BUEHLER®

Guide to Petrography

Specimen Preparation Stages

- Sectioning Specimen
- Impregnation & Encapsulation
- Trimming
- Surface Preparation Prior to Bonding
- Bonding
- Resectioning
- Grinding
- Polishing
- Microscopic Examination
- Image Analysis & Capture

PRELIMINARY DRAFT

BUEHLER®

Introduction

In 1849 when Henry Sorby, the father of petrography, prepared a thin section, he may not have envisioned that one day his technique would be used to analyze many diverse materials such as ceramics, glass, concrete, cement, soils, biomaterials, and polymers, to name a few. Sorby demonstrated to the scientific community that by using simple techniques, one could reveal the microstructure of materials observed with a microscope. Although Sorby's method of preparation was crude compared to the methods used today, his basic technique for preparing and examining a specimen has remained fairly similar.

There are two types of specimens routinely prepared for analysis, thin sections and polished bulk specimens. For polished bulk specimens, 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 typically observed with a transmitted polarized light microscope. The chart to the right describes a general procedure required to prepare both thin sections and polished bulk specimens.

Since 1936, Buehler has been the world leader in developing and supplying high quality laboratory specimen preparation equipment and consumable products. Buehler's experience paired with today's technologies help deliver the fullest product offering for all applications. Together with our technical expertise and lab equipment, Buehler is prepared to be your materials preparation partner.

Buehler is the science behind materials preparation and analysis™.

Geological Sample Preparation

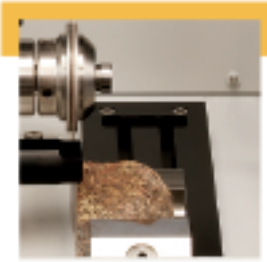
SECTIONING

Bulk Cutting, with either the Lapro® Slab Saw, Delta® PetroCut™ or other Delta Family Cutters, removes a representative sample of manageable size from the bulk material.

IMPREGNATION

Vacuum impregnation is used to remove gases from pores or cracks in friable specimens and allows infiltration of a suitable bonding material that will retard sample fracturing & plucking. A Cast N' Vac 1000 Vacuum Impregnation System is recommended.

PRECISION SECTIONING



Perform trimming and precise, low deformation cutting with the IsoMet® Family of Diamond Precision Saws.

BONDING

Bond a prepared surface to a glass slide for re-sectioning, grinding and finishing with the PetroBond® Thin-Section Bonding Fixture.

RE-SECTIONING

Remove excess material quickly and efficiently with an IsoMet® Saw or the PetroThin® Thin Sectioning System.

GRINDING & POLISHING

Final polishing produces a high quality, damage free surface with thickness of ~30µm or less with Alpha, Beta or other grinder-polishers. The VibroMet® 2 Vibratory Polisher is recommended for ultrathin sections of 10µm or less and ideal for EBSD applications.

Sectioning

A first step in specimen preparation is sectioning, performed for the following reasons.

- Obtain a manageable size specimen from the parent material
- Reduce thickness of the specimen so that grinding time is decreased (as in the case of preparing thin sections)
- Expose the surface of interest

Historically, the sectioning process was considered unimportant. 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 caused in sectioning will be difficult to remove in the grinding and polishing steps that follow. It is important that the proper saw and blade are selected to minimize any damage.

Selection of the saw depends upon the following:

- Size of the specimen to be cut
- Variety of materials in the specimen
- Cutting speed required
- Number of specimens required to be cut in a day
- Serial sectioning requirements
- Level of automation desired

There are two classes of cutters; some are designed for bulk cutting and others are made for precision sectioning.

Bulk Cutting

To get the specimen down to a manageable size when there is no concern about kerf loss, a cutter for removing bulk sections is ideal.

The **11-1360 Lapro® 24" (610mm) Diamond Slab Saw** is a high quality, durable machine designed specifically for cutting rocks, minerals, concrete, glass, ceramics, tile and other hard brittle materials up to 9.5" (241mm) in diameter. It features a unique hydraulic and dead weight controlled feed system that ensures proper feed rate and maximum blade life. A special roller bearing, mounted above the 24" (610mm) diamond blade, is a key component of the built-in blade sharpening mechanism, to provide efficient cutting action every time.

