The Use of SEM and Other Complimentary Techniques for the Determination of Properties of Cementitious Materials

Eric A. Draper & Jan Skalny
RJ Lee Group, Inc., Monroeville, PA 15146

The need for continued rehabilitation of our concrete infrastructure has lead to the adaptation of modern "state-of-the-art" analytical methods for the characterization of concrete and other cementitious materials. Some of these techniques have not, until relatively recently, been commonly associated with the evaluation of concrete but are very useful both as tools for quality assurance and in the determination of the extent of existing damage. The technique of interest here is the coordinated electron-optical microscopic evaluation of concrete.

Concrete is the most widely used building material in the world. Contrary to popular belief, concrete is not inert but chemically very complex and dynamic. While it is true that, pound for pound, concrete and its raw materials (cement, aggregate and water) are the most inexpensive building materials available for construction, it is also true that it responds to its environment in numerous and sometimes very subtle ways. These responses may sometimes result in a loss of durability and tremendous amounts of time and money being expended while searching for the cause(s) of the problem and providing a cost-effect solution. A quick survey of any large metropolitan area and the on-going construction repairs to highways and bridge decks there will quickly confirm this.

Traditional bulk analytical methods for testing concrete typically yield information on the performance of the concrete as a whole. That is to say that information on specific components of the concrete (cement, sand, coarse aggregate, etc.) and their interactions, may not be available. This is especially true when examining concrete for a potentially harmful reaction between the aggregate and cement paste (the product of mixing cement powder and water), known as alkali-silica reaction (ASR). Briefly stated, this reaction involves the production of an expansive gel (called ASR gel) within the concrete structure which can, subsequent to its formation, cause the structure to crack and require replacement. Petrographic methods for concrete examination according to ASTM are also typically used in cases such as these, but positive identification of the ASR gel, which is essential here, may not be possible using optical methods alone. This is especially true if the cracks in the aggregate and cement paste are very fine and only minor amounts of gel are present. Since the chemical composition of the gel can also vary, within limits, it becomes very important to know what this composition is. SEM, microprobe and X-ray analyses are of use in this case, but it is typical, because of different sample prep requirements, to utilize different samples for the various analytical techniques. This can create a problem when trying to correlate the information obtained from each analytical method. One of the greatest problems when analyzing the same sample using different techniques has always been relocating identical areas of interest so that a direct comparison of the analytical results is possible. This process has typically been accomplished by painting or scribing marks on the sample in the area of interest, then searching with the second instrument for the marks so that the analysis may be performed. The recent development of computer-controlled optical and electron microscopes and their stages makes this procedure less time consuming and more accurate. Computer-controlled stages, along with the software to efficiently run them, have been developed to permit relocation of identical areas observed in the stereo optical or petrographic microscopes, and in the SEM or vice versa. In addition, digital imaging and database techniques have been developed which permit the images to be stored or retrieved from any of the instruments.

As an illustration of the effectiveness of this complimentary technique, a section of concrete was prepared and analyzed. Figure 1 is a stereo optical image of a polished section showing a cracked aggregate in cement paste and what appears to be some reaction product (ASR gel?) within the cracks. Since the aggregate type directly influences the formation mechanism and occurrence of ASR gel, the mineralogical composition of the aggregate as well as the composition of the material within the cracks must be determined. Coordinates and images of these features were obtained from the sample and stored. Figures 2 is a BSE image of the area in question at 10X magnification. Figure 3 is an X-ray map of the same area showing the elemental distribution of the area in question and indicating that the cracked aggregate is quartz. It also indicates the presence of ASR gel in the crack. Figure 4 shows the X-ray spectrum obtained from the gel within the cracks and serves as additional confirmation of the identification.

Because of our dependence on concrete as an infrastructure material, we need to employ all the means at our disposal in order to understand how it responds to its environment. This will enable us to more accurately predict how it will perform during its service life. Supplied with this knowledge, we can then design concrete structures for optimum durability. Methods and analytical techniques traditionally developed for materials analysis can be used with excellent results when evaluating concrete, especially when used in conjunction with each other. Computer programs and computer-controlled equipment designed to maximize and compliment each instrument's capabilities are essential for the efficient acquisition of the data.

