

SAMPLE PREPARATION TECHNIQUES IN PETROGRAPHIC EXAMINATIONS OF CONSTRUCTION MATERIALS: A STATE-OF-THE-ART REVIEW¹

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ABSTRACT

Appropriate preparation of a sample is an important step in petrographic examination. Over-preparation, inadequate preparation, or inappropriate preparation can reduce or destroy important information, or may provide wrong information. A state-of-the-art review of various sample preparation techniques for light and electron microscopy is provided. Techniques described are: cleaning, fresh fracturing, oil-immersion mount preparation, encapsulation, vacuum impregnation, sectioning, lapping, grinding, polishing, etching, staining, thin sectioning, fluorescent thin section preparation, half section preparation, staining techniques for rapid identification of alkali-silica reaction gel, sample preparation for air-void analysis of hardened concrete by conventional and image analyses methods, conductive coating for examination in SEM, ultra-thin-sectioning, sample preparation for x-ray diffraction, and a few modern techniques such as cryo-SEM, ESEM, and x-ray microscopy.

KEYWORDS: Petrography, Sample Preparation, Sectioning, Grinding, Polishing, and Microscopy.

INTRODUCTION

Petrography (Greek *petra* - rock, *graphic* - picture) is the 150-year-old discipline of geology that deals with the description and classification of terrestrial (igneous, sedimentary, and metamorphic) and extraterrestrial (e.g., lunar, meteoritic) rocks including the made-made rock (concrete), and various construction materials manufactured by using natural materials. Petrography encompasses microscopical examinations at the nucleus and various supplementary chemical and physical testing needed for detailed description (type, composition, texture, mineralogy, and microstructure) and classification of rocks. Since the mid-nineteenth century, petrography has been applied in the analyses of various construction materials for their characterization, quality assurance, and failure investigation.

This article provides a concise and yet comprehensive review of various sample preparation techniques practiced in petrographic examinations of construction materials. Detailed descriptions of individual techniques, however, are beyond of scope of this article and can be

¹ Dedicated to late Wase U. Ahmed for his significant contributions in sample preparation techniques for almost four decades.

found elsewhere e.g., Hutchinson [1], St. John et al. [2], Ahmed [3-6], ASTM standards C 295, C 457, C 856, and C 1324 [7-10], French [11], Mielenz [12, 13], Walker [14], Campbell [15], Detwiler et al. [16], and Buehler Sum-Met [17]. The present review is mainly adapted from these above references, along with the author's own experience on various sample preparation tips, and several manufacturers' product literature.

MATERIALS AND SAMPLING

Petrography, as the name implies, is a pictorial-based descriptive science that follows the principle that a picture is worth a thousand words. Petrography: (a) provides detailed compositional, textural, mineralogical, and microstructural properties of a material including its detailed characterization and quality assurance, and (b) diagnoses evidences and causes of improper behavior, poor performance, or failure to provide satisfactory performance. Since photographs provide an excellent documentation of the appearance and condition of a material, samples received for petrographic examinations should first be adequately photographed, described in detail, and properly documented prior to the subsequent 'destructive' sample preparations steps for microscopical examinations. Samples, as received, should be adequately representative of the material in question and for the purpose of the examination. Locations of the samples in the field should be adequately photographed (or recorded with a video camera) and provided with the samples.

The sampling strategy should depend on the type of the material and the purpose of the examination. If it is for quality assurance, or simply for materials characterization, any suitable size, number, or amount representative of the material is appropriate. If it is for failure investigation of a structure, sampling should be done after thorough documentation of the location and extent of deterioration. Depending on the circumstances, samples could come from the deteriorated areas alone (e.g., during investigation of chemical, fire, or frost attacks in concrete), or from both deteriorated and relatively sound areas for comparative examinations (e.g., during investigation of scaling, cracking, and other surface distress). A thorough field investigation is helpful not only to visualize the overall condition in place, but also to locate the deteriorated areas, mark the locations for sampling from the sound and deteriorated areas, document the environmental conditions, and observe the extent and severity of the deterioration.

Construction materials amenable to petrographic examinations include: (a) whole or crushed clinker samples and raw feeds; (b) pozzolanic and cementitious materials including lime, gypsum, portland cement, blended cement, high alumina cement, expansive cement, masonry cement, fly ash, slag, silica fume, metakaolin, natural pozzolan, dry pre-packaged cementitious materials; (c) natural sand, gravel, crushed stone, lightweight, heavyweight, and other synthetic or manufactured aggregates; (d) portland cement based hardened concrete, mortar, grout, and plaster; (e) high alumina cement based products; (f) carbon, steel, glass, and organic fibers used in concrete; (g) clay, stone, or concrete masonry units and various masonry mortars (lime mortar, portland cement lime mortar, and masonry cement mortar); (h) lime and gypsum based products; (i) natural stone, dimension stone, and cast stone; (j) ceramic or vinyl tiles and various other resilient floor coverings and gypsum or portland cement based floor underlayments, and (k)

regolith, soils, and other subbase materials. Due to the opaque, non-transparent nature, asphaltic materials (except aggregates in asphaltic concrete) have limited applications in petrography.

A subset of the laboratory sample selected for a specific sample preparation (e.g., grinding, polishing, or thin sectioning) must be representative of the entire sample to be examined. Following is a brief description of field sampling strategies for various materials.

Clinker - In clinker microscopy, various authors practice different sampling preferences (e.g., see Campbell [15] for details). Usually, a representative amount is collected at regular intervals from the kiln during clinker production, mixed, quartered down to smaller size, sometimes crushed, and used for sample preparation. A grab sample is preferable to composite samples. Various sample preparation steps including sectioning, grinding, polishing, staining, etching, thin sectioning, and half sectioning described in this article are common in the microscopy of clinkers and raw feeds.

Cement - For lime, gypsum, portland cement, blended cement, high alumina cement, pozzolans, and other special cementitious materials, a representative amount is selected from the lot to be analyzed, which upon further quartering is reduced down to a manageable amount for appropriate sample preparation. Various modes of petrographic examinations of cements and pozzolans include: (a) immersion of a small amount transferred by a needle to a refractive index oil on a clean glass slide for examination of individual grains in a petrographic microscope (oil-immersion mounts), (b) a small amount of grains sprayed on an epoxy-coated frosted glass slide for grain thin section examination in a petrographic microscope, (c) approximately 10 grams of the representative sample is encapsulated with epoxy as a binder in a castable mold to be used for thin sectioning and examination in a petrographic microscope, (d) polished sectioning for scanning electron microscopical work, and (e) a small amount of bulk sample (~10 grams) selected by quartering for x-ray diffraction or chemical analysis.

Aggregate - ASTM C 295 recommends various methods of field sampling of aggregates, such as: (a) drilled cores from undeveloped quarries, (b) 100 pounds or 300 pieces (whichever is larger) of each size of crushed stone from operating quarries and operating sand and gravel deposits with stock piles of materials produced, (c) at least 4 pounds of material (each piece is greater than 1 pound) from each stratum or bed or a drilled core from the exposed faces of non-producing quarries where stock piles are not available; and (d) at least 50 to 400 pounds (amount should increase with sieve size) of material from test pits dug in undeveloped gravel and sand deposits. During petrographic examinations of crushed stone, natural gravel, and sand, at least 150 particles of each sieve fraction should be examined microscopically. Various modes of aggregate examination are: (a) sieve analysis of a representative portion selected by coning and quartering of the total amount received (the amount selected should provide at least 150 particles in each sieve); (b) examinations of hand specimen, fresh fracture, and lapped sections of grains in core samples or in each sieve fraction of aggregates in a low-power stereomicroscope; (c) examinations of oil-immersion mount, ground section, thin sections, and grain thin sections in a petrographic microscope; and (d) x-ray diffraction.

Concrete - For hardened concrete, either drilled cores or sawn sections are commonly collected. Detailed coring techniques are described in the US Department of Interior *Concrete*

Manual [18]. Strategies of condition survey and sampling concrete in the field are described in ACI Committee 201[19], ASTM C 823 [20], and C 856 [9]. Water-cooled diamond-bit coring using a 1-in. to as large as 12-in. diameter drill bit is common for petrographic sampling in the field; a larger diameter drill is preferable for obtaining a cracked, fragmented or disintegrated concrete. Dry cut diamond or tungsten carbide bits, though generate considerable heat during drilling due to the lack of cooling, is sometimes preferred where leaching of concrete by cooling water is unacceptable (e.g., for chloride analysis). The diameter of the core should be at least three times the nominal maximum size of the aggregate. The length or depth should be adequate to include all the layers of interest including the crucial exposed (sometimes altered) layer, the near-surface transitional zone, and the interior bulk concrete. A 3-in. (75 mm), or preferably 4-in. (100 mm) diameter core is most common. For deteriorated concrete or mass concrete containing larger aggregates, larger diameter cores are preferred for better recovery and representation (possible damage to reinforcing steels during drilling, however, may restrict the core diameter). Sometimes, a rebar-location (pachometer) survey is done in a heavily reinforced concrete to detect the locations of steel in concrete and avoid them during drilling. Rigid clamping of the drilling machine to the concrete surface provides a good core with minimum damage. Wet drilling, use of a good drill bit, less vibration of the bit during drilling, the in-situ condition of the sample that is being drilled - all these factors control the ultimate condition of the core recovered.

The number of samples should be adequate and representative of the field conditions. ASTM C 856 states, “the minimum number of samples should amount to at least one core for each mixture or condition or category of concrete”. If a particular type of deterioration is observed in the field (e.g., cracking, scaling, surface delamination, efflorescence, alkali-silica reaction gel exudation, corrosion, staining, chemical attack, etc.), at least one to three cores should be taken from each of the severely deteriorated, moderately deteriorated, and sound areas for a comparative study. Loose, broken, spalled samples from around the structure, which is exposed to prolonged and considerable atmospheric weathering to mask or complicate the original in-situ deterioration of concrete should be avoided unless they provide any specific information (e.g., loose scales on a scaled concrete slab, or efflorescence deposits on the old concrete surface). Concrete debris, collected in a coring locale may retain deposits washed away by the drilling and could be used for microcopy, x-ray diffraction, or chemical analysis.

Masonry - For masonry units, at least one full size unit or multiple units encompassing all the features of interest should be collected. For mortar sampling, multiple pieces are needed from the exposed face and the interior of the masonry joints from several locations. Mortar samples from bed and head joints should be carefully collected by: (a) using a hammer and chisel (without breaking the adjacent masonry units), (b) carefully sectioning along the interface between the mortar and masonry units with a hand-held dry or wet masonry saw, or (c) drilling a 1 to 3 in. diameter core including the head and/or bed joint mortar and adjacent masonry units through the wall. Sometimes an entire panel (e.g., of 1 ft² size) is saw cut from the wall for investigation of masonry units, jointing mortars, and mortar-masonry unit interfacial bonds. Dry drilling of mortar joints using a tungsten carbide tipped masonry drill collects dry powders useful for chemical analysis and x-ray diffraction. Efflorescence deposits on a masonry wall are carefully brushed or scraped off to a piece of paper and then transferred to a sealed ziploc bag.

Plaster and Gypsum products - For plaster, stone, tile, and floor underlayments, single or more preferably, multiple representative samples should be collected and the petrographer should choose appropriate pieces for sample preparation and subsequent laboratory examinations. Gypsum based plaster or floor underlayments should be collected in dry conditions and handled carefully. Portland cement plaster products (stucco), shotcrete, and swimming pool plasters are examined by the conventional methods of sample preparation described here.

Stone and Tile Products – Natural or dimension stones and samples from stone cladding are prepared by conventional petrographic techniques of thin sectioning and other methods practiced in geology. Efflorescence deposits on stone surface are examined by oil immersion mount, x-ray diffraction, and SEM. Stone, ceramic, quarry, or vinyl tile products and the associated underlying setting bed mortar, adhesive, and adjacent jointing mortars or grouts are examined in as received condition in a stereomicroscope, and in lapped section and thin section for detailed microscopical study.

The amount and selection of samples is a matter of judgment, which is based on the scale of the examination, the nature of the sample, field conditions, the depth and extent of deterioration or of the concerned area, experience, and the project budget.

Surface Area to be Examined - The sample surface area of examination required for adequate characterization of a constituent increases significantly with the size of the constituent of interest. For example, a representative section necessary for adequate characterization of aggregates in a concrete is significantly larger than the usual area required for air void characterization (e.g., at least 77 cm.² for characterizing < 1mm size air voids in a concrete containing 25 mm size aggregate). The area of examination in a scanning electron microscope is significantly less than that covered in a light optical microscope; therefore, information obtained from electron microscopy should be carefully interpreted to assure proper representation of the entire area of study. For example, a set of 10 usual SEM micrographs taken in various places on a 1 sq. cm. area of a cement paste sample surface at a 10,000X provides a sampling of only 0.001% of the surface of that specimen; in a heterogeneous sample as concrete, such a limited sampling may question proper representation of the overall microstructure (Diamond [21]). A small 27 × 47 mm thin section may be adequate for characterization of cement, ground clinker, sand, or plaster of homogeneous composition but multiple large area thin sections (each at least 50 × 75 mm in size) are sometime necessary for better characterization of a heterogeneous material such as a deteriorated concrete. The size of the component of interest and the degree of homogeneity of the material both determine the area of examination.

Samples collected in the field should be properly located, labeled, its field orientation should be marked, and the sample should be wrapped in plastic bags or carefully protected to preserve the on site moisture and other conditions. Information about the location of the sample in the structure and photographs of the field condition should also be supplied. Spalled, weathered, or broken chunks, scales, or small pieces of concrete laying around the structure should either be avoided or should be carefully considered as they may provide only limited information; after dislodgement from the structure loose pieces may have been subjected to weathering and mechanical disintegration, which can erase or alter any in-situ evidence of actual deterioration in the structure. In many cases, a thorough field investigation directly by the

petrographer provides many valuable information about the condition of concrete in the field, structural integrity, locations, severity and extent of deterioration, and may even the cause(s) of failure or poor performance.

A detailed communication between the petrographer and the engineer/architect about the project background and service history is necessary. A field survey, examinations of field photographs, review of the results of previous testing, and information about the original mix design(s) are helpful to determine the total number and locations of representative samples to be taken for examination. Adequate communications between client and petrographer should ensure that: (a) the samples selected for petrographic examinations are representative of the structural unit and field conditions, (b) sample locations were carefully selected considering the variations in structural units, environmental conditions, extent and intensity of damage, and any variation in the type of concretes, and (c) adequate samples are provided for obtaining sufficient data for a reasonable interpretation. Without these preliminary requirements, and without adequate and proper sampling, the data obtained, the interpretation, and conclusion drawn from petrographic examination can be inadequate, and even highly misleading.

PRELIMINARY EXAMINATIONS

Since any sample preparation is essentially a destructive procedure, information susceptible to damage or alteration during sample preparation should be recorded prior to any such step. The sample should be adequately photographed in as-received condition, logged-in, and detailed preliminary investigation should be made including:

- Background information of the project (client name and address; project name, number, and location; date of sampling; geographic location of the structure; sample location in the structure; detailed photographs of field condition including the sample location; sampling procedure; drawing showing sample locations, field and environmental conditions at the sample locations, mix design, weather conditions during concrete or masonry placement and subsequent periods, age of the structure; service history, results of other tests performed on the sample or on other samples from the project; nature and extent of deterioration, when was it first noticed, and any other information that may seem relevant).
- Dimensions, integrity, appearance, condition (a broken piece or total number of pieces, core, saw-cut section, powdery, dusty, fragile, hard, intact, etc.) of the material including any unusual features.
- Nature of the exposed surfaces and any surface deposits on the samples from the field.
- Nature of the fresh fractured interior surface of solid material.
- Size, location, and condition of embedding materials (steel or fiber reinforcement).
- Size, location, and distribution of voids, cracks, joints, sealants, etc.
- Properties of basic ingredients (such as aggregate, paste, and air in concrete)
- Signs of chemical and physical deterioration.
- Variations in materials or properties in the sample.
- Nature of interfaces between multiple layers in a composite sample.

