

TECH NOTES

Petrographic Examination Methods

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Petrography is the study of rocks and minerals using a microscope. Traditionally, petrography was limited to the identification of rocks, minerals and ores and to the characterization of properties such as cleavage, twinning, reflectance, and so forth. Today, however, petrographic techniques are employed to analyze many materials other than minerals, for example, ceramics, glass, concrete, cement, soils, biomaterials, polymers, to name just a few.

The birth of petrographic analysis began with an English scientist, Sir Henry Clifton Sorby. His first thin section, that of a calcareous rock, was prepared in 1849. Although Sorby did not record the details of the procedure he used at that time, he did so later in 1868.

Sorby's method of preparation was quite crude compared to the methods used today. He would simply fracture the rock as thinly as possible and attach the chip to a glass slide using Canada balsam. For rough grinding, he used emery or "congleton" stone and for finishing the surface a "waterof-ayer" stone (a fine grain sandstone). Sorby was very conscious of the surface he produced; he strived for a surface that was essentially scratch-free and damage-free. His concern for minimizing relief is still an important concern in modern day specimen preparation. Sorby's work on metallic meteorites made him interested in preparing metallic specimens. "It was a natural thing," wrote Sorby in 1897 "that I should be led from the study of the microscopical structure of rocks to that of meteorites, and in order to explain the structure of meteoric iron, I commenced the study of artificial iron."

Because of his pioneering work, Sorby is considered the father of both petrography and metallography. Others had tried to look at the structure of minerals and metals before him, but they could not prepare specimens to see the true structure. Sorby was the first to understand this problem and learn how to solve it.

There are two types of specimens prepared for petrographic analysis, thin sections and polished bulk specimens. Polished bulk specimens are similar to metallographic specimens in that the surface is prepared for examination with a reflected light microscope. Thin sections, on the other hand, are extremely thin, generally 30µm or thinner, and are observed with a transmitted polarized light microscope. The following chart, Figure 1, describes a general procedure required to prepare both thin sections and polished bulk specimens.

To prepare a polished bulk specimen or a thin section, the specimen must be subjected to a series of steps consisting of sectioning, impregnation and encapsulation, grinding, and polishing. The primary purpose of specimen preparation is to reveal the true

microstructure; so care must be exercised in all preparation stages to avoid altering or masking the true microstructure.

Figure 1. Petrographic Preparation Procedures

Pre-Preparation

- Sectioning
- Grinding one surface flat
- Vacuum Impregnation & Encapsulation

Grinding & Polishing Polished Sections

- Grinding
- Polishing
- Reflected light microscopic examination

Thin Sections

- Mounting (attaching to slide)
- Resectioning
- Grinding
- Polishing
- Transmitted light microscopic examination

Preparing Bulk Polished Sections

Sectioning

The first step in the specimen preparation process is sectioning, which is performed for the following reasons:

- to obtain a manageable size specimen from the parent material
- to reduce the thickness of the specimen so that grinding time is reduced (as in the case of preparing thin sections)
- to expose the surface of interest

Historically, the process of sectioning was considered unimportant. In reality, however, sectioning can be the most damage-inducing step in the whole process of specimen preparation. This is especially true when sectioning brittle and poorly consolidated materials. Any severe damage induced in sectioning will be difficult to remove in the steps that follow sectioning, that is, grinding and polishing. Because sectioning can be a high damage-inducing step, it is important that the proper saw and blade are selected to minimize this damage. Diamond wafering saws, such as the ISOMET® 4000, are ideal for cutting sensitive materials. The cutting parameters, such as the force applied for sectioning, blade speed and feed rate, can all be controlled precisely. Equally as important as the saw is the blade chosen for sectioning. Figures 2 and 3 show the differences in deformation produced when cutting a brittle material (graphite composite, Figures 2a and b) and a ductile material (copper, Figures 3a and b) using two different types of blades.