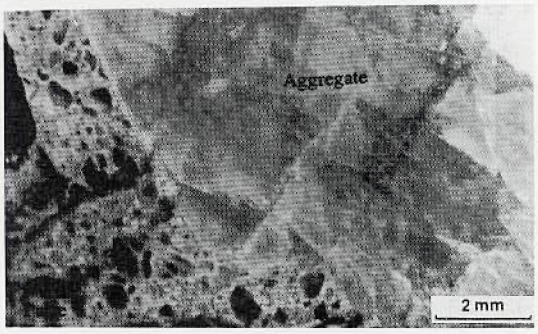

Figure 1

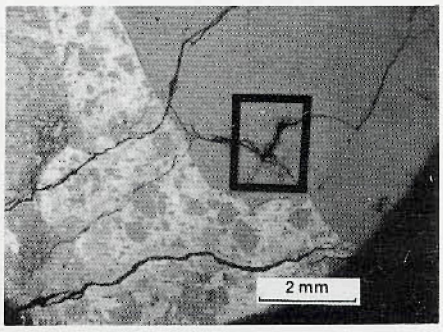

Figure 2

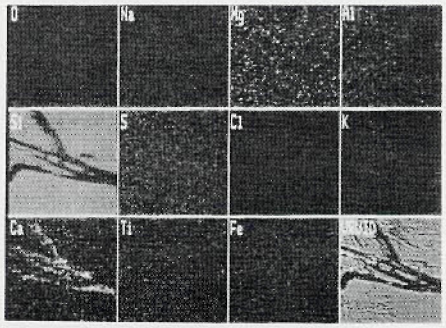

Figure 3

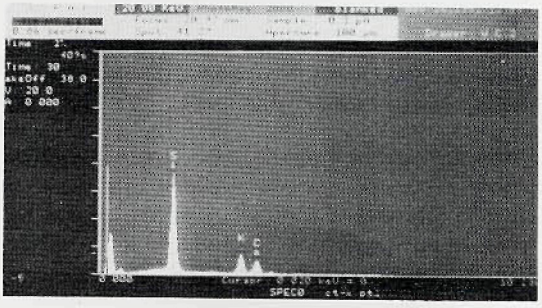

Figure 4

MICROSCOPY SOCIETY OF AMERICA

Affiliate Society of American Institute of Physics • Affiliate Society of A.A.A.S.

Purpose. The Microscopy Society of America (MSA) was founded to "increase and diffuse, for scientific and educational goals only, the science and practice of microscopy, imaging and compositional analysis and the instruments and results relating to such activities." The Society is unique in that biological and physical scientists, industrial and clinical workers, technologists, and students all benefit from membership and can exchange information on techniques and instrumentation common to a broad range of studies.

History. The Microscopy Society of America was formed in 1942 as The Electron Microscopy Society of America (EMSA) by a small group of scientists and engineers in the early days of the development of electron microscopy. Over the years, the Society not only has grown in size, but has broadened in scope. After a full fifty years as The Electron Microscope Society of America, in 1993 the name of the Society will be changed to The Microscopy Society of America to better reflect and encompass the diversity of microscopies and associated techniques that have evolved. In addition to instrumentation and techniques, the Society is concerned with a wide range of applications in both the physical and biological sciences, as well as the development and use of all forms of microscopy, imaging and compositional analysis.

MSA Today. Currently there are over 5,000 members, including about 450 student members. The membership reflects a healthy balance among all aspects of the field. The Society is particularly interested in encouraging the activities of young scientists, and offers scholarships and reduced annual dues. As further described, the Society holds an Annual Meeting, often in conjunction with other Societies, and carries out a number of activities and services for its members throughout the year. The Society represents the profession in public affairs, and serves as a center for education and information dissemination for microscopy-related knowledge.

Benefits of Membership. The MSA is an internationally recognized professional society and is the world's largest organization concerned with microscopy. Specific membership benefits include:

- ✓ Advance information and reduced registration fees for the Annual Meeting.
- ✓ Access to educational materials, such as videotapes and book lists.
- ✓ Access to the Society's electronic bulletin board.
- ✓ Membership Directory.
- ✓ Reduced prices for a number of book and journals.
- ✓ A subscription to the MAS *BULLETIN*, a high quality journal of scientific articles and the major source of current information about MSA.
- ✓ Use of the Employment Placement Office.
- ✓ Eligibility for the Technologists' Certification Program.
- ✓ Eligibility for membership in the Technologists' Forum and other special interest groups.

In addition, members benefit from MSA's affiliation with the American Physical Society, the American Association for the Advancement of Science, and the International Federation of Societies for Electron Microscopy.

Types of Membership. MSA has Regular, Sustaining (Corporate), Student, Emeritus, and Honorary members. Regular membership in MSA is open to anyone with a professional interest in microscopy and/or related areas. Sustaining membership is available to corporations, institutions, or organizations desiring to support the Society in a special way. Full-time students pursuing microscopy-related studies are eligible for student membership.