Petrographer should observe the sample as thoroughly as possible and gather sufficient information regarding the condition of sample in terms of its integrity, composition, fabric, and imprints of any external or internal deterioration processes in the field. Preliminary laboratory examinations should be made visually, with the help of a hand (magnifying) lens, and with a low power stereomicroscope.

During the visual and low-power stereomicroscopical examinations, multiple sample photographs should be taken by using a 35-mm color camera, or a digital camera with at least 3-mega pixel resolutions, and a flatbed scanner. Preliminary examinations should determine the areas of interest in the samples and mark locations for subsequent sample preparation steps.

SAMPLE PREPARATION TECHNIQUES

The primary purpose of sample preparation is to reveal the *in-situ* microstructure and composition of the sample. Care must be exercised in all preparation stages to avoid any loss or alteration of the original composition and microstructure. Appropriate sample preparation techniques are, therefore, crucial for obtaining maximum and accurate information. Ability to observe various minute details in the microstructure depends strongly upon the sample preparation procedures. Inadequate preparation may not reveal the details clearly whereas over-preparation may lose much of the *in-situ* information, which is almost always crucial for interpretation; both can introduce preparation-artifacts that are not related to the original material or problem. Depending on the nature and condition of a sample, various preparation methods such as epoxy impregnation, sectioning, grinding, polishing, staining, etching, thin sectioning, coating, pulverizing, etc. are followed. Brief descriptions of these procedures, mainly from the references mentioned in the introduction, are given below.

(1) Cleaning

Cleaning of a hardened concrete core in running tap water removes the dust, dirt, and muddy coating of drilling dust that are sometimes present on the cylindrical surface due to wet drilling. Such cleaning can highlight: (a) the pristine condition of the sample after removing the artifacts from coring; (b) the type of aggregates and their grading and distribution; (b) large voids, cracks, joints, and reinforcements; (c) variation in color, density, water absorption, and other properties through depth or in a single location in the sample, which can indicate variation in the concrete composition; and (d) different layers in a composite core. Wetting is sometimes helpful to highlight many fine or wide cracks in a concrete core; cracks become prominent during differential drying of concrete between the cracked and uncracked portions. Exudations of white alkali-silica reaction gel through the cracks are sometimes distinct when a core dries out from its moist condition and contains alkali-silica reactive aggregates. Drying after cleaning of concrete and masonry units sometimes reveals soft, white, powdery efflorescence deposits on the air-dried surface, which can be characterized by examinations of oil-immersion mounts. Thorough cleaning of a sample is essential within and between various sample preparations steps to wash the debris generate in each step (e.g., cleaning in running water or in ultrasonic cleaner in between the steps of sectioning, grinding, polishing, thin sectioning). Soft, fragile, and water-sensitive materials (e.g., clinker, cement, gypsum based materials), however, should not be

cleaned in water, for which non-aqueous isopropyl alcohol, acetone (Ahmed, [3]), 1,1,1, trichloroethane (Walker [14]), or other non-aqueous media can be used. Cleaning, however, should be done carefully, after knowing its effect on the material being examined, subsequent possible alterations, and its effects on the interpretation of the microstructure.

(2) Fresh Fracturing

Obtaining a fresh fracture surface of a small piece of hardened concrete, mortar, masonry unit or other solid material by hammering from a larger piece and examining that fresh exposed surface at progressively higher magnifications using a hand lens, low-power reflected-light microscope stereomicroscope, and scanning electron microscope can reveal a wealth of information including: (a) physical, compositional, mineralogical, and textural properties of paste; (b) types of aggregates from mineralogy and texture; (c) bond between aggregates and paste; (c) corrosion of reinforcing steel and extension of corrosion products into the paste; (d) coatings, reaction rims, and weathering rims around aggregates; (e) secondary deposits in voids and cracks; (f) depth of carbonation; (g) signs and depth of chemical alterations in concrete by atmospheric alterations and weathering; (h) signs and depths of chemical attacks in a concrete by migration of deleterious chemicals; (i) air content and air-void system in concrete, mortar, grout, and plaster; (j) thickness of different layers in a multi-layered composite system; (k) selection of areas of interest to be further examined for compositional analysis by oil-immersion mounts; etc.

Examination of fresh fracture surfaces of concrete in a stereomicroscope is an important mode of examination in concrete petrography, which, with increasing experience, can provide a great deal of information. Fresh fracture surfaces provide information about the condition and composition of pristine, unaltered interior of a sample, which should be examined immediately after fracturing before any extended atmospheric exposure. Fresh fracturing of an already cracked concrete, however, may open up the preexisting cracks and may not provide information of unaltered interior; many times secondary deposits precipitate in cracks in an old concrete and become exposed by fresh fracturing, which, then, could be examined by oil-immersion mount or SEM.

(3) Immersion Mounts Preparation

Oil-Immersion Mount - Preparation of an oil-immersion mount involves: (a) observation of a fresh fracture surface of a sample or of the original sample as received in a low-power stereomicroscope; (b) selection of an area of interest (e.g., in hardened concrete an area of bulk paste apart from the aggregates, air voids, and carbonated or altered areas; or a secondary deposit in an air void in a concrete); (c) collection of a tiny amount of sample from the area by scratching the area with a sharp-pointed needle and carefully transferring that powder onto a clean glass slide; (d) placing a small cover glass on top of the powder to prevent loss; (e) immersing the powder in an oil of known refractive index by applying the oil along the edges of the cover glass (oil is drawn under cover glass by capillary action); (f) spreading the powder uniformly in the oil and breaking thick, agglomerated portions with a rubber such as the eraser in a pencil; (g) observing individual grains in the powder in a petrographic microscope and characterizing the composition and mineralogy of the grains from their relevant optical

properties including size, shape, relief, color, interference color, birefringence, refractive index, extinction angle, pleochroism, and dispersion staining.

Particle mounts on removable resins or on permanent thin epoxy films – Campbell [15] mentioned examination of fine clinker grains and cement by immersing small powders on a molten Hyrax or Meltmount resin (refractive index 1.70) on a glass slide and placing a cover glass before the hardening of the resin. Fine particles (74 to 149- μm^1) sprinkled on a glass slide coated with a thin film of epoxy (where only the bases of the particles are permanently mounted in the epoxy) can be examined in a series of refractive index oils with intermediate washings of the oils and cover glasses by gentle streams of acetone or xylene, and rinse with isopropyl alcohol.

Applications - Oil-immersion mounts are helpful for rapid identifications of: (a) cement and clinker phases including their relative abundances and various optical properties; (b) clinker raw feeds; (c) secondary deposits in concrete; (d) cement hydration products, residual cement particles, degree of cement hydration in the paste, and the type of cement used; (e) fly ash, ground granulated blast-furnace slag, other pozzolanic and cementitious materials, and microfillers in paste; (f) the presence of hydrated lime, portland cement, and limestone fines in mortars and from that the types of mortars (i.e., lime mortar, portland cement-lime mortar, masonry cement mortar); (g) efflorescence deposits on masonry walls (calcium and alkali sulfates, carbonates, and hydrates); (h) lime and gypsum based products, and various forms of gypsum (gypsum, alpha hemihydrate, beta hemihydrate, soluble anhydrite, and insoluble anhydrite) and lime (calcitic, dolomitic, hydraulic); (i) various potentially expansive materials including alkali-silica reaction gel (e.g., ettringite and gypsum in a concrete exposed to surface attack; epepizite or epizet and brucite in concrete cracked by delayed hydration of free lime and magnesia, respectively); (j) types and mineralogy of aggregates; and (k) depending on the situation and the depth of the examination, the overall type, composition, and characterization of the material, and the cause of deterioration, if any.

(4) Encapsulation and Vacuum Impregnation

Purpose - Encapsulation and/or impregnation of a sample with a resin, especially if it is soft, powdery, cracked, brittle, friable, or broken is helpful to: (a) fill the voids, pores and cracks; (b) improve the overall integrity and ease of handling; (c) preserve the original microstructure and distribution of components and edges in the sample; (d) keep the detached, de-bonded, fragmented portions adhered to the rest of the sample; and (e) prepare a solid mass of the original fragmented or powdery sample for sectioning, grinding, thin-sectioning, or polishing. Encapsulation indicates immersion of the sample within a resin in a mold to improve the external integrity of brittle, friable materials and for better handling for subsequent sampling steps, whereas impregnation indicates injecting or penetrating a liquid resin into a porous sample to improve its internal as well as external integrity. Epoxy-based resins are best for both encapsulation and impregnation.

Encapsulation Methods and Resins - Unlike the common *compression mounting method* for encapsulating metallurgical samples at high temperature and pressure in a press (which uses

¹ μm = micron = 0.001 millimeter.

phenolic or epoxy-based thermosetting resins, or methyl methacrylate-based thermoplastic resin), *castable mounting method* is more common for encapsulating both metallurgical and construction materials, where the material is mounted without using a press in a plastic or metal mold by using an acrylic or epoxy-based resin and is cured at relatively low temperatures (except for the acrylic and some fast-cured epoxy resins, which can generate heat by exothermic polymerization reactions as high as the heat generated in the compression mounting). The term *cold mounting* is sometimes used for the castable mounting method due to the absence of external heat and pressure for mounting, however, the term “cold” can be a misnomer for mounting heat-sensitive materials in an acrylic resin, or in a fast-curing epoxy resin, or in a large-volume epoxy medium, or in an insulating mounting mold - all of which can generate high exotherms during resin polymerization.

Acrylic resins: (a) cure quickly, (b) have low cost, (c) are too viscous for impregnation, (d) can generate heat in excess of 100°C by polymerization, (e) usually do not bond to the sample, (f) have classic problems with high shrinkage during curing, which creates shrinkage gaps between the resin and sample, and (g) have poor edge retentions.

Epoxy resins, although more expensive than acrylics, are more commonly used because: (a) epoxy will physically adhere to the sample with good bond and eliminate shrinkage gap, (b) epoxy will not react with the sample and with other solvents, etchants, chemicals, and oil lubricants used in sample preparation, (c) epoxy can be used at room temperature or at a temperature of up to 60°C and can be cured within 9 hours (the lower the viscosity of epoxy at room temperature, the longer its curing time; lowering the viscosity by slight warming, however, fastens curing), (d) epoxy is hard enough to produce flat surface during grinding and polishing, (e) sets relatively quickly and more so by slight warming, (f) it has a very low setting shrinkage, (g) it provides excellent edge retention of samples (especially for metallurgical samples and thin sections of concrete including surface features) due to better adherence to sample and low shrinkage, and (h) due to low viscosity, epoxy can be drawn into pores and cracks by vacuum impregnation. The curing time of epoxy varies from a few minutes to several hours, depending on the type of epoxy used and the ambient temperature. A slight warming can accelerate the polymeric reactions and shorten the curing time (however, any fast-cure epoxy will have higher polymerization exotherms). Castable resins are sensitive to shelf life, which can be extended by keeping them in a refrigerator.

Dyes - Different types of dyes or colorants are sometimes mixed with epoxy to highlight the cracks, voids, and pore spaces in sample while observing in the plane-polarized light or in ultraviolet-light mode in a petrographic microscope. Various types of dyes are: (a) a dry pigment thoroughly mixed with epoxy (heating epoxy at 100°C with the dye helps thorough mixing), (b) pre-mixed red, green, or blue coloring paste thoroughly dissolved in epoxy at 100°C, (c) dyes soluble in epoxy resin, which are superior to pigments and impart a uniform color to the epoxy, and (d) fluorescent dye mixed in epoxy, which sometime fades with time in exposure to light. A fluorescent dye mixed alcohol applied to a lapped surface of concrete can highlight the micro and macro cracks (by greater absorption of dye in the cracks) during exposure in a short wavelength ultraviolet light. Table 1 provides a list of various castable resins and dyes. The amount of the colorant or fluorescent dye powder commonly added to the epoxy resin is 1 to 2 percent by mass of the resin, depending on the desired intensity of color.

Encapsulation Procedure - For encapsulation, a small sample (with a small paper of sample identification) is placed in a 1 to 1.25-in. diameter plastic (polyethylene, reusable PVC pipe sections), flexible (silicon mold, or Buehler's reusable EPDM cup mold), or metal (brass, copper pipe sections) ring form, cup, or other (square or rectangular-shaped) cold mounting container coated with a thin film of mold release agent (such as petroleum jelly, or Buehler's release agent). The sample should be dry as moisture in the sample can interfere with polymerization of epoxy and affects adhesion. Freshly mixed, clear, or dyed epoxy (at correct resin-to-hardener ratio) is poured on and around the sample to immerse it completely in the epoxy, the epoxy is allowed to cure, and eventually the sample in hardened epoxy is removed from the mold, properly identified, and sectioned. For larger samples such as a severely cracked or fragmented concrete sample, it is wrapped in an aluminum foil to form a bowl or placed in a disposable plastic or aluminum bowl, oven-dried at ~ 30-40°C to remove its free moisture; then encapsulated in epoxy. Sample should be re-identified immediately after removing it from the mounting mold.

Vacuum Impregnation - The lower the viscosity of the epoxy, the deeper its depth of penetration into the sample during impregnation, and the better its mechanical bond to the constituents.

For effective epoxy penetration, the following procedure is helpful. A thin slice of sample, approximately 5-10 mm thick, is first thoroughly dried either in an oven at 30 to 40°C for 12-16 hours to a constant mass, or, by immersing it in alcohol for removing free internal moisture by the solvent replacement method, or, by rapid freeze-drying by immersing the sample in liquid nitrogen followed by sublimating water directly to gas in a cooled chamber under vacuum (Detwiler et al. [16]). A very low-viscosity epoxy (viscosity in the order of 100 to 250 centi-poise at room temperature) should be used for impregnation (e.g., EpoThin, Epo-Tek 301, Epoxy Pack 301, see Table 1). A slight warming of resin in a slide warmer or hot plate immediately before mixing with the hardener further reduces the viscosity and provides deeper penetration into the sample). Epoxy should be poured in the sample in the vacuum; alternately, a container having the dry sample already immersed in an epoxy medium could be placed in a bell jar and slowly evacuate with a vacuum pump until bubbling in the epoxy coat of sample due to air withdrawal ceases completely. Drawing a vacuum on epoxy causes it first to evolve its entrapped air, then eventually to "boil" forming additional air. Breaking and restoring the vacuum several times will help to expel all trapped air from inside the sample and to allow the air pressure to force the epoxy into all voids, cracks and open spaces in the sample. Curing time generally increases with decreasing epoxy viscosity (e.g., Epo-Thin cures in 9 hours at room temperature), which can be accelerated by slightly warming the epoxy and sample in an oven or a hot plate at 40-45°C. Table 1 lists various epoxy resins and their mix proportions, curing temperature, and curing time.

Beside viscosity, the depth of epoxy penetration also depends on: (a) the porosity and connectivity of pores in the sample, (b) the vacuum pressure applied during impregnation (should be at 0.05 bar maximum pressure for 10-20 minutes), and (c) the degree of drying of the sample. The purpose of impregnation in vacuum is to remove air out of the voids, pores, and cracks so that the epoxy can easily flow into these open spaces. Also, vacuum impregnation

removes air bubbles within the epoxy, which usually prevent good bonding. In the absence of such sealing with epoxy, pores and margins of cracks or air voids may be enlarged during sectioning, grinding, or polishing operations and may entrap various foreign materials like grinding and polishing abrasives, solvents and stain-producing etchants. Impregnation with a colored or fluorescent dye mixed low-viscosity epoxy can highlight pores, voids, and cracks. The effective epoxy-impregnation procedure depends on thorough pre-drying of the sample to a constant mass so that all free water in the pores, cracks, and voids are removed to make room for the epoxy. Drying should be restricted to a temperature of around 30-40°C to prevent excessive shrinkage and cracking of the concrete or mortar by dehydration of cement hydration products. Sample is commonly dried overnight in an oven or on a slide warmer or hot plate at 30-40°C. To check the tendency of paste to crack during drying, the oven-dried sample could be compared with the room-temperature-dried sample. High curing temperatures can induce strain birefringence in the epoxy. Drying a field concrete at > 60°C can cause dehydration of ettringite and microcracking due to temperature gradients. For fragmented and powdered samples like mortar fragments, cement, aggregate, and ground clinker successive stages of oven drying, vacuum impregnation and encapsulation into blocks of epoxy resin and hardening the resin under heat (~50°C) are not uncommon before sectioning, grinding, polishing, and thin sectioning operations.