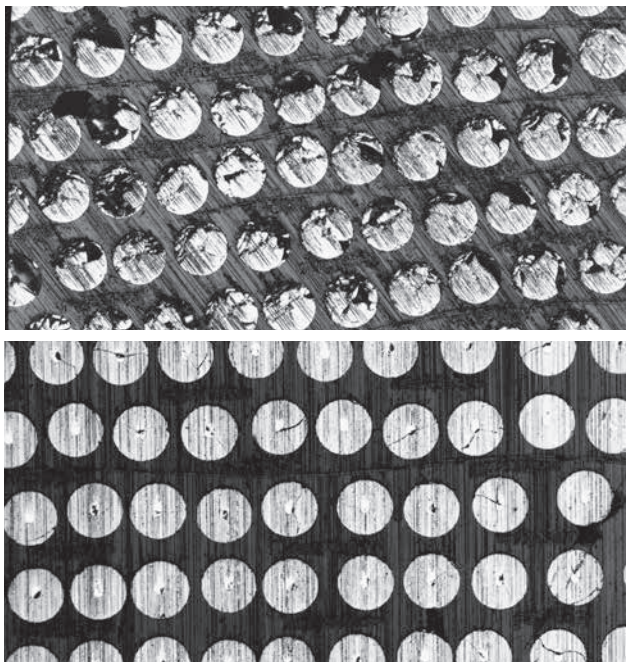


Figure 2a. (top) Graphite fibers in epoxy matrix cut with 20HC blade. Figure 2b. (bottom) Graphite fibers in epoxy matrix cut with 10LC blade.

Because of the wide range of materials that can be sectioned with a wafering saw, it is important to choose the most suitable blade. The following, Table 1, simplifies blade selection.

As a general rule, the quality of the surface finish is proportional to the abrasive size on the blade. For example, the 20HC blade will cut faster, but it will also produce a rougher surface than the 10LC blade.

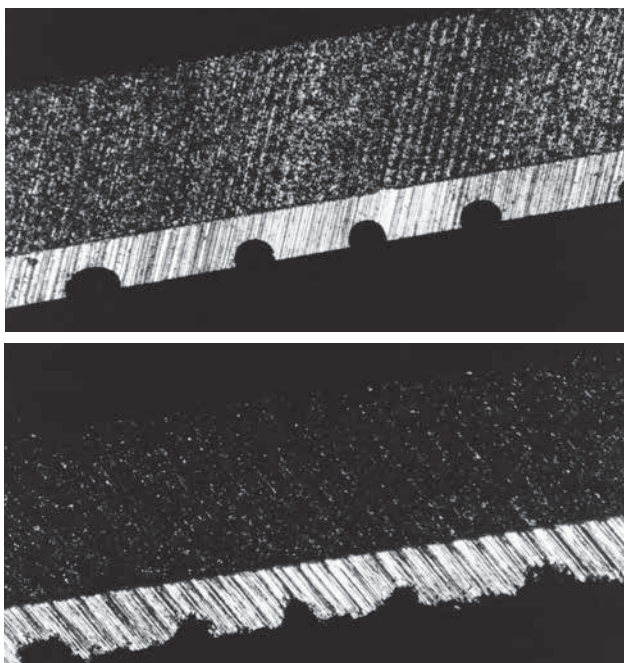


Figure 3a. (top) Copper coating on ceramic substrate cut with 10LC blade – 100x. Figure 3b. (bottom) Copper coating on ceramic substrate cut with 20HC blade.

Encapsulation

Once a specimen has been cut, it should be thoroughly cleaned and dried. Materials that may have pores and cracks, or are poorly

Table 1. Characteristics and Applications of Blades for Precision Sectioning.

Blade Type	Blade Use
30HC	For cutting polymers, rubber, soft gummy materials, embedded bones
20HC	Aggressive blade for sectioning various materials rapidly (expect more deformation than series 15 blades)
15HC	General purpose blade for most materials
15LC	Ceramics, rocks, minerals
10LC	Brittle, delicate and poorly consolidated materials (good surface finish but slower in cutting)
5LC	Very brittle and very delicate materials such as silicon wafers (very slow cutting)
ISOCUT	Ferrous materials, cobalt, nickel and superalloys

consolidated, must be vacuum impregnated with epoxy. Epoxies that have a low viscosity, such as EPO-THIN® resin, are ideal for impregnation. EPO-THIN epoxy resin has a slow curing rate. This can, however, be accelerated by placing the specimen in an oven or on a hotplate (if an oven is not available). The temperature of the oven or the hot plate and epoxy should not exceed 45-50°C.

