METHODS USED IN PETROGRAPHIC STUDIES OF CONCRETE

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KEY WORDS: chemical analysis, concrete, qualitative analysis, quantitative analysis, optical microscopes, electron microscopes, ultraviolet spectroscopy, infrared spectroscopy, X-ray diffraction, X-ray fluorescence, cracks.

ABSTRACT: Techniques for analyzing components of hardened concrete and studying its physical characteristics are reviewed. The methods include: light microscopy, wet chemical analysis, X-ray diffraction and fluorescence, absorption spectroscopy (ultraviolet, visible and infrared), electron microscopy, and crack analysis. The scope and problems of each method are discussed. Five examples of routine analyses using these methods individually and collectively are presented.

REFERENCE: Bernard Erlin, "Methods Used in Petrographic Studies of Concrete," *Analytical Techniques for Hydraulic Cements and Concrete, ASTM STP 395*, Am. Soc. Testing Mats., 1965, p. 3.

There are a number of analytical methods that may be used to identify components and reveal physical characteristics of hardened concrete. Information thus obtained may provide connecting links that help interpret the behavior of concrete. Some of the aspects of investigative petrography, the art of deciphering what has transpired in concrete, are briefly reviewed here.

In investigative petrography, a vast array of possibly relevant facts forces the investigator to hunt for methods that will provide the most meaningful analysis. The basic objective is to bring forth all facts, tacit or explicit, for interpretation and reconciliation with behavior. During the course of the investigation, a juncture may be reached when the scientific art will permit correlation with performance. This is the end product of the investigation—the unravelling of those influences that have directed a concrete's behavior.

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Limitations	Many interpretations are subject to personal judgement based upon experience.	Amorphous compounds are undetectable. Compounds present in low concentrations (i.e. 5% or less) may be difficult or impossible to identify.
Possible Interpretations	Potential effectiveness of the air-void system Aggregate unsoundness Type of cement-aggregate reaction Effect of cement composition on concrete behavior Curtailment of cement hydration Relative cement content Relative water-cement ratio Distribution of stress and probable causative agency Type of distress and whether caused by internal or external agency Deviation from specification	Type of aggregate Type of distress Extent of cement hydration
Information Obtainable	ASTM Recommended Practice C457 air-void system Aggregate-composition, texture, classification, other properties given in ASTM Recommended Practice C295, reaction rims Proportions of aggregate and paste Cracking patterns Identification of solid admixtures Extent of cement hydration Composition, fineness, and dispersement of relic cement particles Identification of hydrated cement compounds Identification and location of secondary compounds Identification and location of secondary compounds Detection of "unaccommodative" chemical reactions Physical properties of the paste such as hardness, granularity, porosity, density	Aggregate mineralogy Identification of secondary compounds Identification of hydrated and unhydrated cement compounds—best determined after crushing and removal of aggregate
Technique or Method	permit correlation with performance. This investigation—the unravelling of those cook oncrete a behavior another Held Research Section Fordard Communication another Stocker III.	X-ray Diffractom- etry

X-ray Spectros-	Identification and relative proportion of elements present in aggregate and paste	Estimate of cement content Presence of elements such as zinc and lead that may indicate the presence of compounds responsible for ab- normal set High concentrations of certain ele- ments that may indicate types of distress	Supplementary microscopy and X-ray diffractometry are necessary to identify the combination of elements as specific compounds.
X-ray Radiog- raphy	Cracking patterns	Distribution of stress	Some difficulty exists in resolving cracking in the mortar fraction of concrete.
Wet-Chemical Analysis	ASTM Method C85 cement content Chemical composition of aggregate Chemical composition of paste Chemical composition of secondary compounds Detection of some organic substances	Excessive quantities of certain substances that may be indicative of specific types of deterioration Quantitative estimate of cement content	Supplementary microscopy and X-ray diffractometry are necessary to identify specific compounds present.
Electron Micros- copy and Dif- fractometry	Identification of compounds in aggregate and paste Identification of secondary substances	Type of aggregate Type of distress	Certain substances are subject to alteration during examination.
Infrared Spectros- copy	Identification of many organic groups	Presence of organic admixtures Deviation from specification	Some organic groups are undetectable.

Petrography is an established science in the field of geology and deals with descriptions of rocks and relationships existing between minerals that constitute rocks. The single tool that has made descriptive petrography an elaborate science is the petrographic microscope. It permits the identification of minerals and the resolution of interrelations between minerals, and provides a rational basis for classifying rocks. Interpretations of these data have provided a basis for deciphering the origin and history of rocks.