Stutzman and Clifton [22] described a three-stage procedure for impregnation of very low viscosity epoxy in cement paste, mortar, and concrete without drying (to avoid drying shrinkage cracking), by solvent replacement, first by replacing the pore solution with ethanol, and then by replacing ethanol with a low-viscosity epoxy, which is then cured at room temperature.

Removable Mounting Medium - If the bonding medium needs to be removed after the examinations, instead of epoxy, which permanently hardens in the sample, other types of temporary mounting media can be used. Roberts and Scali [23] suggested a 1:5 solution of commercially available colorless nylon fingernail hardener in methanol, which can be later removed by soaking in an appropriate solvent. ASTM C 457 mentions use of Carnauba wax that must be used with safety precautions to prevent accidental explosion during the heating over the flash point to remove the excess wax. Alternately, some mounting epoxy can be dissolved in methylene chloride (carcinogenic), or softened by dipping it in boiling glycerin for 1 to 2 hours (Ahmed [6]).

(5) Sectioning

Purposes – Sectioning helps to: (a) obtain a smaller, manageable sized specimen from the parent material, which helps to keep the rest of the sample for other analysis; (b) expose the internal surface of interest for grinding; and (c) reduce the thickness of the sample to minimize time for subsequent grinding as in thin sectioning. Concrete samples should be sectioned at longitudinal and transverse directions relative to their orientations in the field (i.e., direction of placement) to reveal the microstructural details in all orientations including the most crucial near-surface zone. Sectioning can be the most damage-producing step (especially for a brittle or poorly consolidated material) in the entire process of sample preparation. Proper selections of saw and blade are, therefore, crucial to minimize the surface damage. More damage will increase the grinding time, which, in turn, can increase the relief. Minimizing surface damage during sectioning is, therefore, beneficial for subsequent grinding.

Large Saws – Various abrasive-wheel or diamond-bonded sectioning machines containing blades from 8-10 in. to 18-24 in. in diameter and using water or oil as coolant are used for sectioning large samples. Wet cutting produces a smooth, sectioned surface and prevents excessive surface damage from overheating. An oil-cooled saw usually produces smoother sections than do the surfaces produced with a water-cooled saw (Walker [14]). Various abrasive wheels (i.e., abrasive cut-off blades, ~ 9-in. to 14-in. diameter) consisting of alumina, silicon carbide, or cubic boron nitride abrasive filler in a resin, rubber, or resin/rubbed mixed binder are common for sectioning ferrous and non-ferrous metals and minerals in abrasive cutters. Resin-bonded or metal-bonded diamond blades are most common for sectioning rocks and concrete (Buehler [17]). In order to reduce any potential damage to the sectioned surface during sectioning (especially for the brittle materials that are susceptible to grain plucking), thin abrasive or diamond blades are used for sectioning large samples. A 10-in. diameter ($\frac{3}{8}$ in. diameter arbor), good quality thin (blade thickness 0.032 to 0.045-in.), continuous rim diamond blade in a table top tile saw is suitable to cut many construction materials including concrete cores up to 3 in. in diameter. The type and thickness of the blade used, grain-size of diamond or other abrasives in the blade, the pressure applied during sectioning, blade speed and feed rate, and the coolant supply rate are the important factors, which control the ultimate smoothness of the sectioned surface. As a general rule, the quality of the surface finish is proportional to the blade thickness and abrasive size on the blade.

Precision Saws - Precision saws, as the name implies for very precise cuts, are used to section materials that are small, delicate, friable, extremely hard, or where a cut must be made as close as possible to a feature of interest, or where the cut width and material lost must be kept minimal. Precision saws use 3 to 8-in. diameter diamond wafering (ultra-thin) blades and are recommended for sectioning small samples and for thin sectioning (Ahmed [3-6]). Table 5 lists various manufacturers of precision saws. Wafering blades in precision saws are much thinner (from 0.006-in. thick for the 3-in. size to 0.035-in. thick for the 8-in. size blade) and load applied during cutting are much lesser than the abrasive cut-off blades in abrasive cutters. Consequently, less heat generates during sectioning and the sectioned surfaces have minimal deformations. Although cubic boron nitride (CBN), aluminum oxide, and SiC are used for abrasives in precision blades for metallurgical applications, diamond blades are most common for both metallurgical and petrographic sample preparations. Selection of a thin, proper diamond blade is

crucial to reduce the surface deformation. Various types of wafering blades are: bonded blades composed of inner metal core and an outer rim of metal or resin bonded abrasive; plated blades consisting of a solid metal core with diamonds nickel-coated to the rim; and diamond segmented rim or continuous rim blades. Table 5 lists various manufacturers of abrasive cutters, precision saws, and blades.

Coolant – Water is the most common coolant, which should be mixed with a corrosion resistant chemical to prevent rusting of the blade. Water-sensitive materials should be sectioned with a cutting fluid, propylene glycol, isopropyl alcohol, or more economically with a low viscosity cutting oil, or a hydraulic food-line mineral oil (e.g., baby oil or Mobil DTE FM 32, Campbell [15]). Cutting fluids should not be flammable and should not impose any health hazards in the form of vapor or aerosol, in which case proper safety precautions and ventilations should be taken in handling and disposing the fluid. Use of gloves is recommended.

The sectioned sample should be thoroughly cleaned with water, acetone, or isopropyl alcohol to remove the debris formed by sectioning, and any cutting fluid, and then air- or oven-dried prior to subsequent grinding and other preparation steps. Small samples should be cleaned in a sonic cleaner with the appropriate cleaning solution.

(6) Lapping and Grinding

Purpose - Grinding¹ and lapping remove deformations, surface irregularities, and saw marks induced during sectioning and provide a smooth, perfectly flat, and matt-finished surface. A finely ground surface is essential for examination of clinker, aggregate, concrete, and mortar in a low power reflected light microscope (stereomicroscope) and air-void analysis in hardened concrete at a magnification up to 100X.

Procedure - Grinding is usually done by using successively finer-grained abrasives in water, solvent, or oil-based carrier on a horizontal rotary grinding/lapping wheel. In transition from a coarse to the next fine grit size, the ground surface is thoroughly cleaned to remove the loose abrasives and fine particles of sample produced during coarse grinding. In the traditional approach to lapping and grinding, progressively smoother and finer ground surface with lesser sample removal is achieved by grinding with successively finer abrasives. Abrasive used for each grinding step is one or two grit sizes smaller than that used in the preceding step, which removes the surface deformation induced by the former coarser grit. The depth of surface damage decreases with the abrasive size and so does the sample removal rate. For a given size, the damage is greater for a soft material than for a hard material.

Grinding Machines - Three types of grinding machines are common:

- (a) A large, bench top or stand alone unit containing a motor-driven, 18-24-in. diameter horizontal rotary iron lapping wheel (plain, or with radial or concentric grooves), with or without condition rings as sample holding fixtures, which can receive either loose SiC/alumina abrasive powder, or fixed abrasive papers with pressure sensitive adhesive (PSA) backing, or resin and metal-bonded diamond discs with magnetic backings. Large concrete samples up to 6 × 12-in. in cross section can be lapped by this unit;

¹ The term “grinding” here should not be confused with “pulverizing” or making a powder of the material.

- (b) A single or dual-deck bench top or stand alone grinding/polishing unit housing horizontal rotary wheel(s) (usually 8 to 12 in. diameter) to accept clean, or PSA-backed grinding papers, or magnetic discs with or without single/multiple sample holding fixtures (head); a wide variety of samples can be both ground and polished by using various interchangeable magnetic plates with grinding abrasive papers or polishing cloths on the same single or dual grinding/polishing wheels; and
- (c) Micrometer-attached vertical, diamond cup wheel or plate to traverse the sample for controlled and precision grinding; this is used for grinding thin sections in thin-sectioning equipments (diamond particles embedded in cup wheels are usually 60- μm in size).

Grinding Abrasives - Abrasives used for grinding are:

- (a) Either applied as loose grains or powdered form in a premixed slurry or suspension in water, oil, or solvent, or as powders charged concurrently with water spray and applied on a solid iron lapping/grinding wheel where the abrasive particles are free to roll around as they abrade the sample surface, or
- (b) Fixed or bonded to a paper, polymeric, or cloth backing materials of various weights in the form of sheets or discs of various sizes which are attached to a horizontal rotary grinding wheel or as belts in a stationary roll (or belt) grinder, or
- (c) A series of small (8 to 12-in.) or large (18 to 24-in.) diameter, fixed, metal-bonded or resin-bonded diamond discs of various grit sizes that magnetically adhere to the grinding wheels (for the same abrasive size, a metal-bonded diamond disc removes more material faster and produces a rougher or coarser surface finish than a resin-bonded disc).

Fixed abrasives (diamond discs or abrasive papers) are generally more aggressive and remove much more material per unit time for the same abrasive size than loose abrasives and tend to produce somewhat more deformation at the surface than that noted when abrasives roll over the plate during lapping (Buehler [17]). For both types, the size of the abrasives determines the cutting rate and surface damage depth. The coarser the abrasive, the faster the sample removal rate but the greater the damage depth at the surface, and vice versa. Soft and brittle materials should grind with as fine abrasive as possible, which, though takes a longer time to remove the sectioning damage, produces less damage from grinding than a coarser abrasive. Diamond discs have a long service life but can be far more aggressive for grinding soft and sensitive materials than the SiC abrasive papers, which have relatively short service life but are better for grinding soft materials.

Common abrasive grains are: (a) silicon carbide (SiC) or aluminum oxide (Al_2O_3) with the following various ANSI/CAMI (USA) grit numbers (the corresponding median micron size of the particle size distribution is in parenthesis, Buehler [17]): 60 (268- μm), 80 (188- μm), 100 (148- μm), 120 (116- μm), 180 (78- μm), 220 (66- μm), 240 (51.8- μm), 280 (42.3- μm), 320 (34.3- μm), 360 (27.3- μm), 400 (22.1- μm), 500 (18.2- μm), 600 (14.5- μm), 800 (12.2- μm), 1000 (9.2- μm), 1200 (6.5- μm), and 3000 (3.5- μm); and (b) diamond paste or suspension applied on a grinding paper, disc, or cloth attached to a horizontal rotary wheel (10 to 15- μm size diamond paste and 9.5- μm alumina powder slurry are commonly used for the intermediate to final fine grinding operations). The Mohs hardness of alumina and SiC are 9, and diamond is 10 (corresponding Knoop hardnesses are 2100, 2300, and 8000, respectively). Due to its high hardness, diamond is the abrasive of choice for grinding hard materials such as rocks, concrete,

ceramics, and glass. Diamond abrades faster, removes more material per unit time, and produces a more consistent surface finish (usually with less relief) than alumina or SiC. SiC and alumina both occur either as loose powders or as fixed abrasive paper. Aluminum oxide crystals are more blocky than SiC crystals - the former breaks down into uniformly shaped particles. Alumina is a better choice for a scratch-free surface and for grinding a soft material than SiC. Alumina is available in hexagonal and cubic crystal forms and produced by calcination (tends to have agglomerated forms) or by sol-gel process (agglomerate-free). Deagglomerated alumina produces a better surface finish than agglomerated form of the same particle size. Methods for sizing the SiC/Al₂O₃ abrasives in the abrasive papers are by sieving for the coarsest grits, sedimentation grading for the intermediate grits (240-600), and electrical resistance method for the very fine grits. Hack-sawed, bandsawed, and other rough sections produced on a rough or thick diamond blade or abrasive cut-off wheel require coarse grinding to remove surface irregularities by using grit sizes of 60 to 100; whereas samples sectioned by using a thin, precision blade (which produces minimum surface deformation) should start grinding with grit sizes of 320 or 400.

A grinding disc of fine stainless steel mesh attached to a substrate (e.g., Buehler's Ultra-Plan disc charged with diamond slurries or sprays) is promising to produce a surface finish between grinding and coarse polishing, for rapid sample removal without producing large amounts of deformation in the sample, and to minimize surface relief, especially during the final thinning of a thin section on a glass slide from 30 to 40- μ m down to 15 to 20- μ m (Campbell [personal communication]).

Clinker, Cement, and Concrete Grinding – Sectioned whole or crushed clinker samples and encapsulated cement samples are commonly ground on a horizontal rotary wheel with successively finer sized fixed abrasive papers charged with SiC, Al₂O₃, or diamond and lubricated with a solvent (propylene glycol), or oil. Concrete samples are lapped on a larger diameter (18 to 24-in.) horizontal rotary cast iron lapping wheel charged with SiC or Al₂O₃ powder abrasives, or, more efficiently, with a series of diamond magnetic discs. Samples are either lapped by holding in hand (for larger samples) or placed inside a lapping ring on the lapping plate with weights on the samples (for samples up to 4 × 6-in. dimension). Single or dual-wheeled 8 to 10-in. diameter horizontal rotary grinding/polishing machines are also used for grinding concrete samples prior to thin sectioning, and for polishing.

Carrier – Water-sensitive and anhydrous materials are ground with a suitable low-viscosity lapping oil or other organic solvents such as ethanol, glycol, or alcohol. Good non-water-based carriers are propylene glycol, 1:1 mixture of propylene or ethylene glycol and alcohol, or a low-viscosity water-free lapping oil such as denatured kerosene mixed with 1/10th part motor oil (Walker [14]) or a hydraulic food-line mineral oil (e.g., such as the one used in commercial baby oil or Mobil's DTE FM-32, Campbell [15]). The diamond saw manufacturers sell a variety of light lapping oils. The flatness of the finely ground surface can be checked by viewing it at a low angle of incidence in a strong light or in a stereomicroscope. In an air-entrained concrete, the margins of air voids should be sharply defined after the grinding operations.

Lapping – Although in many literatures the term ‘lapping’ is used synonymously with ‘grinding’, lapping is the type of grinding where the abrasive particles are applied as loose grains and roll freely on the surface of a cast iron or plastic lapping wheel or disc. The wheel is usually charged with slurries of small amounts of SiC, alumina, or diamond.

(7) Polishing

Purpose - Polishing produces a smooth, flat, deformation-free, and scratch-free surface, which is bright, shiny, and mirror-like in appearance with sharp edges and good differentiation between the constituents. Polishing minimizes all fine surface irregularities left over during the grinding operation. A polished surface is essential for observations of stained and etched surfaces in a high-power reflected-light (metallurgical) microscope and for detailed microstructural evaluations including secondary and backscatter electron imaging and x-ray microanalysis in a scanning electron microscope. Polishing operations should not introduce “extraneous structures” such as damage on the surface, pitting, scratches, dragging out of inclusions, comet tailing, staining, or relief. Grain mounts, small finely ground sections of concrete, mortar, and other building materials, and uncovered thin sections of materials can be polished by using various polishing abrasives and lubricants on polishing cloths attached to a horizontal rotary wheel. Prolonged polishing can introduce relief or height difference due to differential rates of abrasion of soft and hard components.