For specimens that have fine pores that are not interconnected, use of coarse abrasives is not recommended because this may remove the epoxy-filled pores and may expose pores in which epoxy did not penetrate. (Figure 4 shows this in diagrammatic form.) To easily distinguish pores and cracks with the microscope, EPO-COLOR™ epoxy resin may be used. EPO-COLOR resin appears bright red when using dark field or polarized light illumination, Figure 5. Once the epoxy is completely cured, the specimen is ready for grinding.

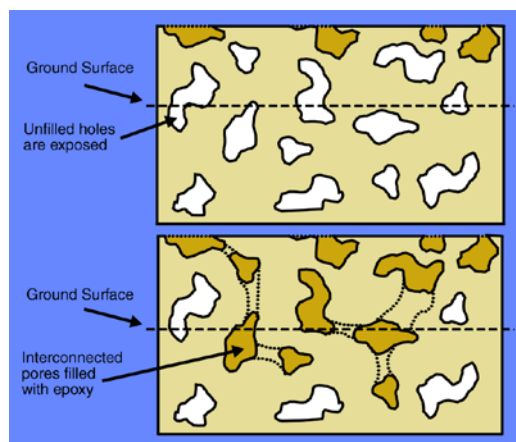


Figure 4. Effect of Grinding on Pores.

Grinding

Grinding is performed to remove deformation induced in sectioning and to make the surface flat. Grinding is generally performed using either fixed abrasives where the abrasive particles are bonded to a substrate and are not free to move; or, by loose abrasives where the abrasive particles are not bonded to a substrate but are free to roll around as they abrade the surface. This latter procedure is often called lapping.

The mechanisms involved in these two processes are quite different. In general, fixed abrasives are more aggressive and remove much more material per unit time for the same abrasive size than loose abrasives. However, they tend to produce somewhat more deformation at the surface. Lapping is noted for producing good flatness. For either process, the size of the abrasive determines the cutting rate and the damage depth. The coarser the abrasive used, the faster the removal rate, but the greater the damage depth at

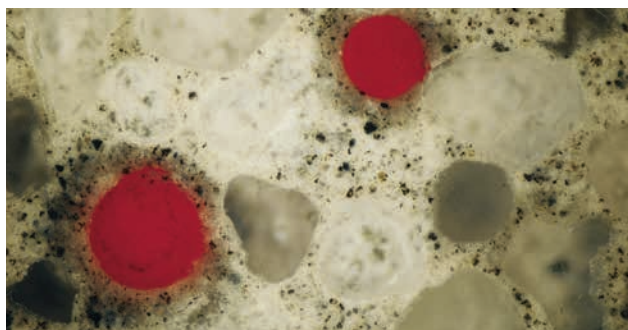


Figure 5. EPO-COLOR in darkfield illumination showing porosity in concrete – 400x.

the surface. Conversely, the finer the abrasive, the lower the removal rate, and the lower the sub-surface damage depth. When grinding poorly consolidated, weak or brittle materials, use as fine an abrasive as practical that removes the existing damage in a reasonable time yet produces less damage than a coarser abrasive.

Fixed abrasives in the form of diamond discs or abrasive papers are used for grinding. Diamond discs have a long service life but may be far too aggressive for many sensitive-to-prepare materials, such as weak sandstones, shales, and clays. Silicon carbide papers are fine for grinding soft materials; however, they have a short service life. Abrasive papers generally exhibit a high cutting rate initially, but the cutting rate decreases rapidly.

There are two different types of diamond discs that are used commonly for grinding petrographic specimens: metal bonded discs and resin bonded discs. The metal bonded disc removes material faster, but produces rougher surfaces. The resin bonded disc removes less material, however, the surface finish is much finer than produced by the metal bonded disc.