Although petrography is often considered a science of light microscopy, in its broadest aspects it may also make use of any discipline that provides factual information about rocks such as: classical wet-chemical analysis; X-ray diffractometry and fluorescence; light spectroscopy (infrared, ultraviolet and visible); electron microscopy; and a variety of supplemental analytical methods. Although all of these analytical methods might not fall strictly within the petrographic province, they are valuable aids in an investigative examination. Often the central figure involved in an investigation is a petrographer whose primary experience and investigative equipment are related to light microscopy. However, he may call upon specialists to furnish information obtained by allied analytical methods, and collate the data obtained.

Investigative petrography may also include determinations or descriptions of physical properties such as permeability, porosity, density, absorption, and cracking patterns. An interpretation of the latter may significantly contribute to an understanding of the development and distribution of stresses within a material.

The techniques used in the investigation of rocks have been applied in studies of concrete and have provided data that has contributed significantly to an understanding of the properties and behavior of concrete. Although advances have been made in equipment, and methods and techniques have become more sophisticated in recent years, there is a demand for further refinement of existing investigative techniques and methods and development of new methods particularly in quantitative analysis.

Investigative petrography has a threefold objective: (1) to provide a compendium of factual information, which permits (2) interpretations of composition and behavior, and provides (3) additional information that may establish a basis for future research.

This paper briefly reviews several principal investigative methods that have been applied to concrete, and also presents the significant aspects of five case histories.

Techniques and Methods

Traditionally, light microscopy has been a principal investigative method. Today, it continues to serve a major role and provides a nucleus around which many methods are dependent. Table 1 summarizes information that may be obtained by the various techniques and methods.

Light Microscopy

Relatively precise microscopical methods are available for determining concrete composition, particularly the air-void system, and for identifying and characterizing compounds in concrete.

Mielenz [1],² in a review of the applications of visual and microscopical examinations, reported on such techniques for examining concretes and aggregates. He included in a tabulation of features or conditions amenable to microscopical examinations the following: (1) general features of the concrete, such as mix proportions, internal structure, cement-aggregate relationships, and deterioration; (2) descriptions of aggregates including composition, grading and quality, and qualitative information on composition and relative fineness of residual cement particles; (3) identification of solid mineral admixtures; (4) determination of characteristics of the airvoid system, and (5) identification of secondary compounds. In a later paper, Mielenz [2] described microscopical techniques used to diagnose concrete failures. Brown [3] included in his discussion of petrographic methods several general comments on the detection of "unaccommodative" chemical transformations by means of strain shadows and fiberous structures in crystals of secondary generation.

Recently, the microscope has been effective in revealing microcracking in concrete and has assisted in studying the distribution and source of stresses. Comments on this subject are presented later in the text.

ASTM utilizes microscopical methods in ASTM Recommended Practice for Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 – 60 T), and in ASTM Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295).

X-ray Diffractometry

The usefulness of X-ray diffraction methods for the qualitative identification of crystalline substances has been long demonstrated as a most useful tool for analytical purposes. The efficacy of the method, however, is in part dependent upon the relative amount of a compound present in a mixture. For example, a compound making up less than 5 per cent of a mixture may not provide enough data for identification. Selective sampling, often conveniently done under the microscope, may provide a sufficient concentration for analysis.

Amorphous compounds, such as true gels, cannot be identified by

² The italic numbers in brackets refer to the list of references appended to this paper.

X-ray analysis. Poorly crystallized compounds, such as calcium silicate hydrates derived from the hydration of the calcium silicate phases in portland cement, do not usually provide sufficient data from which positive identifications may be made.

The use of X-ray diffractometry is often desirable to confirm a doubtful optical identification, especially when the material under examination is extremely fine-grained.

Special specimen removal techniques and specimen-holding devices have been devised to house small specimens. For example, a secondary compound located in an air void or relatively tight fracture may be carefully removed with a finely pointed probe and inserted into a capillary tube of 0.2 mm ID, with one end closed and the other open to receive the specimen. A circular camera must be used to replace the less time-consuming Geiger tube method of analysis. The technique may provide definitive data for identifying specimens as small as one millionth of a cubic centimeter in volume.

X-ray Spectroscopy

This method may be applied to determine, qualitatively or quantitatively, the elemental composition of a material. Qualitative analysis simply requires measurement of the fluorescent X-ray wavelength produced by bombarding a material with a beam of primary high intensity X-rays. By the use of reference standards, relatively precise determinations of the percentage of each element present may be obtained. Although useful in many applications, the analysis allows the identification of individual elements but does not provide information on how they are combined. Other methods, such as petrographic microscopy and X-ray diffractometry, are necessary adjuncts to identify the particular combination or combinations of elements as compounds.

