosing concrete failures"; Mielenz in 1994 on "Petrographic Evaluation of Concrete Aggregates"; French in 1990 on "Concrete Petrography – A review", Idorn in 1967 on "Durability of Concrete Structures in Denmark", Erlin in 1962 on "Methods used in petrographic studies of concrete"; J. A. Ray in 1977 on "Things petrographic examination can and cannot do with concrete"; Jensen et al. in 1985 on "Petrographic analysis of concrete"; Jana and Cole in 1997 on "Microscopy – A practical solution to concrete problems"; Jakobsen et al. in 1997 on "Optical Microscopy – A primary tool in concrete examination"; Thaulow and Jakobsen in 1997 on "Deterioration of concrete diagnosed by optical microscopy". Out of more than 250 publications on petrography applied to various case studies on concrete, the author chose the above-mentioned articles as the key articles that discuss general overall applications of petrography in characterization and failure investigation of concrete.

Some recent advances in concrete petrography in the last 25 years

Following are different areas in concrete petrography where the author has seen notable developments in the last 25 years:

(a) Fluorescent microscopy – Ever since the first application of fluorescence microscopy by B. Romer and G. Dubrolubov in 1972, and then by H. N. Walker and B. F. Marshall in 1979, this technique has came a long way and recently gained a significant impetus by extensive works of the Danish group of scientists, notably by N. Thaulow and U. H. Jakobsen. The last group of scientists used this technique in determining water-cement ratio in hardened concrete and studying microstructures of deteriorated concretes.

(b) Air-void analysis in hardened concrete by automated image analysis – In 1977, S. Chatterji and R. Gudmundsson, R. published an article on measuring air void parameters in hardened concrete by image analysis. Since then, a number of publications have appeared on automated analysis of air-void parameters in hardened concrete by computer-assisted image analysis software (e.g., Laurencot, J.L., 1991; Cahill et al., 1994; Pade et al., 2002; and Zhang et al., 2005). All this research provides a means for more accelerated methods of measuring air void parameters in hardened concrete than the conventional methods of ASTM C 457. The lapped sections of concretes are treated with black ink followed by zinc oxide paste to highlight the air voids, which are then captured to a computer by a video or digital camera and an image capture board, and then processed by a set of software algorithms to determine the air void parameters. Due to the ease of the operation, good accuracy and precision, and good correlation to the results obtained from the ASTM C 457 methods, the automated image analysis method has a significant potential for routine commercial use and even standardization.

(c) Quantitative petrography – Similar to the application of image analysis in determining water-cementitious materials ratios and air contents, several studies have been done on quantitative determination of mix proportions in hardened concrete including the amounts of portland cement, fly ash, and slag. Quantitative petrography, however, is still in an experimental stage and not a standard method. On a concrete of unknown origin and composition, it should be used with caution.

(d) Electron microscopy – Advancements in the field of electron microscopy include: (a) hardware and software refinements for overall user-friendliness in imaging and elemental analysis, (b) analysis of alkali-silica gel and other secondary deposits in concrete; (c) analysis of concrete deterioration by chemical attacks; (d) use of backscatter electron imaging for determining the microstructure of concrete and for estimating the water-cement ratio; (e) stages of development of the concrete microstructure during the hydration of portland cement; (f) effects of various mineral and chemical admixtures in improving or modifying the basic concrete microstructure; (g) examination of wet samples and hydration process in environmental scanning electron microscopes (ESEM); and (h) ultra-high-magnification examinations of cement hydration in a transmission electron microscope (TEM) with modifications for elemental and morphological examinations (AEM and STEM). SEM-EDS is in the process of standardization by ASTM.

(e) Rapid identification of ASR gel by uranyl acetate treatment – The method originally proposed by the Cornell Group of researchers (K. C. Hover and K. Natesaiyer) is included as an annex to ASTM C 856, which involved treatment of a freshly fractured surface of an ASR-suspected concrete sample with uranyl acetate solution and examination of gel fluorescence in a short wavelength UV light.