Polishing Abrasives – Unlike grinding abrasives, the smallest of which are around 5- μm in size, polishing abrasives are usually from 5- μm to submicron in size. Traditional polishing abrasives are: (a) diamond paste or suspension in distilled water, oil, or in an appropriate carrier, (b) deagglomerated aluminum oxide in powder form or in suspension in distilled water, or in an organic solvent (ethylene glycol, alcohol, kerosene, glycerol), or in polishing oil, and (c) amorphous silicon dioxide in colloidal suspension. Water-based carrier is avoided in polishing water-sensitive materials. Cerium oxide, chromium oxide, magnesium oxide, or iron oxide are sometimes used for polishing specific materials (e.g., glass). Diamond pastes or suspensions containing either virgin natural diamond, or, synthetic monocrystalline, or more effective polycrystalline forms of diamonds are excellent polishing abrasives and have been used for metallurgical polishing since the late 1920s (Buehler [17]). Aqueous fine alumina powders and slurries, such as Buehler’s MicroPolish deagglomerated alumina powders and suspensions of alpha alumina (0.3- μm size) and gamma alumina (0.05- μm size) slurries (or suspensions) are good for final polishing (either in sequence or singularly) with medium nap polishing cloths. As mentioned in grinding, deagglomerated alumina produced by the sol-gel process produces a better surface polish than fine alumina abrasives of the same size produced by the traditional calcination process (which always includes some agglomeration). Colloidal amorphous silica suspension is common in metallurgical applications and produces a good surface polish in concrete (Ahmed [5]); crystallization of amorphous silica on evaporation and its precipitation on the surface, however, can introduce scratches, which can be avoided by a 10-15 second spray of water on the polishing cloth at the end of a cycle. Diamond abrasives usually produce less surface relief than other abrasives.

Polishing Cloths - A good polishing cloth should: (a) hold the abrasive media, (b) have a long life, (c) not contain any foreign material, which may cause scratches, (d) have appropriate

hardness/softness and low, medium, or high nap (fiber) depending on the polishing abrasive used, and (e) be clean of any processing chemicals (such as dye), which may react with the sample. Many cloths of different fabrics, weaves, or naps are available. Napless or low nap cloths are good for coarse polishing with diamond abrasives. Napless, low, medium, and occasionally high nap cloths are good for final polishing. A “hard” polishing cloth that does not have a nap is good for minimizing surface relief. A “soft” cloth that has a nap controls scratching and produces a better quality surface finish. Usually, successively finer sized diamond or alumina abrasives on moderately hard to hard napless or low-nap polishing cloths (e.g., Buehler’s TexMet) are used for coarse to fine polishing, and softer, submicron-sized deagglomerated alumina or colloidal silica abrasives on a soft, napped cloth (e.g., Buehler’s MicroCloth) is used for the final polishing.

Polishing Methods - *Coarse polishing* involves the use of successively finer (from 6 or 5- μm to 1- μm) diamond or alumina abrasives charged onto napless or low-nap polishing cloths. *Intermediate and fine polishing* involve the use of successively finer sub-micron-sized (0.3- μm and 0.05- μm) deagglomerated alumina or diamond abrasives on napless or low nap to medium nap polishing cloths. *Mechanical polishing* indicates procedures involving the use of polishing abrasives on cloths; the cloths may be attached to a rotating wheel or a vibratory polisher bowl; the samples may be held by hand, held mechanically in a fixture such as a conditioning ring in a roller arm, or merely confined within the polishing area [Buehler, 17]. *Electrolytic polishing*, common in metallurgical applications, involves a slow sample removal rate (1- μm per minute) and creates a slightly wavy surface, which increases the difficulty of focusing at high magnifications; the method is not common in cement and concrete polishing [17]. *Manual hand polishing* involves holding sample by hand with controlled pressure onto the polishing wheel, rotating it opposite to the rotational direction of the wheel, and back and forth rotation from center towards the edge of the wheel to ensure even distribution of abrasive and uniform wear of polishing cloth [17]. *Automated polishing* involves the use of a mechanical polishing device, either a simple one or a rather sophisticated, minicomputer, or microprocessor controlled unit, which can grind and polish a single or multiple (up to half a dozen or more) samples simultaneously with a higher degree of quality than hand polishing and at a reduced consumable cost [17]. Samples in an automated device are either held in place rigidly and pressed onto the cloth by a central force on the sample holder (produces best surface flatness and edge retention), or, held in place loosely and force is applied to each sample by a piston and planarity is achieved individually rather than collectively. Polishing time depends on abrasive size, cloth type, force applied, and wheel speed, which usually varies from 2 to 5 minutes for each step of polishing. An unnecessary long time spent in polishing is not only wasteful but can also produce undesirable surface relief (Ahmed [6]).

Cleaning and Drying after Polishing - A polished surface should be cleaned ultrasonically for 30-40 seconds with a solvent having a high flash point or no flash point. Excessive ultrasonic cleaning, however, can damage the surface. Sample can also be washed by swabbing with a liquid detergent solution, rinsed in running water, or with forcefully sprayed alcohol or ethanol, and then dried. Thorough cleaning of the surface after grinding and polishing are important to remove the abrasive residues and their interference during x-ray microanalysis in SEM. Rapid drying of the ground/polished surface can be done by applying a stream of forced warm air or compressed air to the surface.

Table 2 provides various grinding and polishing methods. Table 5 provides a list of various manufacturers of polishing abrasives, cloths, and equipments.

(8) Etching and Staining

Etching – Etching a polished section of a clinker, cement, slag, or concrete with a chemical reagent (etchant) highlights various components (e.g., individual clinker phases, residual clinker particles in concrete, slag, etc.) by selective absorption of the etchant with removal of surface layers of the components of interest in solution. The etched surface is observed in a high-power reflected-light (metallurgical) microscope. Etching of carbonate aggregates by dilute hydrochloric acid produces CO₂-effervescence. Limestone aggregates show higher effervescence than dolomitic aggregates. Etching can be performed on a smooth, dry, highly polished section, which is free of any surface irregularities and lubricants from the previous grinding or polishing operations. Usually the polished surface is immersed into a thin layer of etchant in a shallow petri dish (or held above in case of diluted HF acid vapor etchant; HF acid is placed in a platinum crucible and the inverted polished surface is held above it) and is then washed with alcohol to stop the reaction and quickly dried in an air current. Nital, HF vapor, potassium hydroxide in alcohol, and salicylic acid in alcohol are the most common etchants used in clinker microscopy (see Campbell [15]). Borax and sodium hydroxide solutions are the etchants for examinations of high alumina cement. Table 3 provides twenty different staining and etching procedures for examination of polished sections of clinker, cement, raw feeds, slag, concrete, and aggregates (see Campbell [15], St. John et al. [2], Walker [14], and Hutchinson [1] for comprehensive reviews).

Crack Identification – Various authors suggested a dye impregnation method for highlighting cracks in concrete by solvent replacement procedure, which involves immersing a wet, cleaned, finely ground, and polished section of concrete (polished with 6- μ m diamond paste) in a red or fluorescent dye-mixed alcoholic solution, followed by careful re-polishing in water with 1 to 3- μ m diamond to remove any excess dye from the surface (Hornain et al. [24]; Gran [25]). Dye-impregnated micro and macro cracks are easily highlighted in reflected-light examination. A fluorescent dye mixed alcohol treated ground or polished surface can highlight many fine cracks when examined in ultraviolet light. Dye-mixed epoxy impregnated thin sections are also excellent for highlighting cracks (described later).

Staining – Selective staining of finely ground, polished or thin sections of clinker, cement, or concrete with a chemical reagent highlights various components with a reaction product remaining on the surface which is either colored or can develop characteristic colors by further treatment. Tables 3 and 4 describe various staining techniques for identification of different phases in clinker, cement, aggregate, and concrete. Hutchinson [1] and Campbell [15] described various staining techniques applied on uncovered or polished thin sections to highlight various silicates (feldspar, quartz, mica) and carbonates (calcite versus dolomite) in rocks, aggregates, and raw feeds.

(9) Thin Sectioning (Including Fluorescent Thin Section Preparation)

Thin sectioning indicates reducing a sample thickness down to approximately 20- μm (0.020 mm), through which light can transmit. Thin sectioning is an important step in sample preparation, which provides the detailed anatomy of a material's microstructure, and a wealth of information about the overall texture, condition, mineralogy, composition, and the depth of deterioration or alteration in the sample.

The first petrographic thin section (of a calcareous rock) was prepared by an English Scientist, Sir Henry Clifton Sorby in 1849 (published his procedure in 1868), who has led the foundation of modern sample preparation techniques in petrography. Sorby's work on metallic meteorites made him interested in preparing metallic samples. Because of his pioneering work, Sorby is considered the father of both petrographic and metallographic sample preparations.

Thin sectioning: (a) can still be done entirely manually, by hand as Sorby did, or, (b) more rapidly, consistently, and precisely with excellent edge retention by using various modern semi-automated thin-sectioning machines for preparing one sample at a time (e.g., Hillquest's thin-sectioning machine, Buehler's Petro-Thin unit, and Ingram-Wards' thin-sectioning machine – all three units have a separate precision diamond wafering blade for sectioning and a diamond cup wheel for precision grinding), or (c) by using completely automated thin-sectioning equipments for preparing multiple samples simultaneously (e.g., Microtek's Micro-Trim thin-sectioning machine, or, Logitech's horizontal rotary grinding/polishing units with precision grinding jigs, see Table 5).

Irrespective of the equipment used, following is a series of steps generally followed in any thin section preparation; many of these steps are already described in detail in the preceding paragraphs. The exact procedure depends on the type and condition of the sample and is a matter of convenience or preference of the individual.

(a) Encapsulation and/or Epoxy Impregnation – If the sample is a loose, small, powdered, cracked, porous, disintegrated, or deteriorated material, it must be encapsulated and vacuum impregnated with a low-viscosity epoxy. Clinker and cement samples must be encapsulated in epoxy before thin sectioning. A hardened concrete or mortar is usually encapsulated (if fragmented or cracked) and vacuum impregnated with a colored epoxy. A concrete sample is first oven-dried to a constant mass at 30-40°C for several hours to remove its internal moisture and then vacuum impregnated at room temperature with a low-viscosity, easy-flowing epoxy. Fluorescent or other colored epoxy can be used, which highlights cracks, pores, and voids in the final thin section. A slight warming of epoxy on a hot plate or in an oven lowers its viscosity, promotes thorough mixing of powdered dye, and later, during impregnation at room temperature, helps deeper penetration in the sample. A slight warming of the impregnated sample in an oven at 45-50°C accelerates epoxy curing (curing temperature should be restricted to a lower value for the materials containing thermally sensitive phases).

(b) Trimming – In case of a hardened concrete, mortar, or other relatively hard material, the sample is first trimmed down from a large size to a small, manageable, rectangular block approximately 5-10 mm in thickness that will fit in a frosted glass slide (commonly 27 \times 46 mm or 50 \times 75 mm size), where it will be mounted. The trimmed, rectangular block is then epoxy impregnated as described above. The thinner the sample, the faster it dries during the oven-drying. The hardened, epoxy-impregnated sample prepared in the previous step is also sectioned

down to a small slice, slightly smaller than the glass slide of the final thin-section and around 5-10 mm in thickness. Trimming is usually done in a wet tile saw or in a precision saw having a thin diamond-bonded blade, which produces a very smooth cut with minimum surface deformation. For water-sensitive materials, a cutting fluid or oil is used. For soft and brittle materials, a second epoxy impregnation is sometimes required after trimming of the epoxy-impregnated sample if the trimmed surface (which will be the final thin-section) does not achieve the desired hardness and integrity by vacuum impregnation.

(c) Fine Grinding – Fine grinding of the saw-cut surface of trimmed sample is necessary for good bonding of that surface to the glass slide. Fine grinding at 320 or 600 grit abrasive paper is necessary to remove the minimum surface irregularities created during sectioning and to achieve a perfectly smooth and flat surface that will bond intimately to the glass slide. After grinding, the surface should be thoroughly cleaned, freed of oil, dirt, grease or lubricant from previous actions, and oven-dried to a constant mass at 30-40°C.

(d) Bonding the Ground Sample to a Glass Slide – The oven-dried, smooth, flat, clean, ground surface of the sample is glued with an adhesive to a clean, dry, and ground (frosted) glass slide of known thickness. A low-viscosity, fast-setting liquid adhesive is used for bonding (e.g., see Table 1, Canada balsam resin, having a refractive index of 1.54, is commonly used, other choices are Buehler's Epothin, Logitech's Epoxy Pack 301, or Loctite manufactured by Loctite Corporation, which cures in 15 minutes in a 385nm UV light having an intensity of 300 microwatt at 6 in.). Various spring-loaded thin-section bonding fixtures (e.g., Buehler's PetroBond) are helpful for applying uniform pressures over the slide during the hardening of the thin film of epoxy at the sample-slide interface. Glass slides up to 50 × 75 mm in size are usually 1-1.5 mm in thickness; larger slides up to 100 × 150 mm should be 3 to 4 mm thick and all slides should be frosted or ground in one side for uniform thickness of the slide, better adhesion, and flat, continuous contact of the ground sample to the slide. Usually, a few drops or a thin film of epoxy is spread on the ground surface and the frosted side of the glass slide is placed onto the surface from a 45° angle. Pressing and rubbing the back of the slide against the sample surface remove all trapped air bubbles.

(e) First-stage Thinning by Precision Sectioning – The excess portion of the glass-mounted sample block is removed by using a micrometer-controlled diamond wafering blade in a precision saw, leaving a thickness of about 0.5 mm attached to the glass slide (or about 1 mm thick slice if done by hand on a regular tile saw with a thin diamond blade). Table 2 lists various precision sectioning machines that can be used for this purpose.

(f) Second-stage Thinning by Precision Grinding – After the first sectioning, the sample is further thinned by grinding in an automated thin-sectioning machine by a micrometer-controlled, precision grinding wheel or cup down to a thickness of about 25 to 30- μm , a thickness through which light can transmit. Precision grinding can be done on a horizontal rotary wheel with loose 5 to 15- μm sized SiC/Al₂O₃ abrasives (e.g., in Logitech's machines), or in a vertical diamond-bonded cup wheel (where about 60- μm size diamond is embedded to a brown cup wheel, e.g., in Buehler's Petro-Thin), or in a vertical diamond-bonded grinding plate (e.g., in Micro-Trim). Both horizontal and vertical precision grinding methods have some advantages and disadvantages. A precision micrometer (calibrated in microns) controls the final thickness. The

first order gray interference color of quartz indicates 25 to 30- μm thickness of the sample (in thicker samples, quartz grains are usually yellow or purple). In the absence of a thin-sectioning equipment, precision grinding can be done by holding the glass slide with a suitable sample holder (with the sample side down) on a horizontal rotary grinding wheel covered with a fine steel mesh or a metal- or resin-bonded diamond disc or Buehler's Ultra-Pad and charged with fine (5 to 15- μm) diamond or alumina abrasive slurries.

(g) Final Thinning by Hand Grinding - Some authors (Ahmed [5], Campbell [15]) and thin sectioning equipment manufacturers (Buehler, Logitech) suggest thinning of the sample down to 40 to 50- μm by using the precision sectioning/grinding machine and then final thinning down to 20- μm by careful hand grinding of the sample on a glass plate charged with 5 to 10- μm sized alumina or SiC abrasive slurry in water, light oil, or glycol, or, more efficiently, on a fine woven steel-mesh pad (e.g., Buehler's UltraPlan) charged with 15 to 25- μm size diamond suspension or paste. Thinning of the paste of portland cement concrete (relative to much harder aggregates) can be accomplished with the Buehler's Ultra-Pad on a rotary wheel with a slurry of silicon carbide or diamond powder, bringing the paste to a thickness of approximately 20 microns (judged by the bright gray interference color of the calcium hydroxide component of portland cement hydration; Campbell, personal communication).

(h) Polishing or Protecting Thin Section with a Cover Slip- A thin section can be further polished to a shiny surface for various benefits such as: staining tests and reflected-light observations on thin sections, observation in SEM, x-ray microanalysis, opaque mineral study, mineral hardness/microhardness determination, and examinations at high magnification and high resolution in a petrographic microscope by using a 100X oil-immersion objective. A double-polished thin section is sometimes prepared in high-resolution works by polishing both sides of the sample. Polishing should be done first on a hard and then on a soft polishing cloth by using successively finer sub-micron sized diamond or alumina polishing abrasives in water, light oil, or glycol-based lubricant. In the absence of polishing, thin section should be protected from atmospheric oxidation, carbonation, and other alterations by using a 0.17 mm thick glass cover mounted on a clean, dry, freshly ground thin section of the sample either temporarily by an immersion oil, or a temporary mounting media, or permanently by a fast-setting medium (e.g., Canada Balsam, Epothin, Loctite), which is uniformly distributed throughout the sectioned surface in the slide without enclosing any trapped air.