Chemical Analysis

"Chemical analysis" is used here to signify classical "wet-chemical" methods. These methods are often used for rapid qualitative purposes. The detection of mono-element ions such as calcium or chloride is fairly easy, but such analyses are also conveniently done by instrumental methods. The detection of multi-element ions (radicals), such as sulfate or phosphate, is generally easier by wet methods than by instrumental procedures. Quantitative analyses are often more rapidly or accurately carried out by wet-chemical methods than by instrumental ones.

A chemical method for determining the cement content of concrete is available in ASTM Method of Test for Cement Content of Hardened Portland Cement Concrete (C 85). The method is based upon determinations of lime (CaO) and silica (SiO₂) released under the conditions of the test. The determined cement content will establish the maximum proportion

of cement probable in the concrete, however the method may provide erroneously high values if aggregates contribute to the amount of lime and silica thus determined. If separate aggregate specimens are available, corrections may be applied to the values.

Mielenz [2] commented upon chemical analyses for determining cement content and the presence of chemical admixtures and contaminating substances. His discussion includes some limitations and difficulties that may arise when chemical analyses are used.

Electron Microscopy and Diffractometry

These methods have had a limited application to investigations of hardened concrete. The difficulties encountered in specimen preparation, potential dehydration of hydrous salts especially common to concrete, and the extremely small area capable of investigation, are factors that have severely limited its usefulness to date.

Electron microscopy and diffractometry may be used to identify and resolve the morphological characteristics of compounds. Erlin and Stark [4] utilized electron diffractometry to complement an investigation on the identification of thaumasite, a newly found secondary compound in concrete.

Infrared Spectroscopy

Infrared spectroscopy may be used to identify functional groups of most organic compounds. Once infrared spectroscopic characteristics for an unknown organic compound have been determined, a match of the data with data found in a compendium for known organic groups will serve to identify the unknown precisely.

The difficulties of analyzing for organic substances in concrete are compounded for the following reasons: (1) purposeful additions of organic substances to concrete are often made in quantities that represent less than about 0.02 per cent of the concrete by weight, (2) the original organic compound may be altered to other organic complexes after incorporation into a concrete mixture, and (3) extraction methods used to concentrate the organics may further alter the organic compounds.

Techniques for detecting and identifying a number of organics by infrared spectroscopy have recently been reported by Hime, Mivelaz, and Connolly [5]. Among the organic compounds successfully detected were the following: abietates, lignosulfonates, benzoates, stearates, sulfonamides, acetates, phthalic acid esters, and hydrocarbon waxes. These same techniques have also been used to detect surface coatings, such as: silicones, vinyl polymers, alkyds, styrene polymers, and impure organic acids in the surface region of concrete specimens.

Hime et al also report a number of quantitative analyses using these methods.

This investigative technique has breached a difficult technical barrier

and represents one of the more significant contributions to the investigative art that has been made during recent years.

Crack Analysis

Cracking in concrete may be caused by a variety of factors. All, however, have one point in common: they are manifestations of stress relief. Cracks may be visible to the naked eye, or microscopic enlargement of suitably prepared specimens may be required to make them visible. Brown [6] and Roper et al [7] comment upon the microscopic detection of microfractures and interpretations of stresses in concrete. Dylander [8] described an ultraviolet fluorescent method that may detect microcracks. Hsu et al [9] and Hsu [10] in an extensive series of laboratory studies used X-ray radiography techniques to reveal cracking induced in concrete cylinders by the direct application of compressive forces and by drying shrinkage.

Concrete is a heterogenous and, in some respects, a fairly inelastic material. It is relatively volume-stable. However, influences such as freezing and thawing and cement-aggregate reactions may affect the internal stability of concrete and cracking may result. Cracking patterns often permit simple interpretations of the directional propagation of stresses. Further examinations, however, will normally be required to support whatever mechanism is concluded to be responsible for the development of stresses. For example, freeze-thaw scaling of a concrete surface represents the imposition of tensile stresses below the surface. Stress relief is often manifested as a series of incipient fractures that are subparallel to the concrete surface. The association of subparallel fracturing with freezing and thawing is well recognized; the conclusion that freezing and thawing is the causative factor, however, may be further supported if the concrete is shown to be inadequately air-entrained.