The BuehlerHercules™ disc is a newly designed grinding surface that has a wide range of applicability. It can be used with coarse as well as fine abrasives to obtain minimum sub-surface damage, good cutting rates and good surface finishes. Unlike abrasive papers, they maintain a consistent cutting rate over the life of the disc. The BuehlerHercules discs are used with diamond abrasive slurries.

Diamond, because of its extreme hardness and resistance to fracturing, abrades faster and removes more material than other abrasives. It also produces a more consistent surface finish. Diamond stands alone in hardness and abrasion resistance amongst other abrasives. The following chart shows the approximate hardness of various popular abrasives.

Table 2. Hardness of Commonly Used Abrasives

Abrasive	Approximate Knoop Hardness	Mohs Hardness
Diamond	7800-8000	10
Silicon Carbide	2300	9
Aluminum Oxide	2100	9

Diamond is recommended for abrading hard materials such as ceramics, glass, most igneous and metamorphic rocks, and concrete.

Silicon carbide is used as either a loose or fixed abrasive (as in abrasive papers). Silicon carbide papers can be used for grinding soft sandstones and clays, bones and teeth, polymers and other soft materials. Because of its crystal shape, it is difficult to classify in submicrometer particle sizes. Therefore, it is not available in fine particle sizes for final polishing. Aluminum oxide crystals are more blocky in shape than silicon carbide crystals. They break

down into uniformly shaped particles and therefore are easier to classify. Aluminum oxide is available in two crystallographic forms, hexagonal and cubic. When a scratch free surface is desired, aluminum oxide is the choice abrasive. Aluminum oxide particles made by the calcination process do have a tendency to agglomerate. Aluminum oxide particles made by the sol-gel process, as used in the MASTERPREP™ suspension, are agglomerate free and produce the best surface finish for a given alumina particle size. When selecting abrasives for specimen preparation it is important that good quality, well-graded abrasives are used to maintain precise surface finish.

Polishing

The purpose of polishing a specimen is to remove the final deformation induced by the grinding process and yield a surface that is essentially damage-free. Polishing is accomplished by abrading the surface with fine abrasives progressively decreasing to sub-micrometer size abrasives. Because this is the last procedure in the preparation sequence, any deformation still remaining at the specimen surface will be visible when the specimen is observed with the microscope. The polishing time will vary depending upon the size of the abrasive used, force applied, polishing time and the wheel speed. A general guideline, which can be followed with ease, is to observe the specimen surface with a microscope at the end of each step. The specimen surface is examined for pits and scratches. At any given stage, there may be some pits and scratches, which are proportional to the size of the abrasive used in preparing the surface. When polishing ceases to improve the surface regardless of the time spent, the specimen should be cleaned thoroughly and moved to the next step of polishing. This simple method to determine when to stop polishing will be useful in deciding the time required for each step. Unnecessarily long times spent polishing are not only wasteful but also result in producing undesirable relief on the specimen surface. Relief is the topographical (height) difference between soft and hard phases due to differences in abrasion rates, and that makes microscope focusing difficult. As the magnification required to image a structure increases, the numerical aperture (NA) of the objective also increases. Increasing the NA improves resolution but decreases depth of field. The microstructure cannot be fully brought into sharp focus when the relief height exceeds the depth of focus.

To prepare a specimen that contains components of different hardness, an abrasive and cloth must be selected which will minimize relief on the surface. This selection also will determine the surface finish quality. Although there are always exceptions to any rule, diamond abrasives usually produce less relief than other abrasives such as aluminum oxide, colloidal silica, chromic oxide, cerium oxide, iron oxide, etc. The polishing cloth also plays a very important role in minimizing or accentuating relief. If preventing relief is the main objective, a “hard” cloth that does not have nap (fibers), should be selected. If surface finish quality (scratch control) is the primary concern, then a “soft” cloth that has a nap is more desirable.