The **10-2156 Delta® PetroCut® Geological Cutter** is a manual, 4Hp (3 kW) cutter using a 10" (254mm) continuous rim diamond blade for sectioning rock, concrete, refractory, etc. up to 3.75" (95mm) in diameter. Features include a 10-3533 Rock Clamp Assembly with cross-feed table for serial cutting. Select other specimen vises for irregular shapes.

Top) The 11-1360 Lapro® 24" (610mm) Diamond Slab Saw is designed to section larger samples with a 9.5" (241mm) cutting depth. Bottom) The 10-2156 Delta® PetroCut® Geological Cutter features a Rock Clamp Assembly for holding most odd shaped rocks or minerals up to 3.75" (95mm) in diameter.

Impregnation

Once a specimen has been cut, it should be thoroughly cleaned and dried. Materials that may have pores, cracks, or are poorly consolidated, must be vacuum impregnated with epoxy prior to grinding. Epoxies that have a low viscosity, such as Buehler's EpoThin® and EpoxiCure®, are ideal for this process.

Note, in some cases, it may be desirable to impregnate or mount the specimen first and then section it with a saw. This is done for weak, fragile or small specimens, which will otherwise break or are too small to be held securely in a chuck during sectioning.

To impregnate or mount the specimen using a 20-1382 Cast N' Vac 1000, place it in a mold, such as SamplKups®. Place the SamplKup mold and the paper cup containing epoxy in the vacuum chamber. Turn the vacuum pump on to evacuate its chamber. This draws air from pores of the specimen and facilitates filling the pores with epoxy. When a proper vacuum has been attained, tilt the epoxy cup and pour the epoxy into the mold. Keep the mold under vacuum for 3-30 second intervals slowly releasing the vacuum between them allowing air to enter the chamber forcing embedding medium into the pores. A specimen should not be left under the vacuum during the curing cycle. Once the epoxy has cured, specimens can be sectioned, ground, and attached to a slide; or if a bulk specimen for reflected light microscopy has to be prepared, it can be ground and polished.

The 20-1382 Cast N' Vac 1000 is ideal for impregnating geological samples with a suitable bonding material that fills pores, cracks, and retards sample fracturing or plucking.

Precision Sectioning/Trimming

Diamond wafering saws, such as the IsoMet® series, are ideal for precision sectioning of petrographic specimens. Cutting parameters for sectioning, which include blade speed and feed rate, can all be precisely controlled.

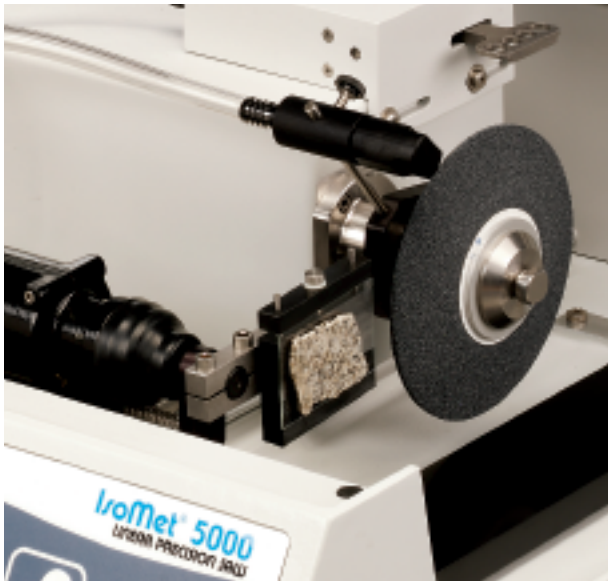
11-1280 IsoMet® Low Speed Saw is designed to section small specimens. Its speed is 0-300 rpm and the maximum blade size is 5" (127mm). A precision micrometer, available with imperial or metric graduations, controls thickness of the cut. Accessories include, but are not limited to, a 11-2487 single saddle chuck, 11-2486 wafer chuck, and 11-1185 irregular specimen chuck. Optional accessories include a 11-2494 bone chuck, 11-2488 glass slide chuck and a 11-2481 goniometer.

Top) A cement sample is precisly sectioned with the 11-1280 IsoMet® Low Speed Saw. Bottom) The 11-2488 Glass Slide Chuck for cutting glass slide mounted samples is shown.

