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SumMet™

The Sum Of Our Experience

A Guide to Materials Preparation & Analysis

Fourth Edition
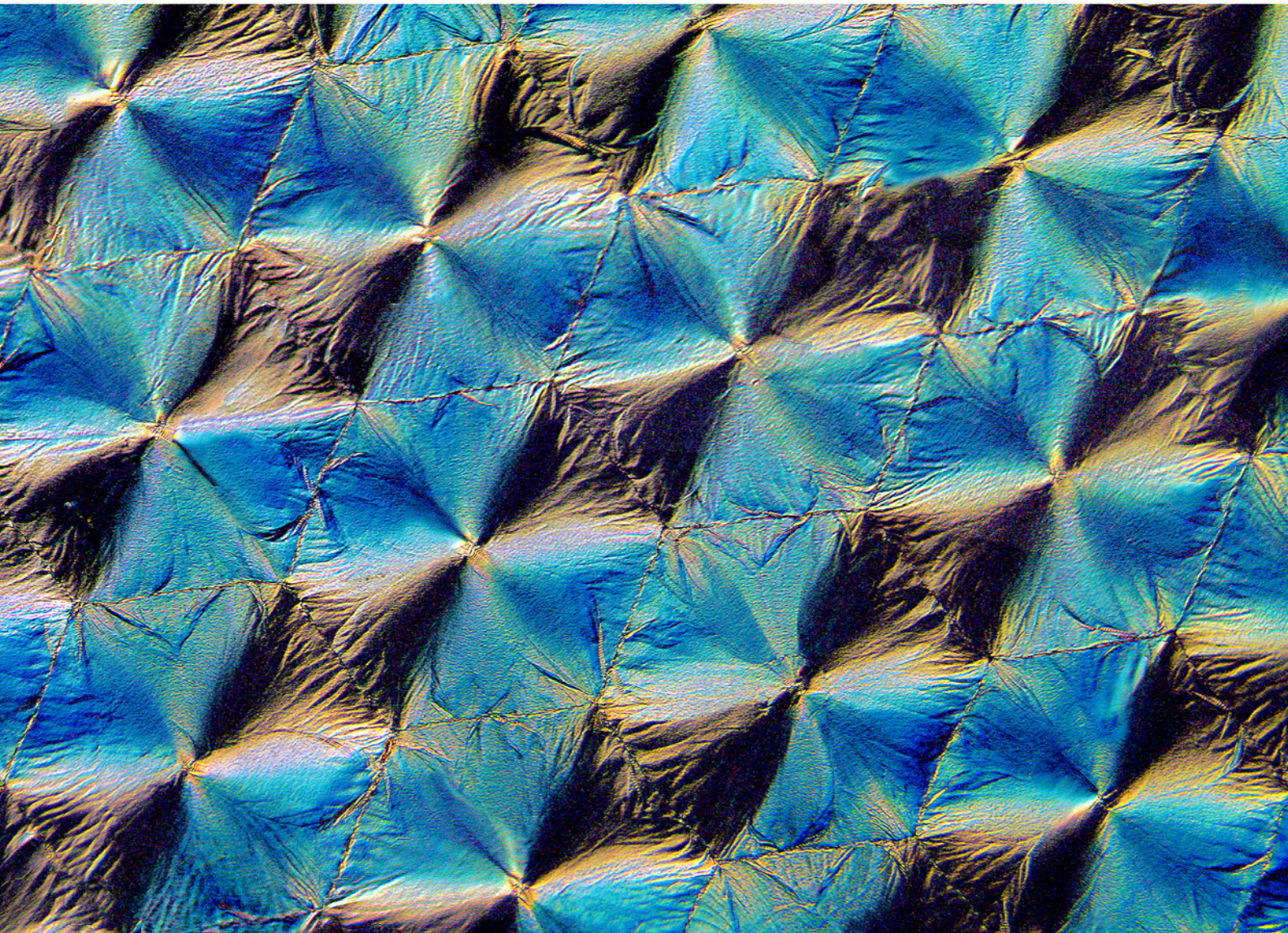

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Strong Partner, Reliable Solutions

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Buehler® *SumMet*™ - A Guide to Materials Preparation and Analysis

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Warning and Disclaimer

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The Sum Of Our Experience

Buehler is a worldwide leader in materials preparation, testing and analysis. As your partner in all aspects, we provide not only a complete line of equipment and consumables, but a strong team dedicated to application knowledge, technical support, and when need be, service. With more than 80 locations across the globe offering telephone and email support, training courses, webinars, and one-on-one custom training, Buehler is there offer support to our customers, industries and applications. Dedicated to your needs, we strive for fast consumable deliveries, efficient service support and 24/7 access to our online preparation guide.