(i) Cautionary notes on fluorescent thin section preparation for determination of water-cement ratio by image analysis (Jakobsen et al., Roy and Idorn [26]) – Several scientists have described the procedures for determination of water-cement ratio in a hardened concrete from image analysis of photomicrographs taken from fluorescent-dye mixed epoxy impregnated thin sections [26]. In this determination, it is important to mix a fixed concentration of dye in the epoxy and use a fixed thickness of thin-section in both the standard and the unknown samples because both these factors have strong influences on the degree of fluorescence of the paste. Too much dye in the epoxy, or too thick section will increase the total amount of fluorescence (show brighter image) and gives an 'apparent' water-cement ratio, which is higher than the actual value. If the epoxy appears too pale or too yellow in large voids this would indicate a wrong dosage of fluorescent dye during the preparation of epoxy. Usually, the amount of dye to add is 1 percent by mass of the epoxy resin. Inhomogeneity in color distribution may indicate insufficient mixing

of dye in epoxy. The thickness of thin-section should provide first order gray interference color of quartz (i.e., 20- μm or 0.02-mm thick). The thin section should be located in the fully epoxy-impregnated part of the sample. Due to the very small depth of impregnation of epoxy into concrete, thin section should be prepared from the top impregnated surface with as little grinding after impregnation as possible. Depending on the water-cement ratio (i.e., density or porosity) of a concrete and the viscosity of epoxy, this depth of epoxy penetration can vary from as little as 0.05 mm to 1.5 mm. Insufficient impregnation is indeed a common problem, therefore, check the small voids in thin-section in plane-polarized light to make sure that they are completely filled with the fluorescent or colored epoxy. Also, since the intensity of fluorescent dye decreases with time and exposure to light, new standards should be prepared relatively often and fluorescent thin-section should be kept in dark cupboard. The intensity of light source in the microscope should also be constant. Areas of carbonated or leached paste should be avoided as these processes change the porosity of paste and hence the degree of fluorescence. Avoid the areas of the paste that are too close to a large void or crack or at the edges of thin-section because the bright light flares from the epoxies may increase the brightness of the paste and, hence, the apparent water-cement ratio. Depending on the inhomogeneities of the paste, approximately 10 to 30 randomly selected areas in each thin-section are selected for ratio determination (avoiding the effects of any inhomogeneous mixing or dispersion of cement) from which the ‘average’ water-cement ratio is measured.

A thick section does not resolve the microstructural details; an ultrathin section may induce cracking, grain plucking, edge loss and other damages, blurred images, and very little microstructural details; variable thickness across the surface (wedged section) complicates the examination and provides poor images. Detailed descriptions of thin section preparation are available in Hutchinson [1], ASTM C 856 [9], Nordtest Method NT Build 361-1991 [27], St. John et al. [2], Walker [14], Campbell [15], and Ahmed [3-6]. Rapidity and reproducibility of thin sections depend on the sample type and size, thorough drying and epoxy impregnation procedures, type of epoxy used, quality of the ground surface bonded to the glass slide, frosty nature and cleanliness of the glass slide, sample thickness left after precision sectioning, quality and condition of the sectioning blade, hardness of the sample to be used for precision sectioning and grinding, and use of diamond paste for final hand grinding on a steel woven disc. Consistencies in thin sectioning procedures and in the final thickness of the sample are important when thin sections are used for quantitative petrography (as described above in the section of determination of water-cement ratios by fluorescent microscopy).

(10) Grain Thin Sections

Unlike the above discussion of thin section preparation of concrete, mortar, aggregate, stone, and other solid samples, thin sections of powdered and fragmented materials such as fine sand, pea gravel, cement, whole or crushed clinker, raw feeds, ground pozzolan, etc. are usually prepared in a different manner. The procedures are simple and require less time than a usual thin section of concrete. Three methods are common.

The first method involves usual encapsulation of powder or crushed material in castable epoxy in a mold as a thick, viscous, paste-like consistency, followed by curing, sectioning,

grinding, bonding the ground surface to a frosted glass slide, precision sectioning, precision grinding to the final thickness, and optional polishing.

The second method involves applying a thin film of epoxy to a clean, dry, frosted glass slide and sprinkling the fine power (of cement, fine sand, clinker, raw feed, etc.) over the epoxy (or placing the powder first and then applying a few drops of epoxy), or applying a thin film of a “paste” of already mixed epoxy and fine powder on a frosted glass slide, letting it to cure either in air or in an oven, grinding the surface down to a smooth plane to expose majority of the grains, and slow, continuous grinding, and occasional coarse polishing to the final thickness. The thin section can be further fine polished or covered.

For relatively coarser grains such as pea-sized whole clinkers, crushed clinkers (1 to 2 mm), sieved fine aggregate particles (having uniform grain size of most of the particles), and finer fractions of coarse aggregates, the grains are either sprinkled over a clean, dry, and frosted “working” glass slide coated with a thin film of epoxy, or, the grains are first soaked in an epoxy medium and then removed from the epoxy with forceps and placed on the working glass slide (epoxy from the wetted grain surfaces will provide the necessary bond to the frosted surface of the glass, Campbell [15]). The grain mounted glass slide is cured either in air, or in an oven, or on a hot plate at 40-50°C and then either thin-sectioned in a precision saw and/or ground down to expose full cross sections of majority of the grains. The sectioned and ground surface is then bonded to a clean, dry, frosted glass slide, which will be the final “sample” slide. The sample sandwiched between the two slides is then further processed by second precision sectioning and grinding of the sample slide down to the final thickness of 30 to 40- μm . The companion side of the thin section left over on the working glass slide from precision sectioning can be further polished on a horizontal rotary wheel with a suitable slide holder for reflected-light or SEM examination.

(11) Polished Grain Mounts (Half Sections, This section is provided by Dr. Donald H. Campbell¹)

Examination of polished tops of grains encapsulated in epoxy is common in SEM studies, particularly in ore microscopy. The method has been modified for use in routine concrete and cement microscopy in which the polished surface passes roughly through the middle of particles. The grains are polished on only one surface instead of two (as in a doubly polished thin section). Hence, the grain mount may be termed a half section. With a one-particle-thick layer, grains can be examined in reflected- or transmitted-light, or both simultaneously with some microscopes. Transmitted light through a transparent mounting medium allows particle observations in three dimensions, but reflected light gives only a planar (two dimensional) view. Both have their phase-identification advantages. The section can easily be etched or stained.

In this method, non-stick paper (for example, the backing from an adhesive-backed polishing cloth) is placed on a slide warmer at 45 degrees C, a drop of epoxy is put on the paper, and the particles are added to the liquid. A clean, labeled, glass microscope slide placed on the mixture and with a light finger pressure and movement the excess epoxy is squeezed out. A

¹ Modified from Campbell, D. H., 2004, "Microscopical Quality Control of Clinker and Cement," in Innovations in Portland Cement Manufacturing, Chapter 8, Portland Cement Association, Skokie, Illinois, USA, CD (1387 pages).

weight is placed on the slide and the epoxy is allowed to harden. After hardening, the encapsulation easily separates from the non-stick paper, the excess epoxy is trimmed from the edges of the slide with a single-edge razor blade, and the sample is ready for coarse and fine polishing with diamond pastes or slurries on Buehler's TexMet or equivalent cloth. The particles can be seen at the base of the epoxy.

Grain mounts normally require no lapping with silicon-carbide papers. No. 2 rubber stopper, or a cabinet door "bumper" is affixed with Super-Glue to the back of the microscope slide, facilitating holding the slide to the horizontal polishing wheel.

The first polishing step is primarily for thinning the grain mount until broad cross sections of individual particles can be seen under the microscope. The entire preparation of the specimen surface can be done on a horizontal polisher-grinder. In the final stages of polishing, seeing the particles with the naked eye is normally difficult if the particles are, say, cement-grain size. Therefore frequent checking with the microscope is necessary until the desired thickness (usually 20 to 40 microns) and degree of polish is attained. The stopper is removed with a single-edged razor blade after final polishing or etching. If no transmitted-light observations are planned for the section, then a ceramic tile and Super-Glue can be used instead of a glass microscope slide and epoxy. The method normally requires roughly 15 to 30 minutes, depending on temperature and embedding liquid characteristics. Gridded microscope slides are helpful for returning to a particular grain.

(12) Thin-Sections for Aggregate Petrography (ASTM C 295)

For aggregate petrography (ASTM C 295), the author uses several 50 × 75 mm thin-sections for preparation of coarse and fine aggregates. Following careful washing, oven drying, and sieve analysis of the aggregates, a representative size fraction retained in each sieve is collected by coning and quartering. At least 150 particles are examined in each sieve. For the size fractions coarser than $\frac{3}{8}$ in., the author uses one or multiple 50 × 70 mm-thin sections, or a large-area (up to 100 × 150 mm. size) thin section per size fraction to include adequate number of grains. For sizes finer than $\frac{3}{8}$ in., one frosted, 50 × 75 mm glass slide is first covered at four sides and partitioned inside into multiple compartments by gluing several small glass or plastic partitions on the glass slide with a rapid setting commercial resin (e.g., superglue). Alternately, a plastic, compartmented, disposable sample mold with 4 to 6 compartments placed on a silicone grease-coated glass plate (or glued to a frosted glass slide by superglue) can also be used (e.g., plastic fluorescent-light diffuser panels with multiple square chambers described for preparation of clinker and raw feed thin sections by Campbell [15]). Each selected sieve fraction is mixed with enough epoxy to create a thick paste-like consistency and poured into each labeled compartment on the glass slide and then vacuum impregnated. For fine aggregate, one 50 × 70 mm glass slide can hold up to 5 or 6 different sieve sizes with more than enough grains to examine. A few minutes in a vacuum chamber removes most air bubbles. The epoxy-encapsulated grains are cured either in air, or in an oven, or on a hot plate at 40-50°C. The hardened sample is then thin-sectioned according to the steps described in thin sectioning. The hardened sample is first sectioned down to 300 to 500- μ m in a precision saw and then reduced to the final thickness by precision grinding. The frosted glass slide on which the epoxy-mixed grains were poured can be used to make the final thin-section. The method is similar to grain

thin section preparation, which is also recommended by ASTM C 295 for examining particles finer than No. 50 (300- μm) sieve.

(13) Phenolphthalein and Rainbow Indicators for Determining the Depth of Carbonation

The above-two solutions are used for determining the depth of carbonation in a portland cement concrete. A fresh fracture surface or a fresh saw-cut section of a concrete is treated with a 1 percent phenolphthalein alcoholic solution - the non-carbonated concrete turns to pink color due to its inherent high alkalinity ($\text{pH} > 9$) whereas the carbonated portion does not change to pink due to a drop in alkalinity by carbonation ($\text{pH} < 9$). Carbonation of paste along a crack can be distinctly observed by application of phenolphthalein. Upward migration of soluble alkalis through a concrete slab by moisture and accumulation of those ions near the carbonated surface can give a 'false' pink coloration, which should be corrected for the true depth of carbonation (usually by further examinations of the true depth of carbonation in a thin-section). Another less common method is the similar treatment of a fresh fracture surface with a solution called rainbow indicator¹, which provides a range of pH-correlative colors from orange (for a pH of 5) through yellow (pH=7), green (pH=9), purple (pH=11) to dark blue or black (pH=13).

(14) Staining Techniques for Rapid Identification of Alkali-Silica Gel in Concrete

Uranyl Acetate Treatment² - In this procedure, a damp, fresh fracture surface of a concrete is treated with a spray of uranyl acetate solution (prepared by thorough mixing 5 grams of powdered uranyl-acetate in an warm 100 ml acetic acid solution). After absorption of the solution to the surface for a minute and rinsing the excess solution from the surface with distilled or deionized water, the treated surface is exposed to a short wavelength ultraviolet light (254 nm) in a viewing cabinet or in a dark room. Alkali-silica gel fluoresces bright greenish-yellow, especially in and around the reactive aggregate particles, in cracks, and in voids. Ettringite, carbonated areas, agglomerated particles of fly ash, silica fume, slag, and some chert particles in aggregate may also fluoresce; however, these fluorescence can be distinguished from the bright greenish-yellow fluorescence of gel at the specific locations. Alkali-silica gel identified this way should be confirmed by other petrographic techniques such as oil-immersion mounts and/or thin-section microscopy. The procedure is described in detail in Natesaiyer and Hover [28] and ASTM C 856 [9]. Uranyl acetate is a hazardous material and should be handled, stored, and disposed following the environmental regulations; eyes and skins should be protected from the UV light.

Cuprammonium sulfate staining – In this method a broken surface, ground surface, or thin section of concrete is absorbed in a 4M cuprammonium sulfate solution for 7 hours and then washed in water. Alkali silicate gel exudations in voids and cracks stain blue. The method is described in detail in Poole et al. [29].

¹ Both phenolphthalein and rainbow indicators are available from Germann Instruments, Inc. 8845 Forest View Road, Evanston, IL, 60203, USA 847-329-9999 (Tel).

Sodium Cobaltinitrite Staining² – Guthrie and Carey [30, 31] proposed staining of a fresh fracture or aged, exposed, pre-rinsed surface of concrete with a saturated aqueous solution of sodium cobaltinitrite solution, which reacts with exchangeable potassium in the ASR-gel to form a bright yellow precipitate on the gel surface. The intensity of yellow staining provides a rough indication of the variation of potassium content in the gel. A counter stain with a rhodamine B base compound highlights calcium-rich ASR gel (and can also stain carbonated zones or other deteriorated and porous pastes) in pink and provide a high contrast to the yellow stained gels, making them easier to observe. Yellow staining of high alkali ASR-gel by sodium cobaltinitrite is more consistent and diagnostic than pink staining by rhodamine, which highlights both Ca-rich ASR gel and other deteriorated concretes. Unlike uranyl acetate, the chemicals pose minimal health risks and are environmentally benign. Since ettringite deposits in voids or cracks remain unstained, this method helps to distinguish secondary ettringite from ASR-gel [30, 31].

It is important to remember that detailed petrographic examinations of concrete, including examinations of: (a) fresh fracture surface of concrete in stereomicroscope for detection of clear, shiny, or white, matt-finished alkali-silica gel in cracks and voids, (b) oil immersion mounts of alkali-silica gel in petrographic microscope, (c) gel in cracks, voids, around the reacted aggregate particles, and in other places in thin section, and (d) sawn, fractured, or polished surface in SEM provide more unquestionable evidences of such a reaction in the concrete than the staining methods, which are not necessarily unequivocal. Therefore, conclusions from staining methods must be confirmed by detailed petrographic examinations.

(15) Sample Preparation for Air-Void Analysis in Hardened Concrete by ASTM C 457

The conventional method of sample preparation for determining air-void parameters in hardened concrete is described in ASTM C 457 [8]. In this method, a sample is first sectioned by using a conventional water or oil-cooled diamond saw at a direction perpendicular to the direction of placement or finished surface, and then lapped/ground to a smooth, flat ground surface. Grinding is done either by using successively finer loose SiC abrasives on an iron lapped plate, or, more efficiently, with metal-bonded magnetic diamond discs of 80 (used for initial grinding of very rough, sectioned surface), 240, 320 grits and then resin-bonded diamond discs of 400, 600, 1200, and 3000 grit sizes. The ground surface should be thoroughly cleaned in moving from one to the next finer abrasive size. In case of a soft, friable material, a concrete with weak aggregate-paste bond, or a concrete with excessive air, an epoxy or a carnauba wax-impregnation stage is recommended after sectioning and oven-drying, and prior to grinding, which strengthens the sectioned surface and preserves the air void margins during grinding.

