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Standard Guide for Examination of Hardened Concrete Using Scanning Electron Microscopy¹

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1. Scope

1.1 This guide provides information for the examination of hardened concrete using scanning electron microscopy (SEM) combined with energy-dispersive X-ray spectroscopy (EDX or EDS). Since the 1960s, SEM has been used for the examination of concrete and has proved to be an insightful tool for the microstructural analysis of concrete and its components. There are no standardized procedures for the SEM analysis of concrete. SEM supplements techniques of light microscopy, which are described in Practice C856/C856M, and, when applicable, techniques described in Practice C856/C856M should be consulted for SEM analysis. For further study, see the bibliography at the end of this guide.

1.2 This guide is intended to provide a general introduction to the application of SEM/EDS analytical techniques for the examination and analysis of concrete. It is meant to be useful to engineers and scientists who want to study concrete and who are familiar with, but not expert in, the operation and application of SEM/EDS technology. The guide is not intended to provide explicit instructions concerning the operation of this technology or interpretation of information obtained through SEM/EDS.

1.3 It is critical that petrographer or operator or both be familiar with the SEM/EDX (EDS) equipment, specimen preparation procedures, and the use of other appropriate procedures for this purpose. This guide does not discuss data interpretation. Proper data interpretation is best done by individuals knowledgeable about the significance and limitations of SEM/EDX (EDS) and the materials being evaluated.

1.4 The SEM provides images that can range in scale from a low magnification (for example, 15 \times) to a high magnification (for example, 50 000 \times or greater) of concrete specimens such as fragments, polished surfaces, or powders. These images can provide information indicating compositional or topographical variations in the observed specimen. The EDX (EDS) system

can be used to qualitatively or quantitatively determine the elemental composition of very small volumes intersecting the surface of the observed specimen (for example, 1-10 cubic microns) and those measured compositional determinations can be correlated with specific features observed in the SEM image. See Note 1.

NOTE 1—An electronic document consisting of electron micrographs and EDX (EDS) spectra illustrating the materials, reaction products, and phenomena discussed below is available at <http://netfiles.uiuc.edu/dlange/www/CML/index.html>.

1.5 Performance of SEM and EDX (EDS) analyses on hardened concrete specimens can, in some cases, present unique challenges not normally encountered with other materials analyzed using the same techniques.

1.6 This guide can be used to assist a concrete petrographer in performing or interpreting SEM and EDX (EDS) analyses in a manner that maximizes the usefulness of these techniques in conducting petrographic examinations of concrete and other cementitious materials, such as mortar and stucco. For a more in-depth, comprehensive tutorial on scanning electron microscopy or the petrographic examination of concrete and concrete-related materials, the reader is directed to the additional publications referenced in the bibliography section of this guide.

1.7 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with the use of electron microscopes, X-ray spectrometers, chemicals, and equipment used to prepare samples for electron microscopy. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This guide is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.65 on Petrography.

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2. Referenced Documents

2.1 ASTM Standards:²

- C125** Terminology Relating to Concrete and Concrete Aggregates
- C294** Descriptive Nomenclature for Constituents of Concrete Aggregates
- C295/C295M** Guide for Petrographic Examination of Aggregates for Concrete
- C457/C457M** Test Method for Microscopical Determination of Parameters of the Air-Void System in Hardened Concrete
- C856/C856M** Practice for Petrographic Examination of Hardened Concrete
- C1356** Test Method for Quantitative Determination of Phases in Portland Cement Clinker by Microscopical Point-Count Procedure

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *BSE*, *n*—backscatter electrons; these are high-energy electrons emitted back from the specimen surface. Elements of higher atomic number will have stronger emissions and appear brighter.

3.1.2 *brightness*, *n*—the amount of energy used to produce an X-ray.

3.1.3 *charging*, *n*—the buildup of electrons on the specimen at the point where the beam impacts the sample. Charging can alter the normal contrast of the image (usually becomes brighter) and may deflect the beam. Coating the specimen with a thin layer of conductive material (such as gold or carbon) can minimize this effect.

3.1.4 *contrast*, *n*—the difference in intensity of the energy produced by varying elements when excited.