11-2180 IsoMet® 1000 Precision Saw has a large cutting capacity with built-in inch or metric digital micrometer cross-feed for sample positioning. Its greatest speed is 975rpm with a maximum blade size of 7" (178mm). When sample size prohibits the use of standard chucks, an optional 11-2182 cutting table can be installed for manually sectioning or trimming. Accessory chucks are similar to the IsoMet Low Speed saw but have the capability of handling larger specimens.

11-2680 IsoMet® 4000 Linear Precision Saw features a SMARTCUT system which automatically monitors and adjusts the feed rate to provide consistent, quality cuts and prevents specimen and machine damage. A precision digital micrometer controls the thickness of a cut. The cutting chamber has a safety hood. A digital panel controls cutting parameters such as speed and feed rate. Maximum blade size is 8" (203mm). The control panel is capable of displaying cutting parameters in several languages.

11-2780 IsoMet® 5000 Linear Precision Saw is a fully automatic linear feed precision saw. Similar to the IsoMet 4000, it is however, capable of automatic serial sectioning of a specimen to a precise thickness. This is achieved by using an automatic linear feed motor, which advances the specimen for repeated cuts of desired thickness. Thickness, speed, feed rate and other parameters are adjustable. The saw is ideal for serial sectioning bones or teeth where several thin sections can be used for mapping the specimen. In addition, a cup grinder attachment is available for grinding to a targeted depth which is useful for thin sections.



11-2780 IsoMet® 5000 with 11-2740 cup grinder shown. Cup grinding can be used for thin section preparation.

Precision saws are capable of cutting very thin specimens, although there is a tendency for a thinly cut specimen to curl. Unless specimens are to be used in the as-cut condition and do not have to be cemented to a slide, it is advisable to cut them thick enough so that they can be held by hand and ground easily.

Selection of a proper blade
Blade selection is dependent on characteristics of the material being sectioned. Low Concentration (LC) Diamond Blades are recommended for non-metallic materials that are hard and brittle such as rocks and minerals. High Concentration (HC) Diamond Blades are suggested for metallic specimens which have biomaterials for example, bone with metal. Additional information is available in the Buehler Consumables Buyers Guide.

Bonding

The Buehler **38-1490 PetroBond® Thin-Section Bonding Fixture** is designed to provide a uniform thickness of bonding media between specimens and glass slides. The spring activated loading fixture can accommodate up to 12 thin-section slides 2″ x 3″ (50 x 75mm). If the bonding agent requires heat, the entire fixture can be placed directly onto a hot plate.

38-1490 PetroBond Thin-Sectioning Bonding Fixture assists in bonding 12 ground specimens to glass slides prior to re-sectioning.

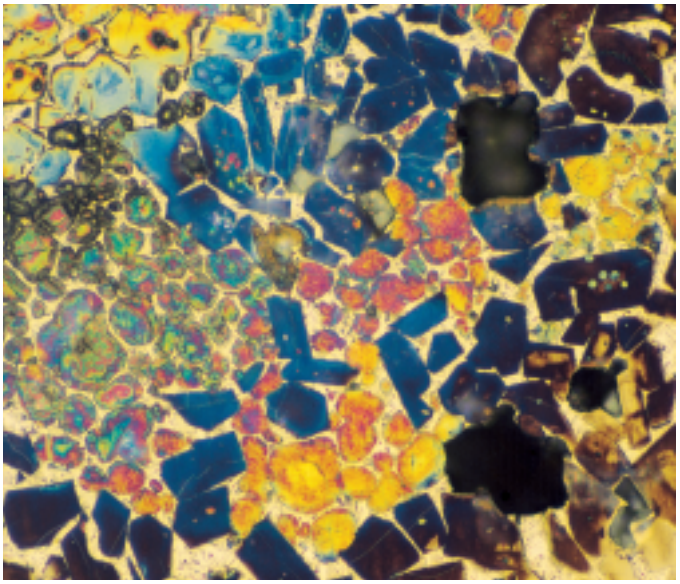
Impregnated sections must be ground flat before cementing to the glass slide to ensure good adhesion between specimen and slide. Hold the ground surface towards a light at approximately a 45° angle to determine if the entire surface of the chip has been ground flat. An even, reflective surface indicates that it has been ground properly. A non-uniform, dull surface may indicate it has not been ground flat and should be re-ground for a longer time.