(f) Modeling of concrete microstructure – Since the early 1990's, Dale P. Bentz and other scientists at the National Institute of Standards and Technology (NIST) have been studying predicting the properties and behavior of concrete from various computer modeling of concrete microstructure. Their recent virtual testing of cement and concrete as a tool for predicting cement and concrete properties and concrete durability inputs microstructural data from SEM-BSE-EDS (Garboczi et al., 2004). This modeling approach has the potential for some future predictions, although it should be done with caution in the exceedingly complex natural environment of concrete.

Qualification of concrete petrographers

A concrete petrographer, as the name implies, should have sound knowledge and understanding in both petrography and concrete technology. The person should have:

(a) Adequate background in geological sciences with coursework and training in crystallography and various branches of mineralogy (optical, descriptive, physical and chemical mineralogy), x-ray diffraction, igneous, sedimentary and metamorphic petrology, petrography, and geochemistry, and

(b) Thorough understanding of the behavior and properties of concrete and concrete-making materials, various construction practices followed, effects of various ingredients on the proprieties of fresh and hardened concrete, and the effects of various deleterious agents on the properties of concrete.

Certainly, a unique combination of expertise in both fields is necessary. Unfortunately, a petrographer from a geological science background has little or no college training in concrete and a civil engineer from an engineering department rarely gets training in petrography. Therefore, the person should come from the field of geology with adequate prior training in petrography and get subsequent first-hand training in concrete by reading books and journals on concrete, supervision from experienced concrete technologists, and first-hand experience on as many aspects of concrete as possible (from the preparation of mix design to various construction procedures to various types of field deterioration of concrete). Since the quality of a professional report on concrete petrography depends on the interpretation of the information obtained from such examination, experience and a thorough understanding of the materials and properties of concrete are two essential requirements of a good concrete petrographer. The quality of the petrographic report also depends on several other factors including (but not limited to) the depth of knowledge of the person in petrography and concrete, the depth of examination, the details of communication between the petrographer and the person or engineer interested in doing such an examination about the background information of the project, and experience in interpreting the wealth of data obtained from petrographic examination.

Concrete Petrography: The Promising Future

Where can we go from here? Are there any new petrographic techniques available for examining concrete? Although the common petrographic methods have a long history and proven benefits, the future will surely bring many new techniques to view concrete with a different perspective, not only with the ease and accelerated methods, but also with the minute details of the development of concrete microstructure during the cement hydration. Although optical microscopy remains at the nucleus of petrography with scanning electron microscopy as its essential companion, exploring the possibilities and usefulness of other microscopy tools in the study of concrete has recently begun.

Studying cement hydration in wet-cell electron microscopes, such as in a low vacuum SEM or environmental SEM, is an area where research has been ongoing for the last 20 years to observe microstructural changes during cement hydration mostly at the surface region. Fourier transform infrared microscopy, confocal microscopy, ESEM, and cryo-SEM are the techniques and methods that are either advancing or still in an experimental stage and can be extended to provide a variety of additional analyses in different conditions of materials.

From light and electron, the techniques of microscopy have recently been extended to x-ray microscopy in studying cement hydration (Garci Juenger, 2004). Soft x-ray transmission microscopy and computed axial tomography (CAT or CT scanning) are techniques used routinely use in biological science for wet cell examinations and in medical science for imaging the human body, respectively. The former method is gaining acceptance for studying cement hydration in its natural wet state and the effects of various mineral and chemical admixtures on hydration and microstructural developments. The industrial CT scanner has applications in detecting reinforcing steel in concrete, characterization of aggregates, sulfate attack in concrete, and investigation of hydration of calcium aluminate cement.

