Sample Preparation Laboratory

CMC, Inc.

CMC has a state-of-the-art sample preparation laboratory, which houses various equipment for impregnation, encapsulation, sectioning, grinding, polishing, thin-sectioning, pulverizing, sieving, and conductive coating of materials for petrographic examinations and chemical analysis.

Sectioning -The sample preparation laboratory uses a heavy duty sectioning machine manufactured by Covington Engineering, which has a reliable 20-in. diameter diamond saw. The machine can cut up to a 6-in. depth of sample and up to a 10 in. long sample. Regular tile saws are used for trimming purposes. The tile saws use 10-inch diameter continuous rim diamond blades, 0.032 to 0.045 inch in thickness. Since all these saws use water as the coolant (mixed with a corrosion inhibitor), water sensitive materials (e.g., cement clinker) are not processed in these saws. Water-sensitive materials are sectioned either by using an oil-coolant based saw or with Buehler's Isomet 1000 precision saw described below. The type of diamond blade used is very important to reduce the deformation of the sample from sectioning. Following a detailed visual preliminary examination and photographic documentation of the samples, selected areas of the samples are chosen for sectioning purposes. Cores and saw-cut sections of samples received for petrographic examinations are first sectioned into small pieces by using these saws.

Encapsulation and Vacuum Impregnation - For brittle, soft, porous, or fragmented materials, vacuum impregnation with a low-viscosity epoxy improves the overall integrity of the sample and helps further sample preparation steps. CMC uses several different epoxy-hardener combinations sometimes mixed with a blue or a fluorescent dye to highlight cracks, pore spaces, and voids in material. Sample is impregnated while in the vacuum and cured within 4 to 12 hours at room temperature (some UV-cured epoxy can hardened in an hour while exposed to the long wavelength UV light). If the material received in the laboratory is too fragmented to do the initial sectioning, a portion of the material is first impregnated with wax or an epoxy and then used for sectioning and subsequent grinding.

CMC uses Buehler's vacuum impregnation unit (Cast N' Vac 1000), epoxy dispensing unit (EpoEase), and a custom-made unit for simultaneous encapsulation and vacuum impregnation of multiple samples. Samples are encapsulated in flexible cold mounting molds (EPDM, urethane, or silicone molds), or rigid, custom-made metallic and plastic molds coated with a release agent.

Lapping and Grinding - For coarse, intermediate, and fine grinding, CMC uses a horizontal rotary floor-standing lapping machine from ASW Diamond Products (Model SW-1800) with an 18-in. heavy-duty precision ground steel plate for accepting various metal and resin-bonded diamond magnetic discs (from grit sizes of 60, through 260, 320, 600, and 1200 discs). The machine houses a 3/4 HP Dayton Motor, heavy duty steel shaft with welded flange, removable steel guard, and forward/reverse motion switch. Large concrete or other samples are often ground to a smooth flat, and matt-finished surface by hand, by holding the sectioned surface on the successively finer diamond discs lubricated with water. The machine accepts an additional sample holding fixture at the top for automatic grinding of multiple small samples. Fine grinding is done by two Lapmaster's Model 15 compact, tabletop lapping machines, each containing three conditioning rings for simultaneous grinding of six to nine samples. Both Model 15 units accept a rigid, lightweight cast aluminum base for polishing and serrated cast iron lapping plate for grinding operations. The "ring lapper" configuration provides a high standard of flatness and surface finish on a continuous basis through specific positioning of the conditioning rings. Both machines have an automatic digital cycle timer for operation without supervision, and automated, controlled slurry discharge facilities. A variety of micarta and delrin discs with openings of various shapes are used within the conditioning rings for accommodating various circular and rectangular samples. Alumina or SiC abrasive (9.5 or 15 micron size) in water or oil is used for fine grinding in the Lapmaster models.