It is sometimes helpful to pre-grind one side of the glass slide surface. This produces a slide with more uniform thickness and the roughened surface aids in establishing a good bond. Generally, loose silicon carbide abrasive powders, with grit sizes of 600 (P1200) or 1000 (P2000), may be used on a cast iron lap for grinding slides.

Re-sectioning

The **10-2156 PetroThin® Thin Sectioning System** is a semi-automatic device that can prepare thin sections very rapidly without compromising accuracy or quality. The PetroThin 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 control cutting and grinding of the thin section. A vacuum chuck holds the thin section during preparation and assures accuracy that other mechanical holding devices cannot provide.

38-1450 PetroThin® Thin Sectioning System allows for the re-sectioning and grinding of rocks or minerals, ceramics and geological specimens in one machine. Grind to a thickness of ~100µm.



Microstructure of a cement clinker (Portland cement used for construction), showing various phases such as alite and belite. The specimen has been etched with 2% nital, ~260x. Reflected light.

Grinding & Polishing

Grinding is performed to remove deformation induced in sectioning and to planar grind; and, in the case of thin sections, this step removes excess material. Grinding is generally performed using either fixed abrasives where the abrasive particles are bonded to a substrate and are not free to move, or loose abrasives where the abrasive particles are not bonded to a substrate but are free to roll on a special platen as they abrade the specimen. This latter procedure is called lapping.

Once a section is ground to the desired thickness, the specimen can be examined using transmitted light or it can be further polished. The purpose of polishing a specimen is to remove any 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. Any deformation still remaining at the specimen surface, after the last step will be visible when the specimen is observed with the microscope; so, the goal is to remove any damage prior to the last step.

A polished thin section can be examined with either a transmitted or reflected light microscope. Advantages for polishing during the thin section preparation process 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
- Cellular detail is revealed

There is an array of preparation systems available for accomplishing both grinding and polishing. Selection of a grinder-polisher depends upon the following.

- Size of the cross-section
- Number of specimens prepared each day
- Level of automation desired

69-1000 MiniMet® 1000 Grinder-Polisher is designed for low volume laboratory work preparing small single specimens. Its design employs a patented geometric action that combines the advantage of hand lapping as well as mechanical polishing. This motion provides a random polishing action, thus eliminating any induced directional polishing scratches.

Polishing bowls eliminate cross-contamination problems between preparation steps. Attachments for the precision thinning of materials (69-1566), wafer polishing (69-1590), electromechnical polishing (69-1570), and thin section preparation (69-1583) are available.

The **49-5500 Alpha and 49-5100 Beta** are manual grinder-polishers designed for use with 8″ (203mm) and 10″ (254mm) platens, available in two speeds or variable speed for greater versatility. Both the Alpha and Beta can be upgraded to semi-automatic operation with an addition of the 60-1990 Vector® Power Head. This enables sample preparation using either single or centrally applied pneumatic loading. Single force loading is best for petrographic specimens. The **Petrographic/Histolic Thin Section Specimen Holders** 60-3000 & 69-1584 are ideal for automating your thin section sample preparation.

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 that is not uniform in thickness or to have completely lost the section by applying more pressure than is required during grinding.

Automate thin section preparation with the Petro Histolic Single Force Specimen Holder 60-3000 which holds 4 Petro Histolic Single Force Specimen Holders 69-1584 and can be mounted on the Vector Power Head.

Eliminate preparation inconsistencies and improve productivity by using the **Histolic Precision Grinding Fixture** 60-8087. This fixture assists in obtaining a specimen of very uniform thickness and has carbide stops that prevent a specimen from being over ground. The fixture is designed for slides that are 27 x 46mm (1 x 1.8″) in size. Three 60-8087 fixtures can be used in specimen holder 60-8017 for semi-automatic preparation on the PowerPro® Family of Grinder-Polishers for greater productivity and consistency.