The quality of the final ground surface is very important for good results. Poor quality of the lapped surface can give an erroneously high air content, especially when the concrete has a high amount of air. The finished surface should be free of scratches, it should have excellent reflection of a distant light source when viewed at a low incident angle, there shall not be any noticeable relief between the aggregates and paste, and the air void margins should be sharp and not eroded or crumbled.

² Uranyl acetate is available from Fisher Scientific International, Inc. (800-766-7000); Sodium cobaltinitrite stain is available from Fisher Scientific Instruments, Inc. and James Instruments, Inc. (800-426-6500).

Though not common, dye-mixed epoxy impregnated large-area (50×75 mm) thin sections prepared from the top, middle, and bottom portions of a concrete core, or, an ultra-large 100×150 mm thin section prepared from a 4-in. (100 mm) diameter concrete core can be used to study air voids in a stereomicroscope or petrographic microscope equipped with an automated air void analyzer (Buckingham and Spaw [36]). An area of at least 12 in.^2 (77 cm^2) is needed for examining a concrete containing $\frac{3}{4}$ -in. or 1-in. nominal maximum sized aggregate. Inadequate filling of the air voids by the dyed epoxy, however, can reduce the air content.

ASTM C 457 provides the minimum area to be covered for concrete containing various aggregate sizes, which increases with increasing the maximum size of aggregate. The area to be covered also depends on the overall homogeneity of the sample. If the concrete is heterogeneous in distribution of aggregates, or has large voids (e.g., poorly compacted), the total area of examination should be increased proportionately. For small size samples (e.g., concrete core less than 3-in. in diameter and length), two parallel sections are prepared and are oriented perpendicular to the finished or exposed surface and studied either side by side or one after another in a stereomicroscope; the distance between the parallel sections should exceed the maximum size of the aggregates to ensure that each section intersects new concrete material.

(16) Sample Preparation for Air-Void Analysis in Hardened Concrete by Image Analysis

During the past two decades, a number of techniques have been developed for rapid measurements of air-void parameters in concrete by automated binary image analysis techniques, where the air voids are highlighted on a lapped section. The lapped section is treated with a black ink to darken all phases except the air voids, which are then filled with a white paste of alumina, zinc oxide, wollastonite, or cornstarch; voids in aggregates are then darkened by a marker pen. The prepared section is photographed by an image-grabbing device (e.g. digital camera, video camera, or a flat bed scanner). Either the whole lapped section or a series of adjacent lapped areas are captured at regular intervals. The captured images are processed for measuring the amount, size distribution, and air void spacing by various softwares [see references 3, 4, 32-38].

In order to increase the contrast between the air voids and the surrounding matrix in an image, one method suggests treatment of a finely ground, clean, and oven dried concrete section to black ink (by slightly wetting a surface of a stamp pad with black ink and gently pressing that surface against the ground surface without filling the voids with ink), followed by oven drying the ink and filling all the voids with white zinc oxide paste (smearred over the warm, dry, black ink-treated surface). The surface is then cooled and re-dried to harden the paste, carefully scrapped with a plastic scraper or putty knife to remove the excess paste, and slightly re-ground to highlight the white paste-filled air voids and pore spaces against the black background (Ahmed [4]). Void spaces within the aggregate particles should be manually darkened with a black marker pen to reduce errors in air void calculations.

Another method suggests vacuum impregnating the sample with a low-viscosity epoxy, which is thoroughly mixed with a white titanium dioxide pigment (where the voids will be filled and thus highlighted by the white epoxy), treating the epoxy impregnated, lapped and oven dried clean surface with black ink, drying the ink in an oven, followed by slightly re-grinding the

surface just to remove the ink stain from the white epoxy filled voids so that the white voids can stand out against the black background (Ahmed [3]).

Several other authors have proposed determination of air void parameters by semi-automated image analysis techniques where similar white filling materials were used to highlight the air voids against the dark background of surrounding matrix in the finely ground or polished section and to enhance their contrasts in the image (Chatterji and Gudmundsson [32]; Cahill et al. [33]; Roberts and Scali [23]; Peterson et al. [34]; Laurencot et al. [35], Buckingham and Spaw [36], Pade et al. [37], and Zhang et al. [38]).

Any surface defects resulting from sample preparation (e.g., grain plucking) may increase the void content in an automated image analysis procedure. Coalescence and clustering of excessive voids resulting in irregularly shaped coarse void clusters can complicate the results, which could be avoided by the manual (i.e., ASTM C 457) method. Refinements in software programs and sample preparation techniques are, therefore, attempted to overcome these difficulties [34, 35, 37, 38].

(17) Conductive Coating of Sample for Scanning Electron Microscopy

During observation in a scanning electron microscope under high voltage, high vacuum, and high magnification, a thin coating of an electrically conductive material (carbon, gold, or gold-palladium alloy) is necessary to prevent accumulation of electrical charge on the surface. Coating is applied on dry, as received, fresh fracture, polished, powdered, or thin section of a nonconducting material (such as concrete) to prevent direct charging by the incident electrons. Metals, on the other hand, do not require a conductive coating. Detailed procedures for preparation of polished surfaces of metals, ceramics, and geological materials, including various conductive coating techniques (e.g., thermal evaporation of C, Au, Au-Pd, or Pt-C at high or low vacuum; sputter coating by direct-current, plasma-magnetron, ion-beam, or penning sputtering of Au, Pt, Au-Pd or other noble metal alloy targets), are given in Goldstein et al. [39]. In the absence of conductive coating, a sample directly exposed to incident electron at high vacuum will be charged resulting in distortion and blurring of image and thermal and radiation damage, which can lead to a significant loss of mass from the sample. Carbon coating minimizes the absorption and attenuation of the incident electron and outgoing x-rays. Gold coating has interference with elemental sulfur in the sample. The coated sample must be electrically grounded by providing an electrical connection between the coated surface and the metal specimen holder by using an electrically conductive silver or carbon paint. A small amount of a sample of interest (e.g., power mount), however, can be transferred by a needle to a conductive carbon tape (which is attached to a sample holder) for rapid examination and characterization in some SEM (e.g., low-vacuum or variable pressure SEM) without necessarily providing a conductive coating.

(18) Ultra-thinning

Ultra thinning can be of two types. The first type involves ultra thinning a usual 20 to 25- μm thick “thin section” down to 6 to 10- μm to reveal many ultra-fine grains that are smaller than the usual 20 to 25- μm thickness of thin sections and can stack one on top of the other. Due to the

very fine grain size of many cement clinkers and portland cement hydration products in a concrete (finer than 25- μm), ultra-thin sections are necessary. They are usually prepared by a vibratory polisher (e.g., Buehler's Vibromet, Ahmed [3, 5, 6], Ray [40]) which slowly and very gently polishes a conventionally prepared thin section to carefully grind it down to the desired thickness and provide microstructural details by removing the grain stacking. The vibratory polisher uses a polishing cloth attached to a polishing bowl and receives a micron to submicron size abrasive. Generally, a steel mesh cloth (e.g., Buehler's Ultra-Plan) charged with 5 to 6- μm diamond slurry is used for first stage ultra-thinning down to 12 to 15- μm and then a hard, napless cloth (TexMet pad) charged with 1- μm diamond slurry, or a soft napped cloth (MicroCloth) charged with submicron sized alumina is used for final delicate thinning down to 6 to 10- μm (Ahmed [5, 6]). Due to the very slow sample removal, the entire process can take 12 to 16 hours depending on the sample hardness.

The second type, i.e., ultra thinning a slice of concrete down to less than 0.1- μm , is in fact a very delicate procedure, which is needed for observation in a transmission electron microscope. This is done first by mechanical abrasion and then by careful slicing, polishing, and finally bombarding the doubly polished disc of sample with argon ion or atom beam until perforation. Differential rates of thinning by different constituents, and heat and radiation damage during beam-milling make sample preparation a crucial part of examination. Operating at low ion/atom beam current and the presence of a cooling stage reduce the heating damage whereas decreasing the operating voltage and the angle of incidence of the ion/atom beam at the end of the thinning process can reduce the radiation damage. At present, this technique is used only in research (e.g., observation of hydration products around clinker or silica fume particles at ultrahigh magnification) and is not essential for regular petrographic examination.

(19) Sample Preparation for X-Ray Diffraction in Powder Diffractometer

For x-ray diffraction, a small amount of a previously crushed bulk sample should be pulverized in a bench top laboratory mill to a very fine power (less than 45- μm , preferably in 1 to 10- μm size) of talc-like consistency. Fine particle size produces strong intensity diffraction peaks and reduces the problems of microabsorption, extinction, preferred orientation, and sample homogeneity, all of which can result in peak intensity errors. Pulverization should not generate heat high enough to dehydrate any hydrous phases in the sample. Sample should, therefore, be mixed with acetone, alcohol, ethanol, or other solvent to minimize the frictional heat. During pulverization, the sample should not be contaminated with any foreign or mill material. Depending on initial fineness and softness, the sample can also be pulverized by hand in a small agate pestle and mortar with an acetone or alcohol lubricant. Excessive grinding or dry grinding may deform or decompose the crystalline phases, or may produce an amorphous component, which is indicated by a decrease in peak intensity and an increase in peak width. Bulk samples are used for examination of cement, lime, gypsum, crushed/ground clinker, efflorescence deposits, mortar and concrete. In concrete, however, selective collection and pulverization of paste and mortar fraction from a crushed bulk concrete can enhance detection of secondary deposits and cement hydration products in the x-ray diffraction pattern by reducing the influence of aggregates. Besides pulverized fine powder, polished and thin sections of concrete and other materials can also be examined by placing them in suitable sample holders in the x-ray diffractometer. Very small amount of sample can be analyzed by smearing on a sample holder

containing a single-crystal quartz glass plate, or by using a thin-film sample holder. Other methods for analyzing fine samples are smearing the fine mineral powder on a normal petrographic slide and slurry it with acetone (can lead to preferred orientation of platy minerals), or, sprinkling powder onto a tacky adhesive coat on a glass slide (will provide less preferred orientation than the slurry with acetone). Hutchinson [1] described various other preparation techniques for clay minerals such as powder pressing, smearing clay paste on a glass slide, suction on ceramic tile, and centrifugation on ceramic tile.

Various selective extraction techniques of portland cement and clinker¹, such as potassium hydroxide/sugar extraction, salicylic acid/methanol extraction, and nitric acid/methanol extraction selectively dissolve the interstitial (aluminates, ferrite) phases, calcium silicate phases, and silicate/aluminate phases in the cement, respectively, which, thereby, reduce the peak interference problem, and highlight the selectively un-dissolved phases in the diffraction pattern. Cement and other moisture-sensitive materials should be handled carefully during the preparation steps, and stored in a vacuum desiccator to eliminate hydration.

(20) Sample preparation for environmental and cryo-SEM and other new x-ray microscopical techniques

Cryo-SEM and environmental SEM (ESEM) offer examination of early hydration of cement and associated microstructural development without any possible alteration induced by drying of sample in a conventional SEM. In cryo-SEM, sample is freeze rapidly in liquid nitrogen at -195°C to stop any ongoing hydration (e.g., of freshly mixed cement paste) where the water in sample freezes to an amorphous solid or sublimates; the frozen sample is examined in the SEM with the possibilities for x-ray microanalyses of selected phases (including frozen pore solution at any given time). Unlike examination of a static frozen hydration, in ESEM sample can be examined in a moist or wet condition, which provides an excellent opportunity to examine progressive microstructural development during cement hydration. Unlike normal SEM, samples do not have to be coated and elemental analysis can be done on a moist sample. Examination in ESEM, however, cannot be done in the presence of any excess water film on the sample. Soft x-ray transmission microscopy and x-ray computed tomography provide microstructural examination of cement hydration and three-dimensional imaging at high resolution in wet or dry conditions (Juenger [41]). All these relatively new techniques require minimum sample preparation for examination of cement hydration in wet condition. X-ray microscopy is a promising technique to examine hardened concrete in an ambient condition for imaging, quantitative microanalysis (similar to EDS and x-ray mapping in SEM), and phase identification by x-ray diffraction (Sutter et al. [42]).

CONCLUDING REMARKS

Various sample preparation techniques described in this article are the essential parts of detailed petrographic examinations, which should preferably be done by the petrographer rather than by a commercial sample preparation laboratory. Information obtained during sample

¹ For different extraction techniques, see Stutzman, P.E., "Guide for X-ray Powder Diffraction Analysis of Portland Cement and Clinker", NISTIR 5755, US Department of Commerce, 1996.

preparations (e.g., ease of sectioning and grinding, depth of epoxy penetration, etc.) can provide clues about the nature of the sample or even the cause of deterioration. Table 6 provides various common sample preparation steps for clinker, cement, aggregate, concrete, mortar, and plaster materials. The basic methods of preparation remain the same with some necessary and minor modifications depending on the type of materials to be examined. Table 7 provides sample preparation methods for specific modes of microscopical examinations. After sectioning, grinding, and polishing: (a) surface deformations induced by these processes should be removed or be at minimal; (b) scratches from grinding or polishing abrasives should be removed; (c) pullout, pitting, cracking or hard particles, smear, and other preparation artifacts should be avoided; (d) relief or excessive surface height variations between components of various hardness should be minimized to obtain uniform focus of the surface at high magnifications; and (e) the surface must be flat with edges properly retained. Sample must be cleaned adequately between preparation steps, after preparation, and after etching. The preparation methods should be as simple as possible, should yield consistent high quality results in a minimum time and cost, and must be reproducible. Sample preparation itself should not destroy or alter the composition of the material and its microstructure. Table 8 provides examples of various artifacts of sample preparation and the consequent misinterpretations of materials and microstructures.

The number of sample preparation techniques to be followed for a particular sample in a specific investigation should be minimal and yet adequate enough to provide all the information possible to extract for the purpose of the investigation. Applying multiple sample preparation techniques on a sample and obtaining similar information from each is not a cost effective approach. For example, to detect the presence of fly ash in a powder or in a hardened concrete requires only oil-immersion mount examination; a thin section, though can provide the same information, is more time consuming and may not be a cost effective approach for simple fly ash detection. Many times, examination of oil-immersion mounts is the best technique for rapid identification of an unknown material, which can be further explored with other techniques of sample preparation. Examinations of as received samples, oil-immersion mounts, fresh fracture surfaces, ground surfaces, thin sections, and polished surfaces are the common modes of observations of concrete and masonry in optical microscopes. Powder mounts on a conductive carbon tape, carbon or gold coated fresh fracture surfaces, polished sections, and polished thin sections are the common modes of examinations in scanning electron microscopy. A pulverized sample is used for x-ray diffraction and chemical analysis.

There are some specific claims of minimum sample preparation and examination of as received, fresh fractured, or saw-cut (unlapped, carbon-coated) section for concrete materials characterization and failure investigation in SEM (Marusin [43, 44]). Minimum sample preparation may be preferable at some specific cases, such as examination of water-soluble salts, efflorescence deposits, or specific areas of interest in a sample, which could be lost by subsequent sample preparation. Despite such specific cases, careful and appropriate sample preparation, often adequate and satisfactory enough to show the necessary microstructural details are very crucial for detailed examination and correct interpretation of materials and microstructures.