Resolution of crack patterns may provide the relationship that converts subjective conjecture into established fact. There is often a tendency to presuppose what has had an effect without specifically observing the causative feature. For example, microscopic observations on paste may reveal what appears to be an abnormally high percentage of free lime or magnesia contained within relic cement particles. Conjecture may infer that the lime and magnesia have contributed to abnormal concrete performance. This direct interpretation, however, although probably significant, would be more meaningful if radial cracking were observed to extend from the particle into and through the cement paste.

Case Histories

Five case histories that are given below illustrate the applications of various investigative methods. They also demonstrate the desirability of applying a number of methods in order to obtain new or complementary

information and more securely validate whatever inferences are drawn from the determined data. These cases represent routine laboratory studies. In two of the cases the particular problem was not resolved, but direction for continued or future studies was gained.

Case 1

Several concrete blocks were plagued with popouts and cracking that precluded their use. Each affected unit contained a "contaminant" associated with the popouts and cracking. Little background information on materials and manufacturing processes was available. Six thumb-sized pieces of "contaminant" were forwarded for laboratory examination.

The particles were examined microscopically to determine whether one or more materials were involved. Each particle was subrounded to angular in shape and possessed a mottled light grey to grey or buff color. Several particles displayed a type of pattern-cracking characteristic of shrinkage, but were relatively dense and broke with a semiconchoidal fracture that had a waxy luster. Others were relatively soft throughout and contained subparallel and random partings. General observations indicated the particles contained similar mixtures of compounds.

One particle was ground to minus-100-mesh fineness and analyzed by X-ray spectroscopic (fluorescence) methods. Major amounts of aluminum, calcium, sulfur, and minor traces of potassium, silicon, and titanium were detected.

Chemical analysis performed upon a second particle revealed an oxide composition of 43 per cent lime, 17 per cent alumina, 13 per cent sulfate, 4 per cent silica, and traces of ferrous iron, magnesia, and alkalies. Ignition loss was 22 per cent.

Identification of compounds in several particles by X-ray diffractometry and petrographic microscopy indicated the presence of two anhydrous compound phases, $3\text{CaO}\cdot3\text{Al}_2\text{O}_3\cdot\text{CaSO}_4$ and CaSO_4 (anhydrite), and what might be anticipated as hydrous phases derived from some combinations of the anhydrous phases with water: $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaSO}_4\cdot12\text{H}_2\text{O},$ $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot3\text{CaSO}_4\cdot32\text{H}_2\text{O}$ (ettringite), and $\text{Ca}(\text{OH})_2$.

A particle that under the microscope appeared to contain a maximum of anhydrous phases was crushed to about 100-mesh fineness, placed into the well of a flat X-ray specimen holder, and analyzed to determine whether its compound composition was similar to the material previously identified. The material was then removed, gauged with water, and examined by X-ray diffractometry over a period of about 24 hours.

Interpretation of the X-ray data revealed that a progressive change in relative proportion of several of the compounds was occurring. The anhydrous phases and the lower watered hydrous phase were decreasing in proportion relative to an increase in the proportion of ettringite. Ex-

pansion of the material was also evidenced by a slight billowing of the surface, which had to be releveled for subsequent examinations.

The mixture of substances comprising the "contaminant" was indexed and identified as anhydrous compounds found in a synthetic cement reported by Klein and Troxell [11], and Halstead and Moore [12], and hydrous phases derived from their hydration as reported by Klein and Troxell.

During the course of investigation, wet-chemical analysis, X-ray spectroscopy and diffractometry, and petrographic microscopy were utilized to identify and confirm the composition of the "contaminant," and during the course of X-ray study, evidence of the expansive potential of the material was directly observed.

Case 2

A 6 by 12-in. concrete cylinder and pieces from two concrete cylinders that had been tested in compression were examined to determine the cause for lower than anticipated compressive strengths that purportedly occurred during a period involving the use of a particular air-entraining agent.

Concrete mix design specifications required 658 lb of cement (7 U.S. bags) per cubic yard of concrete and an air-entraining admixture.

Microscopic examinations of the specimens revealed that each contained similar coarse aggregates: a quarried limestone of variable texture but predominantly fine-grained and dense. The fine aggregate in all specimens was subrounded, brown, dense chert in the coarser sizes and subrounded to rounded clear quartz in the finer sizes. All cylinders appeared to be deficient in the smaller sizes of the coarse aggregate. This deficiency was compensated by an increase in the proportion of fine aggregate.

