SAMPLE 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 phenolic or epoxy-based thermosetting resins, or methyl methacrylate-based thermoplastic resin), *castable mounting method* is more common for encapsulating both metallurgical and earth or building 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 cuing, 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 100ºC with the dye helps thorough mixing), (b) pre-mixed red, green, or blue coloring paste thoroughly dissolved in epoxy at 100ºC, (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. 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 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. 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). 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.

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. For fragmented and powdered samples like fragile rocks, 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 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 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.