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Table 1: List of various encapsulating and impregnating castable resins and dyes¹

Epoxy and Acrylic Resins	Reference, Manufacturer	Resin-to-hardener mix ratio	Peak temperature (°C)	Curing time at room temperature	Comments
Epoxy resin and hardener	Buehler Ltd.	5:1, by weight	29	6-8 hours	Medium viscosity; best for encapsulation, refractive index is 1.565.
EpoKwick	Buehler Ltd.	5:1, by weight	85	90 minutes	Fast cure, some shrinkage, high heat, transparent
EpoThin	Buehler Ltd.	5:1.95, by weight	27	9 hours	Low viscosity (250 cps at 25°C), low shrinkage, transparent, best for vacuum impregnation
EpoColor	Buehler Ltd.	-	79	90 minutes	Red dyed epoxy
SamplKwick	Buehler Ltd.	-	79	5-8 minutes	Very fast cure, translucent, high heat, some shrinkage
CIBA GEIGY BY 158 resin and CIBA GEIGY HY 2996 hardener	Nordtest Method NT Build 361; Jakobsen et al. [26]	28 grams of hardener to 100 grams of fluorescent resin	27	15 hours	DAYGLO HUDSON YELLOW or equivalent fluorescent dye (such as Epo-dye from Struers) added at 1 percent level by weight of the epoxy
Araldite AY 105 epoxy and hardener 935F	Chemical and Engineering Co., Inc. 221 Brook Street, Media, PA 19063, USA	Equal proportions of epoxy and hardener, 1 part mixture is dissolved thoroughly in 3 volumes of toluene	100	1 hour	Mentioned for rock sectioning by Hutchinson 1974. Refractive index of epoxy medium is 1.55.
Araldite 506 Resin with Hardener HY-2964	Ciba-Giegy Corp. Mktg. Resins Division, Brewster, NY, 10509, 800-222-1906 (Tel)	2 parts resin to 1 part curing agent	-	-	Low viscosity resin good for impregnation, refractive index 1.56,
Epon 828 plus Curing agent "Z" heated to 60°C in a oven and so does the sample	Shell Chemical Co.	Suitable amount of epoxy is mixed with 10-15 ml of curing agent	60	Overnight	Recommended method for impregnating highly friable or porous rocks (Hutchinson 1974). Refractive index of Epon epoxy is 1.59.
EpoFix resin and hardener	Struers Inc. 810 Sharon Drive, Westlake, OH, 44145, 800-321-5834 (Tel)	9:1, by weight (8:1, by volume)	43	8 hours	Low viscosity resin good for impregnation, refractive index 1.57
Epo-Tek 301	Epoxy Technology, Inc., 14 Fortune Drive, Billerica, MA 01821, 978-667-3805 (Tel)	4:1, by weight	-	-	Low viscosity resin, refractive index 1.56.
Permanent mounting media	Various petrographic supply houses	Canada Balsam Resin or Lakeside 70 synthetic resin	-	-	Refractive indices 1.537 for Canada Balsam and 1.54 for Lakeside 70 used for attaching sample to glass slide
Meltmount for grain mounts	Cargille Laboratories, Inc. Cedar Grove, NJ 07009,	-	-	-	Refractive indices 1.539 to 1.704 for grain mounts

	973-239-6633 (Tel)				
Quickmount	Fulton Metallurgical Products Corp., PO Box 118A, Saxonburg Blvd., Saxonburg, PA 16056, 724-265-1575 (Tel)	2 parts powder to 1 part liquid, by volume	-	-	Used for grain mount and encapsulation, a medium viscosity resin
LR White	Ernest F. Fullam, Inc. 900 Alabny Shaker Road, Latham, NY 12110, 518-785-5533 (Tel)	Premixed resin	-	-	Low viscosity good for encapsulation and especially impregnation
Loctite 363	Henkel Loctite Corp. Rocky Hill, CT 06067	-	-	-	365 nm wavelength UV-light cure rapid setting glue for bonding samples to frosted glass slide for thin sectioning
Epoxy EPON Resin 815C, HELOXY Modifier 61, and EPI-CURE Curing Agent 3223 (DETA)	Miller-Stephenson Chemical Company, Inc., George Washington Highway, Danbury, CT 06810, 203-743-4447 (Tel) E. V. Roberts, 8500 Steller Drive, Culver City, CA 90232, 800-374-3872 (Tel);	70% epoxy+25%Heloxy modifier+15% Curing agent by weight	-	6-8 hours	Low viscosity epoxy good for impregnation
Epoxy Resin GCR 324 mixed with EPI-CURE 3234 (TETA)	Gulf Coast S & S, 6322 E. Hwy 332, P. O. Box. E, Freeport, TX, 77541, 408-233-7277; EPI-CURE 3234 is from Miller-Stephenson	12 mL Epi-Cure in 100 mL GCR 324 Epoxy	-	6-8 hours	Good for encapsulation and impregnation
Epoxy Pack Type 301 two-part epoxy resin	Logitech Inc. 810 Sharon Drive, Westlake, OH, 44145, 800-321-5834	4:1, by weight (3:1, by volume)	-	Overnight; (1 hour at 65°C)	Very low viscosity (100 cps at 25°C) resin best for impregnation; refractive index 1.538 to 1.540; shelf life 1 year at room temperature, do not refrigerate

¹The above list of various consumables are for information purposes only. In no case does this imply that these products are the best, nor the only materials available. Colored dyes (usually mixed in resin at high temperature by thorough mixing and kept prior to adding the hardener): (1) Orasol BR Blue BN by Ciba-Geigy Corp., Ardsley, NY, USA – 0.25 gram dye per 100 mL epoxy; (2) Blue Supra Conc. PDR (O5D17) dye from Specialty Coatings & Chemicals, Inc. 7360 Varna Avenue, North Hollywood, CA 91605, 323-875-0233 (Tel); (3) Fluorol Yellow 088 Fluorescent dye from BASF Wyandotte Corp., Holland, Michigan; (4) Epoxy-Dye (fluorescent dye) from Electron Microscopy Sciences.

Table 2: Various grinding and polishing techniques used in petrographic examinations of construction materials

Abrasives	Lubricant	Grinding Papers, Discs, and Polishing Cloths	Purpose
Coarse Grinding			
SiC powders from 60 to 240 grit (268 to 51.8- μ m)	Water, Propylene Glycol, or Light Lapping Oil	Abrasive slurry spread on a fixed or variable speed rotating iron lapping wheel.	Used for removing saw marks on the material.
Fixed SiC abrasives (60 to 240 grits) bonded to paper discs with clear or pressure sensitive adhesive backings	Water, Propylene Glycol, or Light Lapping Oil	Abrasive papers attached to fixed or variable speed rotating iron lapping wheel.	Used for removing saw marks on the material.
Fixed diamond grits from 60 to 240 sizes (268 to 51.8- μ m)	Water, Propylene Glycol, or Lapping Oil	Metal- or resin-bonded diamond discs attached to a rotary iron lapped wheel.	Used for removing saw marks on the material.
Intermediate Grinding			
SiC powders from 320 to 1200 grits (34.3 to 6.5- μ m)	Water, Propylene Glycol, or Light Lapping Oil	Abrasive slurry spread on fixed or variable speed rotating iron wheel.	For successively smooth, flat, fine, matt-finish of the surface.
Fixed SiC abrasives 320 to 1200 grits bonded to paper discs with clear or pressure sensitive adhesive backing	Water, Propylene Glycol, or Light Lapping Oil	Abrasive papers attached to fixed or variable speed rotating iron wheel.	For successively smooth, flat, fine, matt-finish of the surface.
Fixed diamond grits from 400 to 1200 (22 to 6.5- μ m)	Water, Propylene Glycol, or Lapping Oil	Resin-bonded diamond discs attached to a rotary iron lapped wheel.	For successively smooth, flat, fine, matt-finish of the surface.
Fine Grinding			
Loose alumina abrasive powders from coarser to finer grain sizes (the finest size is about 5- μ m)	Water, Propylene Glycol, or Lapping Oil	Abrasive slurry spread on fixed or variable speed rotating iron wheel.	For successively smooth, flat, fine, matt-finish of the surface.
Diamond paste or suspension from 5 to 20- μ m in size	Water, Propylene Glycol, or Lapping Oil (when applied as paste)	Paste or suspension applied to a diamond disc adhered to horizontal rotary grinding/polishing wheel in a single or dual deck tabletop machine.	For successively smooth, flat, fine, matt-finish of the surface.
Diamond size approximately 60- μ m	Water or Glycol or Grinding oil	Diamond bonded grinding cup wheel in a thin sectioning machine.	For precision grinding of a sample down to 35-45 μ m thickness.
Coarse Polishing			
5 to 1-micron sized diamond, deagglomerated alumina, or colloidal silica abrasive	Distilled water for alumina abrasive, water or propylene or ethylene glycol for diamond abrasive	Diamond paste or suspension and alumina or colloidal silica suspension on a hard, napless or low-nap polishing cloth (e.g., Buehler's TexMet).	For polishing a thin section or a small piece of sample for SEM or metallurgical microscope study; to produce a low relief surface.

Intermediate Polishing			
Sub-micron sized diamond, 0.3- μm deagglomerated alumina, or colloidal silica abrasive	Distilled water for alumina abrasive, water or propylene or ethylene glycol for diamond abrasive	Diamond paste or suspension and alumina or colloidal silica suspension on a moderately hard to hard, napless or low-nap polishing cloth (e.g., Buehler's TexMet).	For polishing a thin section or a small piece of sample for SEM or metallurgical microscope study; to produce a low relief surface.
Final Polishing			
0.05- μm deagglomerated alumina powder or colloidal silica	Water or Propylene Glycol or Ethylene Glycol	Soft, napped polishing cloth (e.g., Buehler's MicroCloth).	For fine polishing with a high quality surface finish. MicroCloth produces relief, which helps to identify periclase in clinker, cement, and concrete polished sections.

Table 3: Various etching and staining techniques on polished surfaces of clinker, slag, and cement (adapted from St. John et al. [2] and Campbell [15])

Outline of the Staining or Etching Method and Exposure Time	Observation
10% Potassium hydroxide (KOH)	Alite - Does not etch; Belite-Does not etch; C ₃ A - Bluish gray; Ferrite-Bright white
KOH + 5% Ethyl Alcohol, immersed for 20 secs, followed by washing in 1:1 ethyl alcohol plus water solution and then in isopropyl alcohol solution, buff for 15 sec on MicroCloth wetted with isopropyl alcohol	C ₃ A turns blue
NaOH (2.5 g) + ethyl alcohol (10 mL) + water (40 mL) solution, immersed for 10 sec, followed by washing in 1:1 ethyl alcohol plus water solution and then in isopropyl alcohol solution, no buffing	Darkens alkali sulfates
10% boiling NaOH 20 sec immersion, wash with alcohol	CA in high alumina cement turns blue or brown
KOH (0.1 molar aqueous) immersed for 30 sec, followed by rinse with isopropyl alcohol spray and dry with forced warm air	C ₃ A and alkali aluminate turn blue-brown, alkali-sulfate darkens, free lime turns brown
Dilute salicylic acid stain (0.2 g salicylic acid + 25 mL ethyl alcohol + 25 mL water), immersed for 20-30 sec., followed by alcohol spray wash	Alite and belite are blue green; a solution with isopropyl alcohol (instead of ethyl alcohol) will provide a faster stain on alite than belite and hence distinguishes two phases
Salicylic acid etchant (0.5 g salicylic acid in 50 mL methyl alcohol) 45 sec. etch.	Alite and belite are clear, alite more strongly etched than belite, belite shows lamellar structure
Nital (1.5 mL nitric acid in 100 mL ethyl, methyl, isopropyl, or amyl alcohol) etched for 6-10 sec.	Alite and belite react quickly; alite turns blue to green, belite turns brown to blue – both show detail internal structure. Nital superimposed on a 20-sec KOH etch turns C ₃ A light brown and colors the silicates.
HNO ₃ in alcohol immersed for 2-15 sec.	Alite - Grayish brown; Belite-Blue; C ₃ A - Light gray; Ferrite-Bright white
Hydrofluoric acid (HF) vapor (kept at 20-22°C) exposure for 5-10 sec.	Alite - Buff brown; Belite-Blue; C ₃ A - Light gray; Ferrite-Bright white; C ₂ AS (melilite) in a high alumina cement is colored in HF vapor
10% Isopropyl Alcohol (10mL isopropyl alcohol plus 90 mL water), immersed for 30 sec.	Reacts strongly with alite and weakly with belite; C ₃ A shows a weak reaction
Ammonium chloride (saturated, aqueous), 10 sec.	Hexagonal section of alite perpendicular to the three-fold axis stains light yellow; prismatic section parallel to the c-axis turns blue; zoned alite shows light-blue core and dark-blue rim
Ammonium chloride stain = 1g NH ₄ Cl + 20 mL H ₂ O + 20 mL ethyl alcohol + 10 mL acetone + 150 mL isopropyl alcohol, 10-20 sec.	Alite turns brown, belite is unaffected. After 30-45 sec., Alite turns yellow to yellowish green, belite to brown
Ammonium nitrate solution (1g NH ₄ NO ₃ + 20 mL H ₂ O + 20 mL ethyl alcohol + 10 mL acetone + 150 mL isopropyl alcohol), 20-30 sec. etch.	Alite turns yellow-green; belite turns brown
Ammonium nitrate solution after the salicylic acid stain, 30 sec.	Shows internal structure of alite and belite.
Distilled water, 3-5 sec.	Free lime (CaO) shows rapid etching from dark to iridescent green and blue. C ₃ A turns dark blue. Alite becomes brown, belite is distinguished morphologically. Alkali sulfates are dark.
Water pH 6.8-7 on a saturated microlap cloth, 3 secs. Isopropyl alcohol wash, warm air dry	MgO hard high physical relief, CaO turns dark to green/blue, C ₃ A turns dark blue, C ₃ S turns brown, C ₂ S shows lamellar structure
10% MgSO ₄ solution, etch for 60 secs at 50C	C ₂ S in slag shows striation.
1% borax solution, etch for 30 sec., wash with alcohol	C ₁₂ A ₇ in high alumina cement turns gray
Borax solution (1%, boiling) immersed for 10 sec.	Etches blade-like pleochroite crystals in high alumina cement

Table 4: Various staining techniques in concrete, aggregate, and clinker raw feeds

Specimen	Method	Observation	Reference
Sulfates Lapped, polished or thin sections	Immerse in 2:1 mixture of BaCl ₂ : KMnO ₄ 6% solution for 2 minutes, wash first with water then with saturated oxalic acid.	Ettringite, gypsum, anhydrite, plaster stain pink to purple.	St. John et al. [2]
Feldspar, Quartz, and Calcite for staining raw feeds, aggregates, and thin sections	Two minute exposure to concentrated HF vapor (52%), two minute immersion in sodium cobaltinitrite solution, washing in distilled water, 15 sec. exposure to 10 mL of barium chloride solution, washing in distilled water, two minute immersion in amaranth solution (28 grams of F.D. and C. Red No. 2 pure coal tar dye in 2 liters of water), quick washing is distilled water, and drying.	Plagioclase feldspar (other than albite) stains red, potash feldspar stains yellow, quartz and glassy slags are unstained, calcite stains to various shades of pink, dolomite to deep red.	Hutchinson [1] Campbell [15]
Carbonates Lapped surface Thin sections	10 sec etching in 15% HCl; 30 sec immersion in 1:1 g Alizarin Red S + 0.9 g Potassium. Ferricyanide in 100ml 1.5% HCl. 10 sec immersion in 0.2g Alizarin Red S in 100 ml 1.5% HCl, wash in water.	Calcite stains pink Ferroan calcite stains purple-blue. Ferroan dolomite stains turquoise. Dolomite is unaffected.	Dickson in Hutchison [1]
Evamy stain for calcite, ferroan dolomite, and ankerite applicable to aggregates, raw feeds and thin sections	Immersion in 1% solution of HCL for 2-3 minutes (for thin section 0.2% solution for 30 sec), rinse with distilled water, flood with evamy solution and keep there for 2 minutes, wash with distilled water, dry with warm air. Evamy solution is prepared by mixing 1 gram of alizarin red S with 5 grams of potassium ferricyanide in distilled water, adding 2 ml of concentrated HCl and brining the final solution to 1 liter with distilled water, solution is stored in dark container.	Iron-free calcite stains red, iron-poor calcite stains mauve, iron-rich calcite stains purple, iron-free dolomite not stained, ferroan dolomite stained light blue, ankerite stains dark blue.	Campbell [15], Hutchinson [1]
Copper nitrate staining test for carbonate rock identification, classification (limestone versus dolomite) and detection of impurities including clay or shale inclusions or seams in limestone and dolomite	Immerse aggregates in 0.5M copper nitrate solution (prepared by dissolving 250 grams of copper nitrate trihydrate in 1 liter distilled water and filtering) for 16 hours; to convert stained calcite to the deep blue color, aggregates can be immersed in a strong ammonia solution for a few minutes. Finally aggregates are thoroughly washed in water and air dried.	Splits in clay or shale seams; calcite or dense pure limestone stained deep blue, or greenish if limestone has iron impurities. Dolomite stains very light blue. Non-carbonate or silicate impurities are not colored. Argillaceous limestone or dolomite may show splitting or cracking.	Dolar-Mantuani [45]
Methylene blue staining for detection of smectite in aggregates	Prepare a methylene blue solution containing 4.5 grams of trihydrate methylene blue dye or 4.283 grams of dihydrate dye per liter of distilled water; a uncovered grain thin section	Smectite particles turns blue, muscovite, biotite and other rock forming minerals do not stain,	Higgs [46]

	containing different sieve fractions of aggregate is immersed in the dye solution in a petri dish for 5 minutes, rinse, and then dry. Another method is dye adsorption by finer than No. 200 sieve (75- μ m) fraction of sand grains in a titration process and spot testing of grains on a filter paper until a light blue ring forms around the central dark blue spot of the adsorbed grain.	amount of blue-stained smectite grains can be determined by point counting.	
Alkali-silica gel			
Fractured, grounded or thin section	15 min immersion in 10% uranyl acetate 1.5% acetic acid solution, wash in water.	UV light at 240 nm wavelength gives yellow-green fluorescence of ASR gel.	Natesaiyer and Hover [28]
Fractured, grounded or thin sections	72 hour absorption in 4M cuprammonium sulfate, wash in water.	Gel exudations in voids and cracks stain blue.	Poole, McLachlan and Ellis [29]
Fractured, or aged surface	Aqueous saturated solution of sodium cobaltinitrite followed by a counter stain with rhodamine B base solution.	Bright yellow stain of alkali-rich ASR-gel; pink rhodamine stain of calcium-rich gel.	Guthrie and Carey [30, 31]
Highlighting microcracks by fluorescent dye mixed alcoholic solution	Treatment of a ground section of moist concrete with a fluorescent dye mixed alcoholic solution followed by rinsing excess solution, drying, and observation in a UV light.	UV light at 240 nm wavelength gives yellow green fluorescence of microcracks.	Gran [25], Mayfield [47]