Specimens representing the tested concretes were characterized by a lighter-colored paste than the paste in the third cylinder. Microscopic examinations of polished sections, viewed under incident light, revealed a considerably greater number of residual cement fragments in the cylinder containing the darker-colored paste. This feature suggested variations in cement content existed between the cylinders.

The air-void system for each specimen was determined by the linear traverse method (ASTM Method C 457). The air-void systems were similar in all specimens.

The initial microscopic investigation was then supplemented by chemical analysis for cement content, ASTM Method C 85, and by infrared spectroscopic analyses to determine whether the air-entraining agent could be detected, and if detectable, whether differences in air-entraining agents could be revealed.

Analysis by ASTM Method C 85 showed a marked difference in cement contents. The two concretes displaying lighter-colored pastes contained 8.6 and 11.5 per cent cement, while the concrete containing the darker-

colored paste was found to contain 17.0 per cent cement. The cement contents were based upon the percentage of silica uncorrected for aggregate. The data, recalculated in pounds of cement by the use of design batch weights and the determined air contents as references, indicated the three specimens contained 339, 454, and 671 lb, respectively, of cement per cubic yard of concrete.

The three specimens were analyzed by infrared techniques. Extracts from one of the concretes of low cement content gave an infrared spectroscopic pattern identical to one that resulted when an extract from a laboratory-prepared mortar bar containing a lignosulfonate additive was similarly analyzed. The analyses failed to detect the specified air-entraining agent; however, air-entraining admixtures of the type purported to have been used have, to date, not been detected by the infrared methods utilized.

Three analytical methods—chemical analysis, infrared spectroscopy, and light microscopy—were effectively utilized to reveal pertinent features and characteristics of these concretes. The data indicated a variability in cement content among the three specimens and the presence of an organic compound commonly used in a number of admixtures.

Case 3

A number of small specimens of concrete from the spalled and cracked ceiling of a kiln used to process a mixture of organic and inorganic compounds were examined to determine the cause for poor performance. The ceiling surface was represented by several fragments containing one smooth flat surface superficially stained light to dark brown. These and other fragments contained a number of subparallel microcracks that occurred principally within coarse aggregate particles. The disrupted aggregate was coarsely textured and composed of calcite, dolomite, and traces of clay and quartz. Occasionally cracking extended into the paste. Other coarse aggregate particles flaked and granulated under slight finger pressures. None of the fine aggregate particles, quartz, appeared damaged.

Microscopic examinations revealed that the paste was firm and sound and carbonated adjacent to fractures and other exposed surfaces. The examinations failed to detect secondary compounds in the paste.

Infrared analysis revealed the presence of a petroleum wax and a mixture of impure organic acids in several of the pieces selected for examination. Subsequent chemical analysis indicated 0.006 per cent organic wax was present in a composite specimen of several other fragments.

The presence of organic wax and organic acids in the specimens indicated temperatures in the kiln were sufficiently high to volatilize certain ingredients in the material under cure. The absence of perceivable secondary compounds, however, suggested that some sort of physical phenomena, such as thermal response to varying temperatures, may have been responsible for the abnormal concrete behavior.

Although the investigation failed to reveal the specific condition responsible for the deterioration, the absence of secondary compounds eliminated chemical attack from consideration. The data did suggest, however, that an incompatibility existed between coarse aggregate and environment, and directed attention to a separate investigation of the coarse aggregate.

Case 4

A small specimen taken from concrete purported to have air contents as high as 18 per cent was examined to determine the cause for the excessive air volume. A commonly used air-entraining admixture was reported to have been used.

The unit weight of the section of concrete was determined gravimetrically, and subsequently measured for air content by the linear traverse method performed in conformance with ASTM Method C 457. A comparison of the non-air-entrained weight calculated from batch design data and the gravimetrically determined unit weight, indicated the section had an air content of 5.2 per cent. The measured linear traverse air volume was 4.9 per cent.

The air-voids occurred as discrete units scattered throughout the paste, occasionally as small frothy clusters, and frequently as a froth partially circumscribing particles of coarse aggregate (a crushed, dense, mediumto-dark grey, fine-grained limestone of maximum 1 in. size).

An infrared spectroscopic pattern of a small portion of the specimen compared with the pattern of a phthalic acid ester. A search of the literature revealed that phthalic acid derivatives have been proposed as airdetraining agents. The presence of such an organic compound in concrete posed the question of whether it entered the concrete as: (1) a component of an admixture or contaminant, (2) organic contamination in the aggregate, or (3) the result of interactions between the air-entraining agent and the chemicals used in processing the concrete specimen for the analysis.