3.1.5 *dead-time*, *n*—the time of finite processing during which the circuit is “dead” and unable to accept a new pulse from the X-rays.

3.1.6 *EDX (EDS) (energy-dispersive X-ray spectroscopy)*, *n*—the interaction of the electron beam with atoms in the sample produces characteristic X-rays having energies and wavelengths unique to atoms.

3.1.7 *live-time*, *n*—how the acquisition of X-ray data is timed when the rate of X-ray events between measurements are compared. Opposite of dead-time.

3.1.8 *K, L, or M peaks*, *n*—characteristic X-ray intensities detected for elements.

3.1.9 *raster*, *n*—to scan as when the beam from the filament sweeps back and forth over the sample

3.1.10 *SE*, *n*—secondary electrons; these are low-energy electrons emitted when the specimen is hit with the beam and associated with the topography of the same.

3.1.11 *SEM*, *n*—scanning electron microscope.

3.1.12 *stage*, *n*—platform upon which the specimen is placed within the vacuum chamber that can be remotely moved in various directions.

3.1.13 *working distance*, *n*—the distance between the detector and the sample. Each SEM will have an optimum distance in which X-rays can be collected for EDX (EDS).

3.1.14 *X-ray detector*, *n*—also known as EDX (EDS) system.

4. Description of Equipment

4.1 The principles of the electron system of the scanning electron microscope, the interactions of the electron beam and the specimen under examination, and the detection systems used for the examination are based on concepts that need understanding if the resulting image and other analytical information obtained are to be best resolved and understood. An abbreviated discussion is provided here. A more comprehensive understanding can be obtained from texts devoted to this subject **(1,2)**.³

4.1.1 SEM Optics:

4.1.1.1 An electron beam is generated in a column consisting of an electron gun and multiple electromagnetic lenses and apertures. The electron beam is generated by heating a filament so that it emits electrons. The most common filament for general SEM work is tungsten, but other filaments can be used for increased brightness. The electrons are accelerated towards the specimen by an applied potential and then focused by lenses and apertures. The energy of the electron beam influences resolution, image quality, and quantitative and qualitative X-ray microanalyses.

4.1.1.2 The electron beam is finely focused through electromagnetic lenses and apertures. A smaller beam size improves resolution, but decreases signal intensity.

4.1.1.3 Electron systems operate under vacuum. Specimens should be prepared to minimize alteration or damage when they are exposed to the vacuum (See 5.1.4). Variable pressure scanning electron microscopes, low vacuum scanning electron microscopes (LVSEM), and environmental scanning electron microscopes (ESEM) permit the examination of samples containing some moisture under low vacuum. The ESEM also allows analysis of organic materials. Even in an ESEM, however, some drying occurs.

4.1.2 Signal Generation and Detection:

4.1.2.1 The interaction of the electron beam with the sample generates several types of signals that can be utilized for imaging and X-ray microanalysis. The intensities of these signals are measured by detectors. The signals allow the examination and determination of properties such as surface topography, elemental composition, and spatial distribution of components. Signal intensities are generally used to provide an image on a screen.

4.1.2.2 The signals that are produced when the electron beam strikes the specimen surface are secondary electrons (SE), backscattered electrons (BSE), and X-rays.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ The boldface numbers in parentheses refer to a list of references at the end of this standard.

4.1.2.3 To generate an image, the electron beam is moved repeatedly across the specimen to form a raster. The magnification is the ratio between the size of the raster and that of the screen image.

4.1.2.4 Images produced by secondary electrons are most commonly utilized for topographical imaging. The SE intensity depends mainly on the angles between the electron beam and the specimen surface and between the specimen surface and the detector. The SE intensity is relatively insensitive to the specimen composition.

4.1.2.5 Images produced by backscattered electrons are often used for elemental contrast imaging. The BSE image is useful for identifying different chemical constituents in concrete. The BSE intensity depends on the average atomic number and density of each phase. The BSE intensity also depends on the angles between the electron beam and the specimen surface and between the specimen surface and the detector. Therefore, some BSE detectors can be manipulated to observe the sample topography.