Other factors that may influence relief are the polishing time and applied pressure. Long polishing times generally accentuate relief, especially when soft polishing cloths are used. Excessively high applied forces on soft polishing cloths for extended period also accentuate relief.

As a rule, use diamond abrasives and hard polishing cloths (such as a TEXTMET® pad) in the initial stage of polishing and then switch to a softer abrasive, such as alumina or colloidal silica (MASTERMET), for the final step of polishing on a soft cloth (such as a MICROCLOTH® pad).

Thin-Section Preparation

The preparation sequence for making transparent thin sections is as follows: sectioning, vacuum impregnation, grinding, cementing, re-sectioning, grinding and polishing. The preparation of thin

sections is considerably more difficult than preparing polished bulk specimens. Generally, a thin section must be prepared to a thickness of approximately 30µm, with near perfect parallelism. Thin sections begin with bulk sectioning, followed by thorough cleaning and vacuum impregnation to fill the pores and consolidate the specimen material. Grinding of the specimen "chip" is employed to produce a flat, smooth surface, free of gross deformation. This surface is important because it will be mounted directly to the glass slide. Rough grinding of medium to soft materials may be performed using a cast iron lapping platen with silicon carbide abrasive powder of various grit sizes. Softer materials are sometimes ground flat using abrasive papers. Hard specimens may require the use of various micrometre size diamond grinding discs depending on the hardness and abrasion resistance of the material.

Impregnated sections must be ground flat before cementing to the glass slide. To determine if the entire surface of the chip has been ground flat, hold the ground surface against light at approximately a 45° angle. An evenly reflective surface indicates that the entire surface has been ground properly. A non-uniform, dull surface may indicate that the entire surface has not been ground flat and should be re-ground for a longer time.

Note that some minerals, such as Biotite, may remain dull in appearance even after proper grinding. Do not remove excessive material as this may result in exposing unimpregnated pores.

Before cementing the chip to the glass slide, the specimen must be cleaned thoroughly to remove all loose abrasive and other residue, then dried. It is sometimes helpful to pregrind one side of the glass slide surface. This produces a slide of a more uniform thickness and the roughened surface aids in establishing a good bond. To produce a slide of uniform thickness, use the 30-8001 Glass Slide Holder; or, the PETROTHIN® system can be used to grind the slide. Generally, loose silicon carbide abrasive powders, with grit sizes of 600 or 1000, may be used on a cast iron lap for grinding slides.

When using epoxies to attach the chip to the glass slide and to obtain a uniform glue thickness, the PETROBOND™ bonding fixture is recommended. Place the fixture on a hot plate to hasten curing of the epoxy when using the PETROBOND fixture. The temperature should not exceed 50°C. It takes approximately two to three hours for the epoxy to cure when heat is applied. Without the application of heat, thin sections may take 40-48 hours to cure properly depending on the ambient temperature. Besides epoxy, other mounting media can also be used to attach the chip to the glass slide.

The purpose of re-sectioning is to reduce the thickness of the chip to minimize grinding time. Re-sectioning can be accomplished by using a diamond wafering saw or the PETRO-THIN, thin sectioning system.

Semi-Automatic Preparation of Thin Sections

Thin sections provide more comprehensive microstructural information than bulk specimens, however, they are much more difficult and time consuming to prepare. Preparing thin sections by hand requires a great deal of expertise and time, neither of which are widely available to most laboratories. Also grinding by hand tends to favor one or the other side of the thin section, eventually making one side thinner than the other side.

The PETRO-THIN, thin sectioning machine is a semi-automatic device that can prepare thin sections very rapidly without compromising accuracy or quality. The PETRO-THIN system is self contained consisting of a diamond cutting blade, a diamond grinding wheel, and a vacuum chuck that accepts five sizes of glass slides. Two precision micrometers are used for controlling cutting and grinding of the thin section. The vacuum chuck holds the thin section during