To determine whether the second condition existed, particles of coarse aggregate were broken from the concrete and carefully scraped to remove adhering paste. To determine whether the third condition existed, the type of air-entraining agent reportedly used in the concrete was gauged with portland cement and water and stored for $2\frac{1}{2}$ days at 100 C. Separate infrared spectroscopic examinations of the aggregate and paste failed to reveal the presence of a phthalic compound.

The examinations did not confirm the abnormally high air contents reported for the concrete from which the specimen was removed. The examination did, however, reveal an abnormal distribution of entrained air and the presence of an organic compound. The effect of the compound on the air-void system could be studied by testing of laboratory-prepared concretes.

Case 5

Specimens from four different concrete products examined during a two-year period revealed the presence of secondary compounds possessing similar optical, X-ray, and morphological characteristics. The compound was identified initially on the basis of optical properties as the mineral thaumasite, a hydrous salt reported to contain the following proportions: CaSiO₃·CaSO₄·CaCO₃·14.5H₂O. The compound had not been previously identified in concrete. The material was the subject of a paper by Erlin and Stark [7] who investigated the compound by several methods.

The initial petrographic microscopy and X-ray data provided primary identification of the compound. Electron diffractometry data ruled out a similarly structured material, ettringite, which is of different composition and is commonly observed in concrete; classical wet-chemical methods provided data on oxide composition that was relatively close to the commonly accepted composition of the material. Each analytical method complemented the others. Without such confirming data, a question of precise identification might still exist.

Summary

Investigative petrography may include a number of techniques and methods. Among these are light microscopy, X-ray diffractometry and X-ray spectroscopy, conventional wet-chemical analysis, infrared spectroscopy, and electron microscopy and diffractometry. Each technique and method provides complementing or supporting data, broadens the base from which interpretations may be drawn, and more securely validates the final conclusion.

Light microscopy will permit determinations of concrete composition particularly in resolving the air-void system, identification and characterization of compounds, and resolution of cracking patterns. X-ray diffractometry may be utilized to identify compounds; however, limitations may be imposed if small percentages of compounds are present in mixtures. X-ray spectroscopy may also be applied to determine elemental composition. Petrographic microscopy and X-ray diffractometry are normally required to categorize the particular combination of elements. Conventional wet-chemical methods may be used to detect mono-element or multi-element ions. A wet-chemical method, ASTM Method C 85, may be used to determine cement content. Electron microscopy and diffractometry have had limited applications and may be used to identify compounds. Infrared spectroscopy, a relatively new technique, has been successfully used to identify a number of organic compounds that may represent less than 0.02 per cent of the concrete by weight. Analysis of cracking patterns revealed by microscopic or X-ray techniques may resolve the directional propagation of stresses.

The five case histories presented in this paper demonstrate routine applications of a number of these techniques and methods.

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DISCUSSION

A. A. Tabikh—Mr. Erlin made no mention in his paper of the electron microprobe analyzer This instrument combines some features of of the electron microscope with X-ray emission spectrography for simultaneous observations and elemental analysis of the point under examination. Has Mr. Erlin used such an instrument? What are the future prospects for it in cement and concrete studies?

B. Erlin (author)—We have not had an opportunity to use an electron microprobe analyzer in our work, but the instrument promises to be extremely useful. With it one can determine the distribution of a number of elements between phases in a particular area, and simultaneously obtain the equivalent of a micrograph of the same area. A few electron microprobe experiments have been made using polished sections of portland-cement clinkers. The preliminary results, obtained by Miss Alice Moore of the Cement and Concrete Association in England, indicate that this instrument may provide a means for determining the elemental composition of each of the major phases in portland cement. It should also be possible to determine the distribution of the minor constituents among the different phases. The potential usefulness of this instrument for the study of concrete should be just as great as it is for the study of clinker.

PCA.R&D.Ser.1166-2

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111. "Petrography of Cement and Concrete," by L. S. Brown.

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112. "The Gravimetric Determination of Strontium Oxide in Portland Cement," by C. L. Ford.

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113. "Quantitative Determination of the Four Major Phases in Portland Cement by X-Ray Analysis," by Stephen Brunauer, L. E. Copeland, D. L. Kantro, C. H. Weise and Edith G. Schulz.

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- 115. "A Gravimetric Method for the Determination of Barium Oxide in Portland Cement," by C. L. Ford.
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- 116. "The Thermodynamic Functions for the Solution of Calcium Hydroxide in Water," by S. A. Greenberg and L. E. Copeland.

Reprinted from Journal of Physical Chemistry, 64, 1057-1059 (August, 1960).