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INTRODUCTION

Proper specimen preparation is essential if the true microstructure is to be observed, identified, documented and measured. Indeed, when metallographers have difficulty seeing or measuring microstructures, these problems can almost always be traced to improper specimen preparation procedures, or failure to properly execute the preparation steps. When a specimen is prepared properly, and the structure is revealed properly, or a phase or constituent of interest is revealed selectively and with adequate contrast, then the actual tasks of examination, interpretation, documentation and measurement are generally quite simple. Experience has demonstrated that getting the required image quality to the microscope is by far the greatest challenge. Despite this, the specimen preparation process is often treated as a trivial exercise. However, specimen preparation quality is the determining factor in the value of the examination. This is in agreement with the classic computer adage, "garbage in = garbage out."

Historically, people tend to divide specimen preparation (after mounting) into two steps, grinding and polishing, further sub-divided, somewhat arbitrarily, into coarse and fine stages. Years ago, these divisions were quite clear. Both processes involve the same mechanism, chip formation by micro-machining. Grinding removes material from the specimen surface using randomly oriented, fixed abrasives on a substrate such as paper or cloth. The abrasive size range has generally been in the range of about 240 to 5µm, with silicon cabide (SiC) paper used exclusively until recently.

Traditionally, polishing has been defined in terms of industrial polishing rather than metallographic polishing, simply stating that it is a smoothing of the surface to a high luster. A polished surface was defined as "a surface that reflected a large proportion of incident light in a specular manner." These definitions are true, but they are inadequate for metallography. Polishing also produces micro- machining action, but scratch depths are shallower in polishing, as the abrasives are smaller. They are not fixed initially to a substrate, but are applied using as a paste or suspension. After a short time, the abrasive particles become fixed in the cloth producing cutting action. The cloths used in polishing exhibit a range of resilience depending on whether they are non-woven polymeric or chemo-textiles, woven napless materials, or napped materials. Resilience influences edge retention, relief control and cutting rate ("aggressiveness"). Polishing must remove the damage remaining after grinding, with the final step reducing the damage to where it is insignificant, or can be removed by the etchant. In this way, the true microstructure is revealed. For most metals and alloys, and many non-metallic materials, polishing down to a 1µm, or even a 3µm diamond finish may yield a surface suitable for routine examination, although finer abrasives are usually needed for more critical work, such as failure analysis.

Polishing was historically performed with abrasive slurries, most commonly using alumina, but with limited use of MgO, Cr_2O_3 or CeO for specific materials. Diamond abrasives were introduced in the late 1940s. Cloths such as canvas, billiard, felt, and cotton are not used as much today as they tend to promote relief and edge rounding, or other artifacts. Modern preparation methods have focused on reducing the number of SiC grinding steps and replacing SiC paper with other abrasives and surfaces, although there are some materials that respond better to grinding with a series of finer and finer SiC papers. But, most materials can be prepared with one grinding step, often called planar grinding when an automated system is used, and two or more polishing steps. The second step uses relatively hard, non-resilient surfaces with a relatively coarse diamond size (15 or 9µm, for example) and the abrasive is added as a suspension or paste. Initially, the particles move between the specimen surface and the substrate. Technically, this is lapping. But, after a short time, the diamond particles become embedded in the surface and produce cutting action. We tend to call this polishing, although the historic definitions are not as clear here. If the substrate is a rigid grinding disc, the action may be more correctly called grinding than polishing, as the surface finish will be dull. The same abrasive when used on a hard, woven surface such as an UltraPol silk cloth, will yield a much more lustrous appearance, but with less stock removal and shallower scratches. Napped cloths, if they are used, are generally restricted to the final step with the finest abrasives.

The samples selected for preparation must be representative of the material to be examined. Random sampling, as advocated by statisticians, can rarely be performed by metallographers. A good exception is the testing of fasteners where a production lot can be sampled randomly. But, a large forging or casting cannot be sampled at random as the component would be rendered commercially useless. Instead, test locations are chosen systematically based on ease of sampling. Many material specifications dictate the sampling procedure. In failure analysis studies, specimens are usually removed to study the origin of the failure, to examine highly stressed areas, to examine secondary cracks, and so forth. This, of course, is not random sampling. As testing costs are usually closely controlled, it is more likely that inadequate sampling occurs than excessive sampling.