Local Affiliated Societies. The MSA is formally affiliated with 30 local and regional societies through the United States. Most of these groups meet several times a year and provide more frequent opportunity for professional interaction than the national Society.

Technologist's Forum. This is a special interest group which addresses and promotes the interests of technologists within the MSA. The Forum organizes a one-day symposium at the Annual Meeting and publishes a Forum newsletter. It offers a channel for personal growth and development by increasing contact among its members and expanding their participation and contribution to the MSA. Forum services available to members include the Technical Advisory Network and the Micrograph Information Collection and Referral Office.

Electronic Bulletin Board. The Society has an electronic bulletin board, with 800 number, that enables rapid exchanges of questions, answers, reports, announcements, etc. within the Society. The Microbeam Analysis Society has joined MSA in this endeavor.

Annual Meeting. The MSA's Annual Meeting is the premier scientific event in microscopy and related areas. It presents an opportunity to meet colleagues nationwide, to exchange scientific information, to review progress in the field, and to inspect new products and instruments. Attended by several thousand people. It is held for five days, usually in August, in a major city. The program features symposia, workshops, tutorials, a Technologists Forum and contributed sessions on a variety of subjects, both traditional and topical. A large commercial exhibition is a major part of the Annual Meeting and a variety of social events and tours are included.

The *MSA BULLETIN* is published three times a year. This illustrated publication is a highly professional journal which contains peer reviewed, invited and contributed scientific articles on timely and topical aspects of all fields of microscopy, imaging, compositional microanalysis, etc. The *BULLETIN* also includes information on MSA activities, the Annual Meeting, reviews of recent advances and techniques, book reviews, and technologist's viewpoints.

The *MSA PROCEEDINGS* is a record of scientific presentations at the Annual Meeting. It is comprised of two-page extended abstracts, including high-quality half-tone reproductions, of contributed and invited papers; the *1992 PROCEEDINGS* consisted of two volumes, containing almost 1800 pages.

Educational Activities. The MSA maintains a library of videotapes consisting of more than 150 titles on various practical, theoretical, and historical aspects of microscopy and related areas. For a small fee, these tapes may be rented (and copied), or purchased by members. This committee has recently begun participation with the Lawrence Hall of Science in Berkeley, CA to produce a teaching module on microscopy to be made available to schools and teachers across the country.

International Activities. The MSA has a standing International Committee that is active in establishing contacts with individual microscopists and societies throughout the world, particularly in developing and dollar-poor countries. Committee members have organized and participated in exchanges between scientists from the Society and scientists in other countries such as China and Korea.

Certification for Technologists. The MSA operates the only generally recognized program for examining and certifying the competence of electron microscope technologists in the biological sciences. MSA certification is a widely respected attainment, often advantageous in matters of employment and advancement.

Placement Service. The Placement Office operates a placement service for both prospective employers and employees. The service is free for employees (available only to members), and voluntary contributions are accepted from employers. Listings are published regularly in the *MSA BULLETIN*.

MICROSCOPY SOCIETY OF AMERICA, *Box MSA, Woods Hole, MA 02543* *1-800-538-3672*
FAX 508-548-9053

APPLICATION FOR MEMBERSHIP

Name *(print)*: _____ Dr. ☐ Mr. ☐ Ms. ☐

Mailing Address: _____

_____

_____ Is this a home address? YES☐ NO ☐

Institutional Affiliation *(if not part of mailing address)*: _____

Phone *(days)*: ( ) _____ Major Interest: Physical Sciences ☐ Biological Sciences ☐

Fax: ( ) _____ E-Mail: _____

Signature of nominating EMSA Member: Donald L. Grimes

Signature of advisor *(for student applicants)*: _____

Signature of applicant: _____ Date: _____

Enclose a check *(U.S. funds, drawn on a U.S. bank)* for one year's dues, payable to EMSA, or complete the credit card information below. Also enclose a brief statement of your qualifications, experience, and/or student status.

																Expires		—	

Visa/MasterCard Number (List *all* numbers on card) Month Year

Regular Member: $30 ☐ **Student Member: $5 ☐** **Sustaining Member: $250 ☐**

MICROSCOPY TODAY
PO Box 122 - Middleton, WI 53562-0122

Reader Please Note:
If there is a Code # (1 -17) in the line over your name, the following address is not considered valid by the U.S. Postal Service

Bulk Rate
U.S. Postage
PAID
Permit No. 1547
Madison, WI