- 117. "Investigation of Colloidal Hydrated Silicates. I. Solubility Products," by S. A. Greenberg, T. N. Chang and Elaine Anderson.

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- 118. "Some Aspects of Durability and Volume Change of Concrete for Pre-

stressing," by Paul Klieger.

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- 119. "Concrete Mix Water—How Impure Can It Be?" by HAROLD H. STEINOUR.

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- 120. "Corrosion of Prestressed Wire in Concrete," by G. E. Monfore and G. J. Verbeck.

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121. "Freezing and Thawing Tests of Lightweight Aggregate Concrete," by Paul Klieger and J. A. Hanson.

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122. "A Cement-Aggregate Reaction That Occurs With Certain Sand-Gravel Aggregates," by WILLIAM LERCH.

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123. "Volume Changes of Concrete Affected by Aggregate Type," by HAROLD ROPER.

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124. "A Short Method for the Flame Photometric Determination of Magnesium, Manganic, Sodium, and Potassium Oxides in Portland Cement," by C. L. Ford.

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125. "Some Physical Aspects of the Hydration of Portland Cement," by T. C. Powers.

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126. "Influence of Physical Characteristics of Aggregates on Frost Resistance of Concrete," by George Verbeck and Robert Landgren.

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- 127. "Determination of the Free Calcium Hydroxide Contents of Hydrated Portland Cements and Calcium Silicates," by E. E. Pressler, Stephen Brunauer, D. L. Kantro, and C. H. Weise.

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- 129. "Dimensional Changes of Hardened Portland Cement Pastes Caused by Temperature Changes," by R. A. Helmuth.

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- 130. "Progress in the Chemistry of Portland Cement, 1887-1960," by Harold H. Steinour.

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131. "Research on Fire Resistance of Prestressed Concrete," by Hubert Woods, including discussion by V. Paschkis, and author's closure.

Reprinted from Journal of the Structural Division, Proceedings of the American Society of Civil Engineers, Proc. Paper 2640, 86, ST 11, 53-64 (November.

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132. "Centralized Control of Test Furnaces in the PCA Fire Research Laboratory," by Phil J. Tatman.

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133. "A Proposed Simple Test Method for Determining the Permeability of Concrete," by I. L. Tyler and Bernard Erlin.

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134. "The Behavior at High Temperature of Steel Strand for Prestressed Concrete," by M. S. Abrams and C. R. Cruz.

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- 135. "Electron Optical Investigation of the Hydration Products of Calcium Silicates and Portland Cement," by L. E. Copeland and Edith G. Schulz.

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- 136. "Soil-Cement Technology—A Resume," by Miles D. Catton.

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- 137. "Surface Temperature Measurements With Felted Asbestos Pads," by M. S. ABRAMS.

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- 138. "Tobermorite Gel—The Heart of Concrete," by Stephen Brunauer.

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- 139. "Alkali Reactivity of Carbonate Rocks—Expansion and Dedolomitization," by David W. Hadley.

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140. "Development of Surface in the Hydration of Calcium Silicates," by D. L. Kantro, Stephen Brunauer, and C. H. Weise.

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143. "New Techniques for Temperature and Humidity Control in X-Ray Diffractometry," by Paul Seligmann and N. R. Greening.

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144. "An Optical Method for Determining the Elastic Constants of Concrete," by C. R. CRUZ.

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145. "Physical Properties of Concrete at Very Low Temperatures," by G. E. Monfore and A. E. Lentz.

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- 146. "A Hypothesis on Carbonation Shrinkage," by T. C. Powers.

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- 147. "Fire Resistance of Prestressed Concrete Beams. Study A—Influence of Thickness of Concrete Covering Over Prestressing Steel Strand," by C. C. Carlson.

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- 148. "Prevention of Frost Damage to Green Concrete," by T. C. Powers.

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- 149. "Air Content of Hardened Concrete by a High-Pressure Method," by BERNARD ERLIN.

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150. "A Direct Current Strain Bridge," and "A Biaxial Strain Apparatus for Small Cylinders," by G. E. Monfore.

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155. "The Rheology of Fresh Portland Cement Pastes," by Moshe Ish-Shalom and S. A. Greenberg.

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157. "Extensions to the Long-Time Study of Cement Performance in Concrete," by Paul Klieger.

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- 158. "Durability Studies of Exposed Aggregate Panels," by A. W. ISBERNER.

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- 159. "Corrodibility of Prestressing Wire in Concretes Made With Type I and Type IS Cements," by Borje Ost and G. E. Monfore.