4.1.2.6 The interaction of the electron beam with atoms in the sample produces characteristic X-rays having energies and wavelengths unique to atoms. Chemical analysis (or microanalysis) is performed using an X-ray spectrometer that measures the energies and intensities of the X-rays. The intensities of X-rays depend upon many factors, including electron beam currents and accelerating voltages, as well as chemical composition of the specimen interacting with the electron beam.

4.1.2.7 One important parameter for image quality is the working distance, the distance between specimen surface and the point where the electron beam exits the electron optics. Small working distances maximize BSE collection efficiency and improve the image resolution. Long working distances improve image depth of field for topographical images but decrease image resolution. The working distance generally must be within a predetermined range to perform X-ray microanalysis.

5. Materials and Features

5.1 Important microstructural features include the size and shape of individual constituents (including pores), the spatial relationships between these constituents (what constituents are touching or associated with each other), and the volume fraction of each constituent. Constituents are described in more detail by Taylor (3).

5.1.1 In order to study these microstructural features, it is necessary to recognize the individual phases which are usually recognized by their size, shape, association, backscatter intensity and elemental composition (See Note 1 for examples). These characteristics may sometimes be insufficient to conclusively identify a phase, or to differentiate between two phases, such as chert and quartz (See Terminology C125). In this case, other techniques must be used, such as XRD or polarizing light microscopy. Additional information can be found in Practice C856/C856M.

5.1.2 *Concrete*—Hardened concrete consists of aggregate, hydration products of pozzolanic and cementitious materials, residual cement particles, capillary pores and voids. Some

concrete may also contain supplementary cementitious materials, organic, inorganic, and metallic fibers, and entrained air voids.

5.1.3 *Cement Paste*—The cement paste contains residual cement, frequently supplementary pozzolanic and cementitious materials, and various hydration products that together have a complex and porous microstructure. The paste is initially a mixture of individual grains of cementitious materials and water, and may also contain chemical admixtures. Over time, hydration reactions consume the cement and produce various hydration products, some of which grow on the surface of cement grains, while progressively filling the initial water-filled space.

5.1.3.1 Residual portland cement particles appear dense and angular to subangular. Alite usually has at least one crystal face while belite is usually rounded and sometimes striated. In a BSE image, residual portland cement particles occur as relatively bright objects in a matrix of gray cement hydration products.

5.1.3.2 Calcium-silicate-hydrate is the major hydration product of portland cement and is usually amorphous or very poorly crystalline. Its morphology varies depending on the calcium to silica ratio, water to cementitious materials ratio, curing conditions, degree of cement hydration, and chemical admixtures. At high magnifications, the morphology of calcium-silicate-hydrate varies from very fine fibrous growths, to sheet-like units, to irregular massive grains.

5.1.3.3 Portlandite (calcium hydroxide) is a major phase of cement hydration and occurs in variable sizes and shapes including platy hexagonal crystals and sheet-like masses, depending on the orientation. Calcium hydroxide is normally observed throughout the cement paste and sometimes develops along paste-aggregate interfaces. It also sometimes occurs as secondary deposits in voids and cracks.

5.1.3.4 Ettringite is a primary product of the reactions between calcium aluminates and the sulfate phases in cement. It has a characteristic acicular shape. Ettringite often also appears as a secondary deposit. Secondary deposits of ettringite are commonly found in voids and cracks. X-ray microanalysis is sometimes required for its identification. A compound that has similar morphology is thaumasite (See 5.1.7 on secondary deposits). These two compounds can be distinguished by elemental analysis. In polished sections (See 6.1) ettringite may sometimes appear microcrystalline and high magnification (for example, 50,000X) may be needed to see individual crystals. In some cases ettringite crystals may be too small to be identified.

5.1.3.5 Calcium monosulfoaluminate usually forms platy crystals. Elemental analysis (EDX or EDS) may be required for its identification.

5.1.4 *Aggregates*—Descriptive Nomenclature C294 and Guide C295/C295M outlines methods and information relevant to the identification and classification of aggregates. Microstructural features of individual constituents within the aggregate can be studied using BSE images of polished specimens. The elemental compositions of aggregates can be determined using SEM with EDX (EDS) and sometimes the generic rock type (such as limestone, sandstone, dolomite, and granite) may be inferred. Microstructural properties such as the