[Note: Consult the original reference before using a particular procedure for the first time]

Table 5: Lists of various equipment and consumables used in petrographic sample preparation¹

Equipments	Manufacturers, Brands, and Models
Wet diamond drilling machine for core extraction	Various manufacturers (can be found in an internet search engine)
Large diamond-edged rotary saws housing 12 to 24 in. diamond blades and small tile or trim saws with 4 to 10 in. diamond blades	Various manufacturers (can be found in an internet search engine, e.g., Covington Engineering, Raytech, Diamond Pacific, Lortone), Buehler (SamplMet 2, Delta AbrasiMet, Abrasimatic 2, OscillaMet, Delta PetroCut, Lapro Slab Saw, IsoMet series), Struers (Exotom, Unitom, Discotom-5, Labotom), Logitech (GTS1 Trim Saw), SBT (Model 650 and 660), Pace Technologies Abrasive Cutters (Metalcut, Servocut), Allied (TechCut 10), Extec Corp (Labcut).
Encapsulation and Vacuum-impregnation Units	Struers (Epovac), Logitech (IU-20, IU-30), Buehler (Cast N' Vac system and 1000).
Large horizontal rotary grinding/lapping machine – stand alone or tabletop unit with or without sample holding fixtures (circular conditioning rings and weights)	Covington Engineering (16-in. horizontal single and two-speed lapping units), Diamond Pacific (12 and 18 in. rotting disc flat laps), ASW Diamond Trust (SW Series Lapping Machines), Lapmaster International
Tabletop, single or dual-wheel, single or variable speed grinding and polishing machines with or without automated sample holding fixtures (heads)	Buehler (MiniMet, Polimet, MotoPol, MiniMet 1000, MetaServ 2000, Alpha and Beta Units, EcoMet series, AutoMet, Petro-Pol, PowerPro Family, Phoenix 4000 System); Leco (Spectrum System 1000 and 2000); Struers (RotoPol, RotoForce, LaboPol, LaboForce, Abramin, Abraplan, Abrapol, Planopol); Logitech (PS 2000, WG2, PM5, and LP 50 precision lapping/polishing machines); SBT (Model 900, 910, 920, TL-MC1); Pace Technologies Manual and Automatic Polishing Machines (Gripo, Digiset, Gripomat, Digimat, Forcimat), Lapmaster International, Allied (DualPrep, OptiPrep, MultiPrep, M-Prep, and TwinPrep units with AP-3 power head and AD-4 fluid dispenser), Extec Corp (Labpol).
Automatic Abrasive Dispensing System	Buehler (PriMet, MetLap), Leco, Pace Technologies (Lubomat, Dispomat), Struers (Multidoser).
Vibratory lapping machine for grinding, polishing, and ultrathinning	Buehler (VibroMet), Other Units from Covington Engineering, Diamond Pacific, etc.
Automated thin sectioning (precision-sectioning and/or grinding) machine housing precision blade and/or diamond grinding cup wheel or plate	Buehler, Ltd. (Isomet low speed saw, Isomet Plus, Isomet 1000, Isomet 5000, Petro-Thin thin-sectioning system); Struers (Accutom, Minitom, and Discoplan-TS units); Logitech (PS 2000, WG2, PM5, and LP 50 precision lapping/polishing machines with PLJ2 and PLJ7 precision lapping jigs); SBT (Model 650 and 660); Wards Natural Sciences (Ingram-Ward Thin Section Equipment Ingram Model 103 saw and model 303 grinder); Hillquist's thin-sectioning machine; Microtec Engineering Lab (Micro-Trim Automatic Thin Sectioning Machine); Pace Technologies Diamond Wafering Saws (Finocut, Micracut), Allied (TechCut 4 and 5, Trim Saw). Germann Instrument's thin-sectioning machine has roller grinding components. Buehler's Petrol-thin, Hillquist's unit, and Ingram-Wards unit have separate, parallel 8-in. diamond wafering blade and diamond cup wheel on the same compact tabletop unit for precision sectioning and grinding.
Thin-section Bonding Fixture	Buehler (PetroBond), Logitech (BJ2, BJ6, BJ9, and BJ12 thin

	section bonding jigs)
Miscellaneous equipments - Ultrasonic Cleaner, Oven, Hot plate, Vacuum Impregnator, Sieve Shaker, rock trimmer, hood with exhaust system, sink and drain, water supply and drain lines for sectioning, grinding, polishing equipments, re-circulating systems for sectioning or lapping units, tabletop hot air blower or hand held hair drier	Various manufacturers (e.g., internet search engines, Fisher Scientific, Gilson, etc.).
Consumables	Manufacturers
Loose grinding and polishing abrasives	Buehler, Leco, SBT, Extec, Allied, Pace Technologies, various manufacturers in the internet
Fixed Grinding Abrasives as PSA backing discs or paper for belt grinder	Buehler, Leco, SBT, Struers, Logitech, Extec, Allied, Pace Technologies,
Polishing cloths and diamond polishing pastes and suspensions	Buehler, Leco, SBT, Pacific Diamond, Extec, Allied, Pace Technologies, various manufacturers in the internet
Thin section slides (27×46 mm, 50×75 mm)	Wards Natural Sciences, Fisher Scientific, Erie Scientific, Logitech, Buehler, Electron Microscopy Sciences
Magnetic diamond discs for lapping (18-in. to 24-in.)	Pacific Diamond, ASW, Extec, Allied, Pace Technologies, various manufacturers in the internet
Magnetic 8-in. to 12-in. resin-bonded and metal-bonded diamond discs for grinding	Buehler, Leco, Extec, Allied, Pace Technologies, various manufacturers in the internet
Low-viscosity Sectioning and Lapping Oil	Buehler, Leco, Diamond Pacific, Extec, Allied, Mobil Oil Corp, Fisher Scientific
Solvents for Polishing	Fisher, Buehler, Leco, Extec, Allied, various manufacturers in the internet
Deagglomerated alumina (0.3 and 0.05- μ m) fine polishing powders	Buehler, Leco, Extec, Allied
Various castable epoxy resins and dyes	See Table 1
Miscellaneous equipments - Disposable petri dish, aluminum foil, wax paper, disposable aluminum containers, plastic and metal cylindrical molds or cups, metal trays, fine and coarse permanent markers, carpet tape, dropper bottles, paper towels	Miscellaneous (Fisher Scientific, Electron Microscopy Sciences, etc.).
Consumables for electron microscopy	Electron Microscopy Sciences

The above list of various equipments and consumables are for information purposes only. In no case does this imply that these products are the best, nor the only materials available. Manufacturers' addresses and phone numbers: (1) Buehler Ltd.; 41 Waukegan Road, Lake Bluff, IL 60044; 847-295-6500; (2) Struers, Inc., 810 Sharon Drive, Westlake, OH 44145, 440-871-0071; (3) SBT - South Bay Technology, Inc., 1120 Via Callejon, San Clemente, CA 92673, 800-SBT-2233; (4) Microtec Engineering Laboratories, Inc., PO Box. 636; Clifton, CO 81520; 970-434-8883; (5) Wards Natural Science Establishment, Inc. PO Box 1712, Rochester, NY, 14603, 800-962-2660; (6) Mobil Oil Corp. Products and Technology Dept., 3225 Gallows Rd., Fairfax, Virginia, USA 22037, 703-849-3265; (7) McCrone Associates and Companies, 850 Pasquinelli Drive, Westmont, IL 60559-5531 800-622-8122; (8) Diamond Pacific Tool Corporation, P. O. Box 1180, Barstow, CA 92312, 800-253-2954; (9) Covington Engineering Corp., P. O. Box 35, Redlands, CA 92373, 877-793-6636; (10) ASW Diamond Trust, P.O. Box 116, Gorman, CA 93243, 800-825-2792; (11) Electron Microscopy Sciences, 321 Morris Rd. Box 251, Fort Washington, PA 19034, 800-523-5874; (12) Leco Corporation, 3000 Lakeview Avenue, St. Joseph, MI, 49085-2396, 269-983-5531; (13) Pace Technologies, 1802 W. Grant Rd. Suite 102, Tucson, AZ 85745, 888-PACE-654, (14) Lapmaster International, 6400 West Oakton Street, Morton Grove, IL 60053, 877-352-8637; (15) Hillquist Inc, 1144 S. Bannock Street, Denver, CO 80223, 303-722-9766 (Tel), (16) Allied High Tech Products, Inc. 2376 East Pacific Place, Rancho Dominguez, CA 90220, 800-675-1118; (17) Extec Corp. 99 Phoenix Avenue, P.O. Box 1258, Enfield, CT 06083-1258, 800-54-EXTEC.

Table 6: Some common petrographic sample preparation steps for clinker, cement, concrete, aggregate, mortar, and other construction materials¹

Sample Preparation	Clinker	Cement	Aggregate (after sieve analysis)	Concrete and Masonry Units	Mortar	Plaster and Gypsum Products
Washing and drying	-	-	Optional	Optional	Drying	Optional
Encapsulation	1A, B	1A, B	1A	1A, B If fragile	1A If fragile	1A If fragile
Vacuum Impregnation	2A, B	2A, B	2A	2A, B Of trimmed or impregnated section	2A Of trimmed or impregnated section	2A Of trimmed or impregnated section
Sectioning/Trimming	3A, B	3A, B	3A	1A, B	3A	3A
Coarse Grinding (with SiC/diamond grit)	4A, B	4A, B	4A	3A, B	4A	4A
Fine grinding (320 and 600 SiC/diamond grits)	5A, B	5A, B	5A	4A, B	5A	5A
Coarse Polishing (with 5- μm Al_2O_3 /diamond abrasive on hard cloth)	6A	6A	-	5A	Optional	Optional
Fine Polishing (with 0.3 and 0.05- μm Al_2O_3 /diamond abrasives on cloth)	7A	7A	-	6A	Optional	Optional
Bonding to glass slide	6B	6B	6A	5B	6A	6A
Precision sectioning	7B	7B	7A	6B	7A	7A
Precision grinding down to μm -size thickness	8B	8B	8A	7B	8A	8A
Coarse and fine polishing of thin sections	9B	9B	-	8B	Optional	
Oil-immersion mounts	1C	1C	Fine size, 1B	1C	1B	1B
Grain thin sections	1D	1D	Fine size, 1C	1D	1C	-
Grain mounts in epoxy	1E	1E	Fine size, 1D	1E	1D	-
Pulverization for XRD	1F	Use as is	1E	1F	1E	1C

¹A, B, C, etc. indicate separate modes of sample preparation with separate subsets of a sample. For each subset, successive steps of a sample preparation technique are shown numerically.

Table 7: Various modes of petrographic examinations of construction materials

Sample Preparation	Stereomicroscope	Metallurgical Microscope	Petrographic Microscope	SEM	XRD	Image Analysis
As received	1	-	-	2	-	1
Fresh fracture section	1	-	-	2	-	1
Oil-immersion mounts	-	-	1		-	1
Powdered sample	1	-	1	1	1	1
Saw-cut sections	1	-	-	2	-	1
Ground sections	1	-	-	2	2	1
Polished sections	2	1	-	1	-	1
Thin sections	2	-	1	-	-	1
Grain thin sections	-	-	1	-	-	1
Polished thin sections	-	1	1	1	-	1
Fluorescent alcohol treated ground sections	2	2	-	-	-	1
Fluorescent thin sections	-	-	1	-	-	1

1 = Primary method of examination, 2 = Secondary method of examination, Image analysis system includes image capture device (scanner, camera, digital camera, micro video camera) and an image analysis software.

Table 8: Examples of misinterpretation of microstructure due to the artifacts from sample preparation

Observation	Preparation artifact	Misinterpretation
In Thin Sections		
Microcracks in paste	Oven-drying	Drying shrinkage cracks
Grain cracking	Ultra-thinning	Alkali-silica reaction
A gap or separation between a floor covering and concrete	Drying, Thin sectioning	Moisture condensation on floor
Fly ash in concrete	Air bubbles not impregnated with epoxy due to preparing thin section from beyond the zone of impregnation	Excessive abundance of fly ash
Voids and cracks in a thin section that are free of any reaction products	Washing of alkali-silica reaction gel during excessive cleaning of sample	No ASR in concrete
No salt deposits at the surface	Washing of water-soluble salts	Absence of efflorescence or salt hydration distress in masonry or concrete
Deep carbonation	Prolonged exposure to atmospheric CO ₂ during sample preparation	Poor quality concrete
Patchy appearance of paste in dye-mixed epoxy impregnated thin section	Incomplete epoxy impregnation or deep grinding of impregnated surface	Inadequate mixing of batch or tempering water in the concrete; error in water-cement ratio determination
Bright yellow fluorescence of thin section in a petrographic microscope	Too much dye in epoxy, too thick section	Provide erroneously high water-cement ratio in image analysis (Jakobsen et al. [26])
In Polished Sections		
Cracks, scratches, holes, pits, effects of interference from residual grinding, polishing, etching, and cleaning liquids	Artifacts of polished and thin section preparations of clinkers	Misinterpretation in clinker phase identification, distribution, reactivity, and chemical attack from exudation of cleaning or polishing solutions, etch halo (see Campbell [15])
Carbon, aluminum peaks in x-ray microanalysis in SEM	Inadequately cleaned polished surface on which remains of alumina or diamond polishing abrasives are present	Misinterpreted as elemental composition of the material being examined
In Lapped Sections		
Excess irregularly-shaped or very fine voids during determination of air void parameters by automated image analysis	Voids from grain plucking, voids in aggregates that are filled with the void-filling white paste in the binary image, or surface defects [Cahill et al. [33]]	Erroneously high air content and errors in air void parameters
Indistinct (eroded or crumpled) margins of air voids; poor surface finish	Inadequate grinding of the surface	Erroneously high or low air contents and errors in air void measurements