Other techniques that have the potential for future applications in cement and concrete research include: (a) auger electron spectroscopy (for analyzing the surface composition of a microscopical region as small as 1 μm in diameter and nanometers deep); (b) scanning tunnel microscopy and atomic force microscopy for observation of surface structure of clinker minerals, hydrates, and adsorbed layer of organic admixture on the surface of cement particle; and (c) x-ray absorption fine structure analysis (for analysis of alkali-silica reaction gel in concrete). Details of these techniques, their applications, and various other techniques (e.g., NMR study of ASR gel, chromatography, mass spectrometry, secondary ion mass spectroscopy, mossbauer spectrometry, and thermoluminescence analysis) that have so far limited applications in cement and concrete research are summarized in an article by Hiroshi Uchikawa in a good book entitled "Handbook of Analytical Techniques in Concrete Science and Technology" by V. S. Ramachandran and J. J. Beaudoin.

Despite the technological advancements, the basic techniques of petrography that have been used for more than 100 years and are proven to be so effective in providing a wealth of information about concrete should be continued. For example, oil immersion mount and thin-section microscopy are two extremely important techniques of petrography (these are the most common and oldest methods in the conventional petrography). They should never be completely substituted by any new method. The fluorescent-light modification of the original thin-section microscopy is a step forward for highlighting many features and for quantitative determinations of paste porosities. Similar modifications in other methods including SEM-EDS would be beneficial.

In many cases, a synergetic application of petrography and chemical analysis is essential. To many individuals, even including some practicing petrographers, petrography represents microscopical examinations of materials. The conventional petrography, however, includes not only microscopy, but also chemical analysis of rocks needed for classification (e.g., silica saturation index and other chemical classification of igneous rocks). Microscopy, however, stays at the nucleus of petrography). During failure investigation of concrete, a synergetic approach of petrography and chemical analysis is sometimes helpful for detailed examination.

Conclusion - A link is needed between the research and practice in concrete petrography

Despite the significant research and advancements in techniques and methodology within the realm of concrete petrography, more advancement is needed in the general awareness among engineers, architects, and other construction consultants about the importance of this 150-year-old science of geology in routine materials characterization, quality assurance, and especially failure investigation of building materials. Lectures, conferences, proceedings, and seminars similar to this Euroseminar are helpful not only to improve our understanding on various applications of concrete petrography but also to increase the overall awareness among the engineers and architects about the usefulness of this science. Perhaps even a better way to achieve that would be through the hard and diligent work of a petrographer in successful diagnosis of a concrete problem or concern.

References

Ahmed, W.U. (1994): Petrographic methods for analysis of cement clinkers and concrete microstructure, In: DeHayes, S.M., and Stark, D. (eds.), Petrography of Cementitious Materials, Philadelphia, USA, ASTM STP 1215: 1-12.

ASTM C 295 (1998): Standard Guide for Petrographic Examination of Aggregates for Concrete, In: ASTM Vol. 4.02 (Concrete and Aggregates), Philadelphia, USA, American Society for Testing and Materials.

ASTM C 457 (1998): Standard Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete, In: ASTM Vol. 4.02 (Concrete and Aggregates), Philadelphia, USA, American Society for Testing and Materials.

ASTM C 823 (2000): Standard Practice for Examination and Sampling of Hardened Concrete in Constructions In: ASTM Vol. 4.02 (Concrete and Aggregates), Philadelphia, USA, American Society for Testing and Materials.

ASTM C 856 (2004): Standard Practice for Petrographic Examination of Hardened Concrete, In: ASTM Vol. 4.02 (Concrete and Aggregates), Philadelphia, USA, American Society for Testing and Materials.

ASTM C 1324 (2004): Standard Test Method for Examination and Analysis of Hardened Masonry Mortar, In: ASTM International's Masonry Standards for the Building Industry, Fifth Edition, Philadelphia, USA, American Society for Testing and Materials.

Barnes, P., and Ghosh, A. (1983): The microscopy of unhydrated portland cement, In: P. Barnes (ed.), Structure and Performance of Cements, London , Applied Science Publications: 139-203.