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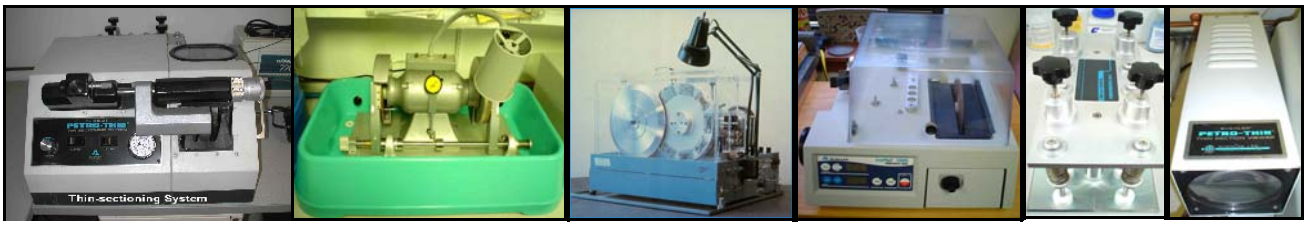
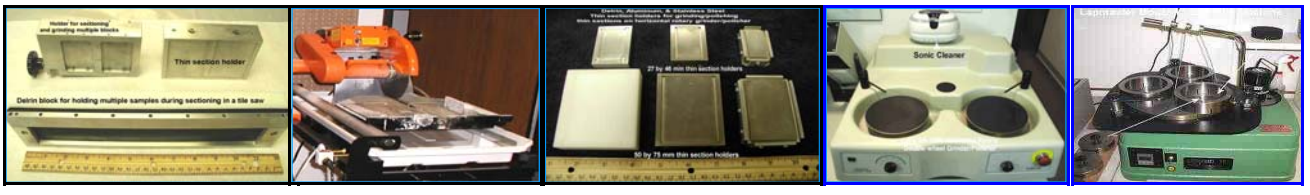
Polishing - CMC uses Leco's Spectrum 1000 dual-wheel (8-in. diameter) horizontal rotary grinder/polisher, Logitech's microprocessor controlled PS2000 automatic precision polishing machine (12-in. diameter grinding and polishing wheels), and Logitech's PM2A-VS2 polishing-vacuuming assembly for fine grinding and polishing of a single or multiple samples. Leco's Spectrum 1000 uses various diamond bonded magnetic discs (320, 600 grits) for coarse and fine grinding; and pressure-sensitive adhesive-backed magnetic discs for fine grinding and polishing. Polishing cloths are charged with 5,1, 0.3, and 0.05 micron deagglomerated alumina oxide or diamond suspension. A rugged 3/4 HP motor drives the simultaneous operation of both 8-in. wheels with built-in timer and variable speed control options. Logitech's machine uses deagglomerated alumina suspensions for grinding and polishing. Water, glycol, and oil are used as lubricants and 5, 0.3, and 0.05 micron diamond or alumina is used as abrasive A large number of clinker, cement, concrete, and mortar samples can be prepared simultaneously with these machines. Logitech and Lapmaster machines are also used for thin-section preparation by using precision digital thin-sectioning jigs on the lapping wheels. Two Lapmaster's 15-inch polishing machines are also used for simultaneous grinding of multiple samples. A variety of sample holders and fixtures are used for grinding and polishing operations.



Thin-sectioning - CMC uses various manual, semi-automatic, and fully automatic thin-sectioning devices for preparation of small (27 × 46 mm) and large-area (50 × 75 mm) thin sections of construction materials. CMC houses thin-sectioning machines manufactured by Buehler (Petro-Thin and Isomet 1000), Logitech (PS2000 and PM2A-VS2), Hillquest, and Microtek Engineering (Microtrim III). The Buehler and Hillquest machines are semi-automatic, which prepare one sample at a time, whereas Logitech and Microtrim machines can prepare multiple samples simultaneously. The Petro-thin and Hillquest machines house an 8-in. precision diamond saw and a parallel diamond-bonded grinding wheel for thin-sectioning, followed by thin-grinding of the material, respectively. Buehler's Isomet 1000 precision saw containing a 5-inch diamond wafering blade is used for thin sectioning of water-sensitive materials (e.g., clinkers; oil is used for sectioning). CMC uses various local machine shops to prepare numerous in-house sample holders and sample preparation fixtures, which assist greatly in thin section preparation. Thin-sections (20 to 25 microns thickness) of concrete, mortar, tile, bricks, and other construction materials are capable of transmitting lights and detecting various inorganic constituents from their characteristic optical properties. Thin-sectioning is an important part of petrographic examinations, which provides a wealth of information on the microstructure and mineralogy of a building material, and evidences of deterioration in the material. For highlighting cracks, pore spaces, and voids in samples, thin sections are often prepared either by vacuum-impregnating the sample with a blue dye or with a yellow fluorescent dye mixed low-viscosity epoxy. Up to 12 finely ground surfaces of samples to be thin sectioned are glued to frosted glass slides with Buehler's "Petro-Bond" bonding fixture. The final thickness of thin section is verified with Buehler's "Petro-Vue" thin sectioning viewer.

Large-area Thin-sectioning - Up to 100 by 150 mm size thin sections of concrete, masonry, aggregate, stone, and stucco samples are prepared by: (a) encapsulating the oven-dried sample in a large-size reusable flexible silicone mold and vacuum impregnating with a low-viscosity epoxy resin, (b) hand-grinding of the impregnated sample on a horizontal rotary grinder (with metal or resin bonded diamond disc) to expose the surface of interest, (c) fine grinding the surface on a Lapmaster grinder with 9.5-micron alumina abrasive, (d) bonding the cleaned, dry, finely ground surface to a large-size clean, dry, frosted glass slide, (e) sectioning of sample on glass slide by holding the slide with a sample holder and thin-sectioning the sample in a tile saw (equipped with a thin, 10-in. diameter diamond blade of 0.015 in. thickness), (f) fine grinding of the sectioned sample on the frosted slide with a slide holder on a horizontal rotary grinder (with 240-grit metal or resin-bonded diamond disc) until the sample is less than a millimeter thick, (g) controlled grinding of sample on Lapmaster grinder with 9.5-micron alumina abrasive, and (h) final hand grinding of sample on a glass plate or a steel mesh charged with 5 or 9.5 micron size alumina abrasive.