The Alpha, Beta and Vector is one of various Grinder-Polishers and Power Head with Single Force (ideal for petrographic specimen preparation) that Buehler offers. Equipped with the optional 60-3000 Petro Histolic Single Force Specimen Holder & 69-1584 Petro Histolic Single Force Specimen Holder Accessories, you now have a semi-automated grinder-polishing system for improved consitency and productivity.

Histolic Precision Grinding Fixture 60-8087 will hold a 27 x 46mm (1 x 1.8″) glass slide and is ideal for precise material removal.

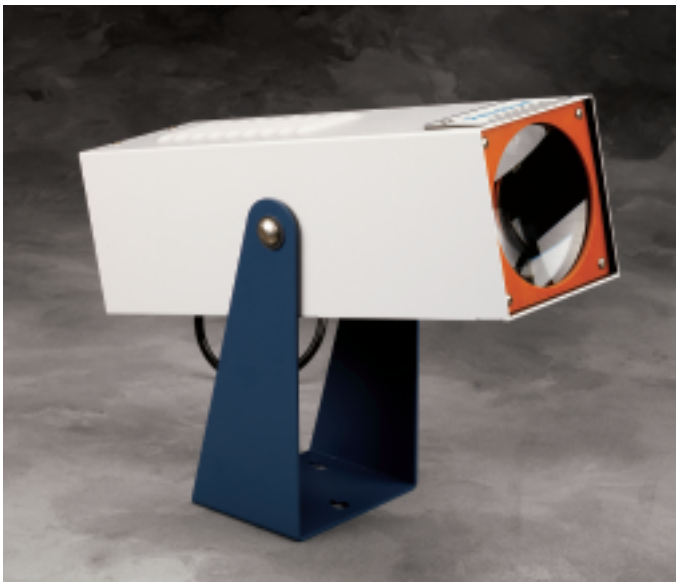
Ultra-Thin Sections

To best examine certain specimens, sometimes it is necessary to prepare ultra-thin sections, much thinner than 30μm. A thin section of standard thickness may contain several layers of fine crystals. The conventional method of thin section preparation may be unsuitable because at this thickness, even light pressure can destroy the specimen. Use of a vibratory polisher, however, such as the 67-1635 VibroMet® 2 Vibratory Polisher, makes it possible to obtain ultra-thin sections. This polishing method is gentle and removes material very slowly, which is essential for preparing ultra-thin sections and EBSD work.

Granite thin section, under cross-polarized light (first order red plate), ~127x. Transmitted light.

Viewing

The **30-8050 PetroVue® Thin Section Viewer** is designed for monitoring section thickness and uniformity during the grinding-lapping processes. This viewer has permanent cross-polarizing filters with a 100W bulb and provides a view of the entire thin section area.



30-8050 PetroVue® Thin Section Viewer, with a polarized light source, is designed to quickly monitor thin sections for quality and thickness without a microscope.

Complete your thin section preparation lab with a selection from the Buehler optical products catalog. The material science based microscopes include compound and stereoscopic instruments that can be configured with both reflected and transmitted illumination as your requirements dictate.

Take your thin section analysis to the next level with a Buehler OmniMet® Image analysis system. Choose from a basic image capture system to a full blown image analysis

OmniMet® Modular Imaging Platform

suite with user definable analysis modules. All OmniMet software suites have database capability, image import/export and report generation utilities.

Accessories

For information regarding other Buehler products such as slide holders, glass slides, petrographic fixtures, ultrasonic cleaners, microscopes, and a complete range of consumables visit www.buehler.com or consult your nearest Buehler sales office. This brochure has been designed to provide information on equipment selection, enabling you to find the best fit for your application and ensure the best results. Additional information on preparation techniques is provided in the **BUEHLER® SUM-MET™, A Guide to Materials Preparation and Analysis** found by joining the Buehler e-Club at www.buehler.com.

Cover Image: Microstructure from Niah Cave West Mouth, Sarawak, Malaysia which shows fine layers from the prehistoric cemetery sequence. The layers are primarily composed of guano. The slide was made by Julie A. Miller at the Thin Section Facility, Department of Archaeology, University of Cambridge, UK.

For a complete listing of Buehler consumable supplies, please refer to Buehler's Consumables Buyer's Guide. Buehler continuously makes product improvements; therefore, technical specifications are subject to change without notice.

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