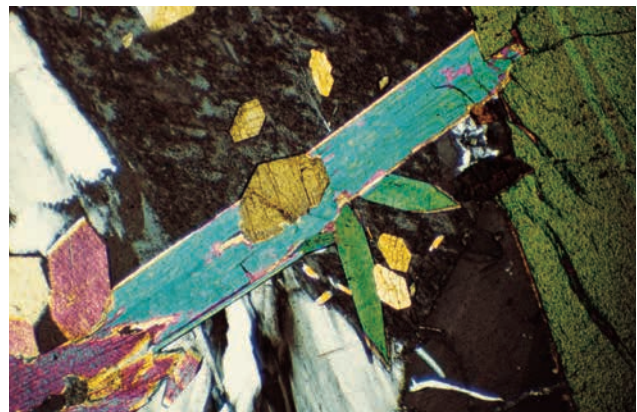


Figure 6. Acmite, thin section, in polarized light, 100x.

preparation and assures accuracy that other mechanical holding devices cannot provide.

To grind the thin section, it is moved into the path of the grinding wheel as the micrometer accurately advances the thin section. Depending on the hardness and the friability of the specimen, 10-20µm of the specimen surface can be removed in one pass. For some materials, removing more than 20µm thickness in a pass may cause fracturing of the grains and should be practiced only with soft materials. Once a section is ground to the desired thickness, the specimen can be examined using transmitted light (Figure 6) or it can be polished*. A polished thin section can be examined with either a transmitted or reflected light microscope. Other advantages of a polished thin section include the following:

- Mineral hardness may be determined
- Chemical tests can be performed on the polished surface
- The time consuming procedure used for applying the cover glass is eliminated.

Thin section preparation requires much more accuracy than polished bulk specimens. Automation assists in preparing specimens more accurately than is possible when thin sections are prepared by hand. It is not uncommon in hand preparation, to end up with a thin section which is not uniform in thickness or to have completely lost the section by applying more pressure than is required during grinding.

**The PETROPOL polishing system can polish several sections simultaneously.*

Ultra-Thin Section Preparation

Very fine grain materials such as cement consist of crystals that are smaller than 30µm in size. To analyze such a specimen, the standard 30µm thick thin section may not be suitable. This is because a thin section of standard thickness may contain several layers of the fine crystals that may make microscopical observations difficult. In order to best examine such a specimen, it is sometimes necessary to prepare ultra-thin sections, much thinner than 30µm. The conventional method of thin section preparation is unsuitable because at this thickness even light applied pressure can destroy the specimen. However, by using a vibratory polisher, such as the VIBROMET® polisher, it is possible to obtain ultra-thin sections. The vibratory polishing method is very gentle and removes material very slowly, which is essential for preparing ultra-thin sections. Most specimens may be left in the VIBROMET polisher for several hours. Generally, specimens are polished with fine abrasives, such as 1µm diamond, on a TEXMET pad, or with sub-micrometre size alumina on a MICROCLOTH pad.

The PETROPOL polishing device mentioned earlier may also be used, however, it removes material at a much faster rate than the VIBROMET polisher. Consequently, frequent monitoring of the specimen is required.

Summary

Petrographic analysis has been used to reveal the microstructure of ores, minerals and rocks for a long time. Further, this procedure can be very successfully used to analyze many other materials, such as concrete, cement (Figure 7), ceramics, refractories, biomaterials (Figure 8) and polymers. Specimen preparation can provide a wealth of information for microscopical analysis of various materials. It is up to us to do it correctly, so that erroneous conclusions are not drawn because of poor specimen preparation practices.