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Precision Sectioning, Grinding, Polishing, and Thin-sectioning of Clinker Samples - For clinker and other water-sensitive materials CMC uses Buehler's popular Isomet low speed and Isomet 1000 precision saws housing 5 and 7 in. diameter diamond wafering blades, respectively. The blades are lubricated with a low viscosity oil (Mobil or baby oil). Grinding is done with 320 and 600 grit SiC abrasives in oil on the Buehler's Minimet and Minimet 1000 grinder/polishers. Polishing is done on Minimet with TexMet polishing cloth by using 5 and 0.3 micron alumina suspensions in glycol (with optional final polishing with 0.05 micron alumina on Microcloth). A bench top 8-in. diameter horizontal rotary wheel grinder/polisher is used for hand grinding and polishing water-sensitive samples with micron and sub-micron sized diamond, alumina, or SiC abrasives on pressure-sensitive adhesive backed papers or cloths. Grain thin section and polished grain mounts are prepared by using the mounted grains on frosted glass slides and holding the slide with thin section holders on the horizontal rotary grinder. Slow and continuous polishing and ultra-thin sectioning can be done by Buehler's Vibromet 2 vibratory polisher.

Sample Preparation for Air-Void Analysis by ASTM C 457 and Automated Image Analysis - For air-void analysis in hardened concrete by the procedures of ASTM C 457, a representative sample is first sectioned by using a slab saw or a table saw and then ground to a smooth, flat, surface by successively finer grit diamond discs (e.g., 60 and 240 grit metal-bonded diamond discs, followed by 600, 1200, and 3000 grit resin-bonded diamond discs with intermediate thorough cleaning of slurries). For rapid measurements of air void parameters in hardened concrete by a binary image analysis technique, a conventionally prepared lapped section of concrete 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, cornstarch or barium sulfate powder, pressed into the voids, Voids in the aggregates are darkened by a marker pen. The prepared surface shows white air voids in black background, which is then photographed by an image-grabbing device (digital camera, high resolution flatbed scanner, or a stereo microscope with an attached video camera). The image capturing process by the scanner, or camera collect binary images of air void distribution, which is then processed by appropriate softwares to determine the air void parameters. CMC uses both the flat-bed scanner and the stereomicroscope equipped with a video camera for image capturing procedures and various softwares for calculations of air void parameters. The data, so obtained, is often compared with the air void parameters of the sample surface prior to black-and-white treatment determined by the conventional (ASTM C 457) methods.

Etching and Staining Techniques - CMC uses various etching and staining techniques for microscopical examinations of clinker materials, aggregates, and concrete (see the Reference mentioned below for detailed procedures). Treatment of a freshly fractured surface of a concrete by uranyl acetate, cuprammonium sulfate, or sodium cobaltinitrite solutions are sometimes helpful for rapid identification of alkali-silica reaction gel in the concrete.

Pulverizing and Sieve Analysis - CMC uses a Sepor Mini-thor ring pulverizer for preparation of samples for chemical analyses (e.g., chloride and sulfate analysis), and x-ray diffraction. The chromium ring pulverizer reduces contamination and provides a consistent 100 micron-size fractions from 1/2-in. size fragments in 10-15 seconds. Both dry and wet pulverizing can be done on a wide variety of samples for XRD, XRF, wet chemistry, and other instrumental methods. CMC has an automatic sieve shaker with various sieves for sieve analysis of aggregates prior to petrographic examination. For XRD and XRF analysis, CMC uses the Sepor Mini-thor ring pulverizer to ground samples to a fine powder, and then further pulverization to ~10 micron size particles in McCrone Micronizing Mill.

Conductive Coating of Sample for SEM Studies - CMC uses the Desk II Denton Vacuum cold sputter/etch unit for gold and carbon coating of samples for SEM examination. Desk II gives a uniform, conductive, fine-grained 100A coating in less than three minutes from pump down to venting. In the etch mode, the Desk II cleans non-delicate specimens contaminated with molecular films of water and oil to provide a clean substrate for sputtered atoms to adhere to.

Other Miscellaneous Equipments - In addition to the above equipments, the sample preparation laboratory houses various digital balances (calibrated annually), ovens, hot plates, UV-light fixtures, and other equipments, which are simple yet indispensable tools for materials testing.

For various sample preparation techniques, see

Jana, D., Sample Preparation Techniques in Petrographic Examinations of Construction Materials: A State-of-the-art Review, Proceedings of 28th International Cement Microscopy Association, Denver, CO, pp. 23-70, 2006.