Brown, L.S. (1959): Petrography of Cement and Concrete, Journal of the PCA Research and Development Laboratories, 1 (3): 23-34.

Brown, L.S., and Carlson, R.W. (1936): Petrographic studies of hydrated cements, Proceedings of the American Society of Testing Materials, part II, 36: 332-350.

BSI 1881 (1988): Testing concrete Part 124, Methods for analysis of hardened concrete.

BSI 812 (1994): Method for qualitative and quantitative petrographic examination of aggregates, Part 104.

Cahill, J., Dolan, J.C., and Inward, P.W. (1994): The identification and measurement of entrained air in concrete using image analysis, In: Sharon DeHayes and David Stark, (eds.), Petrography of Cementitious Materials, ASTM STP 1215, American Society for Testing and Materials: 111-124.

Campbell, D.H. (1986): Microscopical examination and interpretation of portland cement and clinker, Construction Technology Laboratories, Skokie, Illinois, USA, Portland Cement Association.

Chatterji, S., and Gudmundsson, R. (1977): Characterization of entrained air bubble systems in concretes by means of an image analyzing microscope, Cement and Concrete Research, Vol. 7, 423-428.

DeHayes, S and Stark, D. (1994) Petrography of Cementitious Materials, ASTM STP 1215, Philadelphia, USA, American Society for Testing and Materials.

DePuy, G. W. (1990): Petrographic investigations of concrete and concrete aggregates at the Bureau of Reclamation, In: Erlin B., and Stark, D. (eds.), Petrography Applied to Concrete and Concrete Aggregates, ASTM STP-1061, Philadelphia, American Society for Testing and Materials: 32-46.

Diamond, S. (1986): The microstructure of cement paste in concrete, 8th International Congress on the Chemistry of Cement, Rio de Janeiro 1: 123-147.

Dolar-Mantuani, L. (1983): Handbook of concrete aggregates – a petrographic and technological evaluation., New Jersey, USA, Noyes Publications.

Erlin, B. (1965): Methods used in petrographic studies of concrete, Analytical Techniques for Hydraulic Cements and Concrete, ASTM STP 395: 3-17.

Erlin, B., and Stark, D. (1990) Petrography Applied to Concrete and Concrete Aggregates, ASTM STP 1061, Philadelphia, USA, American Society for Testing and Materials.

French, W.J. (1991): Concrete Petrography: A Review, Quarterly Journal of Engineering Geology, 24 (1): 17-48.

Garboczi, E.J., Bullard, J.W., and Bentz D.P. (2004): Virtual Testing of Cement and Concrete – USA 2004, Concrete International 26(12): 33-37.

Garci Jeunger, M. C. (2004): X-ray vision for cement-based materials, Concrete International, 26(12): 38-41.

Gille, F., Dreizler, I., Grade, K., Kramer, H., and Woermann, E. (1965): Microscopy of cement clinker – Picture atlas, Benton-Verlag GmbH, English translation by P. Schmid 1980, Dusseldorf, Germany.

Idorn, G.M. (1967): Durability of Concrete Structures in Denmark – a study of field behaviour and microscopic features, Technical University of Denmark, Copenhagen, Denmark.

Insley, H., and Frechette, van D. (1955): Microscopy of ceramics and cements including glasses, slags, and foundry sands, New York, USA, Academic Press.

Jakobsen, U.H., Johansen, V., and Thaulow, N. (1997): Optical Microscopy – A Primary Tool in Concrete Examination, Bulletin No. 65, Ramboll: 1-12.

Jana, D. (2005) Petrography and Concrete Repair – A Link is Needed, Concrete International, 27 (1): 37-39.

Jana, D., and Cole, A.A. (1997): Microscopy – A Practical Solution to Concrete Problems, Concrete Industry Board Bulletin, 33 (3): 18-22.