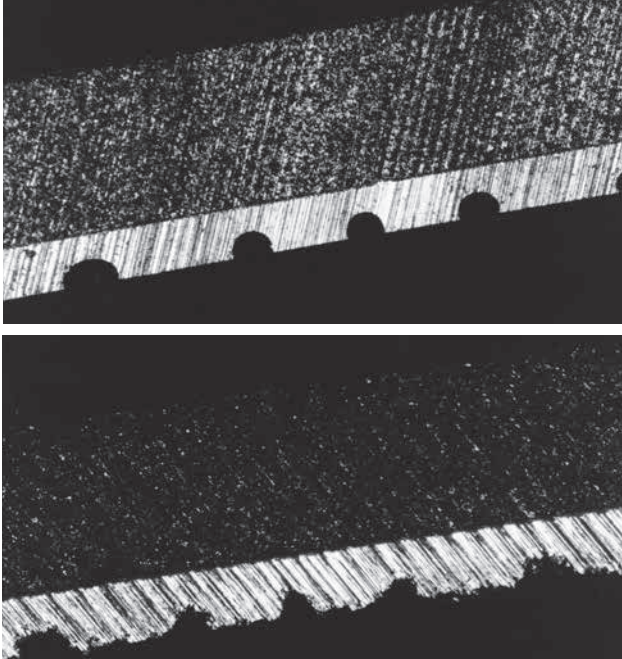


Figure 3a. (top) Copper coating on ceramic substrate cut with 10LC blade – 100x. Figure 3b. (bottom) Copper coating on ceramic substrate cut with 20HC blade.

Tech-Tips

Question: When preparing a thin section manually, it is difficult to hold the glass slide, if a thin section holder is not available. What can I do to prevent them from slipping out of my hand?

Answer: Cut a piece of METGRIP liner (a double stick pad) slightly smaller than the slide. Peel the backing paper of the METGRIP liner and attach it to the back of the glass slide. Peel the backing paper from the other side and place your fingers on the sticky surface. This will prevent slipping of the glass slide.

Question: How can I prevent cast iron wheels from rusting?

Answer: Scrub the wheel clean and apply a solution consisting of equal amounts of No.10- 3330 soluble oil and water.

Question: How can I hold a glass slide when using the VIBROMET for polishing?

Answer: Insert the No. 69-1580 Thin Section Attachment into a 1.5 inch VIBROMET holder and tighten the screw of the VIBROMET holder. Apply a thin coating of oil to the thin section holder and insert the slide.

Question: How can I prevent abrasive papers and polishing cloths from sticking too firmly to the platen?

Answer: Clean and dry the platen. Turn the platen on and apply a light coating of No. 20- 8185 release agent to the platen surface. After the liquid has evaporated, remove the excess from the platen surface by wiping it lightly with a paper towel. Too much release agent will prevent the abrasive paper or the polishing cloth from adhering to the platen.

Question: How can I prevent excessive pull out from the surface?

Answer: Try using an abrasive as fine in particle size as practical. Use diamond abrasives and a hard polishing cloth such as TEXMET 1000 for polishing to minimize pull out.

Question: What can I do to resolve fine details in my thin sections?

Answer: A double-polished thin section will resolve minute details which are difficult to resolve otherwise. To prepare a double-polished thin section, grind and polish the specimen surface with fine abrasives. Attach the polished surface to the glass slide, if possible, using the Petro Bond thin section bonding fixture. Resection the specimen and grind and polish the top surface of the thin section.

Question: I am encapsulating very hard ceramic specimens in a room temperature setting resin. How can I prevent the edges from rounding?

Answer: Add FLAT EDGE FILLER™ (No.20- 8196) to the resin, which will minimize edge rounding.

Question: I am using epoxy for encapsulation. I would like to retrieve the specimen after polishing. How can I do that?

Answer: You have several choices. You can try dissolving the epoxy by submerging the specimen in methylene chloride, provided the specimen will not react with the solvent. Methylene chloride is a strong solvent and carcinogenic, therefore, care must be exercised in using it. Use a fume hood. It evaporates rapidly, so cover it; or pour a little water which will float on top of methylene chloride and prevent evaporation.

You can also dip the specimen in boiling glycerin for one or two hours, which will soften the epoxy. Take the mount out and twist the mount. The specimen should come out easily.

If the specimen is sensitive to solvent or heat, grind or cut around the specimen until it is mostly exposed. Prying the specimen with a screwdriver will dislodge it from the molding material.

Sectioning	Mounting	Grinding & Polishing	Imaging & Analysis	Hardness Testing
AbrasiMet • AbrasiMatic • IsoMet	SimpliMet	EcoMet • AutoMet • MetaServ	OmniMet	Wilson® Hardness



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