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160. "A Small Probe-Type Gage for Measuring Relative Humidity," by G. E. Monfore.

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161. "Abnormal Cracking in Highway Structures in Georgia and Alabama," by Calvin C. Oleson.

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163. "A Sonic Method to Determine Pavement Thickness," by RICHARD MUENOW.

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164. "Effect of Restraint on Fire Resistance of Prestressed Concrete," by S. L. Selvaggio and C. C. Carlson.

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165. "Effect of Surface Grinding and Joint Sawing on the Durability of Paving Concrete," by William Perenchio.

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166. "Quantitative Determination of the Major Phases in Portland Cements by X-Ray Diffraction Methods," by D. L. Kantro, L. E. Copeland, C. H. Weise, and Stephen Brunauer.

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167. "Pore Structures and Surface Areas of Hardened Portland Cement Pastes by Nitrogen Adsorption," by R. Sh. Mikhail, L. E. Copeland, and Stephen Brunauer.

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169. "Silicone Influence on Concrete Resistance to Freeze-Thaw and De-Icer Damage," by Paul Klieger and William Perenchio.

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170. "Properties of Expansive Cement Made With Portland Cement, Gypsum, and Calcium Aluminate Cement," by G. E. Monfore.

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171. "Fire Resistance of Prestressed Concrete Beams. Study B. Influence of Aggregate and Load Intensity," by S. L. Selvaggio and C. C. Carlson.

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172. "Petrographic Studies on Concrete Containing Shrinking Aggregate," by HAROLD ROPER, J. E. COX and BERNARD ERLIN.

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173. "Corrosion of Aluminum Conduit in Concrete," by G. E. Monfore and Borje Ost.

Reprinted from the Journal of the PCA Research and Development Laboratories, 7, No. 1, 10-22 (January, 1965).

174. "Topics in Concrete Technology — 1. Geometric Properties of Particles and Aggregates, 2. Analysis of Plastic Concrete Mixtures, 3. Mixtures Containing Intentionally Entrained Air, and 4. Characteristics of Air-Void Systems." by T. C. Powers.

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- 176. "Alkali Reactivity of Dolomitic Carbonate Rocks," by DAVID W. HADLEY.

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- 177. "Alkali-Reactive Carbonate Rocks in Indiana—A Pilot Regional Investigation," by David W. Hadley.

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178. "Water-Vapor Adsorption-Desorption Characteristics of Selected Lightweight Concrete Aggregates," by ROBERT LANDGREN.

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179. "Determining the Water Absorption of Coarse Lightweight Aggregates for Concrete," by Robert Landgren.

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180. "Investigation of Colloidal Hydrated Calcium Silicates. II. Solubility Relationships in the Calcium Oxide-Silica-Water System at 25°," by S. A. Greenberg and T. N. Chang.

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181. "Concrete Drying Methods and Their Effect on Fire Resistance," by M. S. ABRAMS and D. L. ORALS.

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182. "Thermal Conductivity of Concrete at Very Low Temperatures," by A. E. Lentz and G. E. Monfore.

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- 183. "An Improved Procedure for Proportioning Mixes of Structural Lightweight Concrete," by R. Landgren, J. A. Hanson, and D. W. Pfeifer.

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- 184. "Particle Size Distribution of Portland Cement from Wagner Turbidimeter Data," by W. G. HIME and E. G. LABONDE.
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- 185. "Studies of Early Hydration Reactions of Portland Cement by X-Ray Diffraction," by Paul Seligmann and Nathan R. Greening.

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- 186. "Application of a Small Probe-Type Relative Humidity Gage to Research on Fire Resistance of Concrete," by M. S. Abrams and G. E. Monfore.

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187. "A Rapid Method for Studying Corrosion Inhibition of Steel in Concrete," by Venice K. Gouda and G. E. Monfore.

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188. "Oxygen Enrichment of Combustion Air in Rotary Kilns," by ROBERT A. GAYDOS.

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- 189. "Cement Hydration Reactions at Early Ages," by George Verbeck.

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- 190. "Expansive Concrete—Laboratory Tests of Freeze-Thaw and Surface Scaling Resistance," by A. H. Gustaferro, N. Greening, and P. Klieger.

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- 191. "Elastic Properties of Concrete at High Temperatures," by Carlos R. Cruz.
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- 192. "Penetration of Chloride into Concrete," by Borje Ost and G. E. Mon-FORE.
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- 193. "Methods Used in Petrographic Studies of Concrete," by BERNARD ERLIN.

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