Jensen, A.D., Eriksen, K., Chatterjee, S., Thaulow, N., and Brandt, I. (1985): Petrographic analysis of concrete, Danish Building Export Council 12.

Johannsen, A. (1938): A Descriptive Petrography of Igneous Rocks, Vol. 1, University of Chicago Press.

Johnson, N.C. (1915): The microstructure of concretes, Proceedings of the American Society of Testing Materials 15, Part II: 171-213.

Laurencot, J.L., Pleau, R., and Pigeon, M. (1991): The microscopical determination of air voids characteristics in hardened concrete: Development of an automated system using image analysis techniques applied to micro-computers: 259-273.

LeChatelier, H. (1882): Comptes Rendues hebdomandaires des séances de l'academie des Sciences 94: 13.

LeChatelier, H. (1905): Experimental researches on constitution of hydraulic mortars (translated by J. L. Mack), New York, McGraw-Hill.

Mather, K. (1952): Applications of light microscopy in concrete research, Symposium on Light Microscopy, ASTM STP 143: 51-70.

Mather, K. (1966): Petrographic Examination of Hardened Concrete, In: Symposium on Significance of Tests and Properties of Concrete and Concrete-Making Materials, ASTM 169A, Am. Soc. Testing. Mats: 125-143.

Mielenz, R.C. (1962): Petrography Applied to Portland-Cement Concrete, In: T. Fluhr and R. F. Leggert (eds.), Reviews in Engineering Geology, The Geological Society of America, Vol. 1: 1-38.

Mielenz, R.C. (1964): Diagnosing Concrete Failures, Stanton Walker Lecture Series on the Materials Science, University of Maryland, College Park, Maryland, USA: 5-47.

Mielenz, R.C. (1994): Petrographic Evaluation of Concrete Aggregates, In: Klieger, P. and Lamond, J.F. (eds.), Significance of Tests and Properties of Concrete and Concrete-Making Materials, ASTM STP 169C, , American Society for Testing and Materials, Philadelphia, USA: 341-364.

Pade, C., Jakobsen, U.H., and Elsen, J. (2002): A new automatic analysis system for analyzing the air void system in hardened concrete, Proceedings of the ICMA, ed. Jany L. and Nisperos, A. San Diego: 204-213.

Ramachandran, V.S., and Beaudoin, J.J. (eds) (2001): Handbook of Analytical Techniques in Concrete Science and Technology – Principles, Techniques and Applications, New Jersey, USA, Noyes Publications.

Ray, J.A. (1983): Things petrographic examination can and cannot do with concrete, Proceedings of the 5th International Conference on Cement Microscopy: 66-85.

Scrivener, K.L. (1990): Microstructure of Concrete, In: Skalny, J. (ed.), Material Science of Concrete, American Ceramic Society.

St. John, D.A., Poole, A.W., and Sims, I. (1998): Concrete Petrography – A handbook of investigative techniques, London, Arnold.

Thaulow, N., and Jakobsen, U.J. (1997): Deterioration of concrete diagnosed by optical microscopy, Proceeding on 6th Euroseminar on Microscopy Applied to Building Materials, Iceland: 282-296.

Törnebohm, A.E. (1897): The petrography of portland cement, Tornindustrie Zeitung 21: 1148-1150, 1157-1159.

Walker, H.N. (1992): Petrographic Methods of Examining Hardened Concrete: A Petrographic Manual, Virginia Transportation Research Council, Richmond, Virginia, USA: 1-286.

Walker, H.N., and Marshall, B. F. (1979): Methods and equipment used in preparing and examining fluorescent ultrathin sections of portland cement concrete, Cement, Concrete, and Aggregates, 1(1): 3-9.

Zhang, Z., Ansari, F., and Vitillo, N. (2005): Automated determination of entrained air-void parameters in hardened concrete, ACI Materials Journal 102 (1), 42-48.