Normally, a sample must be removed from a larger mass and then prepared for examination. This requires application of one or more sectioning methods. For example, in a manufacturing facility, a piece may be cut from a bar of incoming metal with a power hacksaw, or an abrasive cutter used dry, i.e., without a coolant. The piece is then forwarded to the laboratory where it is cut smaller to obtain a size more convenient for preparation. All sectioning processes produce damage; some methods (such as flame cutting or dry abrasive cutting) produce extreme amounts of damage. Traditional laboratory sectioning procedures, using abrasive cutters, introduce a minor amount of damage that varies with the material being cut and its thermal and mechanical history. It is generally unwise to use the original face cut in the shop as the starting point for metallographic preparation as the depth of damage at this location may be quite extensive. This damage must be removed if the true structure is to be revealed. However, because abrasive grinding and polishing steps also produce damage, where the depth of damage decreases with decreasing abrasive size, the preparation sequence must be carefully planned and performed; otherwise, preparation-induced artifacts will be interpreted as structural elements.

Many metallographic studies require more than one sample and sectioning is nearly always required to extract the sample. A classic example of multiple sample selection is the evaluation of the inclusion content of steels. One sample is not representative of the whole lot of steel, so multiple sampling becomes more important. ASTM standards E45, E1122 and E1245 give advice on sampling procedures for inclusion studies. To study grain size, it is common to use a single specimen from a lot. This may or may not be adequate, depending upon the nature of the lot. Good engineering judgment should guide sampling, in such cases. In many cases, a product specification may define the procedure rigorously. Grain structures are not always equiaxed and it may be misleading to select only a plane oriented perpendicular to the deformation axis, a "transverse" plane, for such a study. If the grains are elongated due to processing, which does happen, the transverse plane will usually show that the grains are equiaxed in shape and smaller in diameter than the true grain size. To study the effect of deformation on the grain shape of wrought metals, a minimum of two sections are needed – one perpendicular to, and the other parallel to, the direction of deformation.

HELPFUL HINTS
FOR SAMPLING

When developing a sampling plan for an unfamiliar part or component, determine the orientation of the piece relative to the original wrought or cast starting material and prepare sections on the longitudinal, transverse and planar surfaces, or radial and transverse surfaces, to reveal the desired information. Remember that the microstructure may look much more homogeneous than it is on a transverse plane compared to a longitudinal or planar surface. If the starting material was cold worked, or not fully recrystallized after hot working, grain structures will appear to be equiaxed and smaller on a transverse plane, but non-equiaxed and larger on a longitudinal or planar surface

The preparation procedure and the prepared specimen should have the following characteristics to reveal the true microsctructure*:

- Deformation induced by sectioning, grinding and polishing must be removed or be shallow enough to be removed by the etchant.

- Scratches introduced during coarse grinding must be removed; very fine polishing scratches may be tolerable for routine production work.

- Pullout, pitting, cracking of hard particles, smear, and other preparation artifacts, must be avoided.

- Relief (i.e., excessive surface height variations between structural features of different hardness) must be minimized; otherwise portions of the image will be out of focus at high magnifications.

- The surface must be flat, particularly at edges (if they are of interest) or they cannot be imaged.

- Coated or plated surfaces must be kept flat if they are to be examined, measured or photographed.

- Specimens must be cleaned adequately between preparation steps, after preparation, and after etching.

- The etchant chosen must be either general or selective in its action (reveal only the phase or constituent of interest, or at least produce strong contrast or color differences between two or more phases present), depending upon the purpose of the investigation, and must produce crisp, clear phase or grain boundaries, and strong contrast.

If these characteristics are met, then the true structure will be revealed and can be interpreted, measured and recorded. The preparation method should be as simple as possible, should yield consistent high quality results in a minimum of time and cost and must be reproducible.

Preparation of metallographic specimens [1-3] generally requires five major operations: (a) sectioning, (b) mounting (c) grinding, polishing and etching (d) image analysis and (e) hardness testing.

* Not all of these apply to every specimen.

Method Development

The methods presented in this book use times that are conservative so that the vast majority of compositions of alloys or materials of a specific type can be prepared to yield the true microstructure without artifacts or observable scratches. They were developed using an 8in [203mm] diameter platen system with six 1.25in [32mm] diameter mounted specimens. For each category, except beryllium, a wide variety of specimens with different processing histories were prepared using the stated methods.

For any metal or material category, there are compositions or processing conditions that make those specimens easier, or more difficult, to prepare compared to the "average" specimen. In general, even the most difficult specimens of a given type can be prepared perfectly using these methods with no more than one etch and re-polish (repeat last step) cycle. For perfect color etching results, it may be necessary to follow the cycle with a brief vibratory polish, depending upon the material.

When working with easier to prepare specimens, or for routine work where a high level of perfection is not required, the user can reduce the times, or eliminate one or two steps, or the final step, and get satisfactory results. Modifications to the recommended procedures are left to the user based upon their knowledge of the degree of difficulty in preparing their specimens and the desired quality of preparation.

SECTIONING

Bulk samples for subsequent laboratory sectioning may be removed from larger pieces using methods such as core drilling, band or hack-sawing, flame cutting, or similar methods. However, when these techniques are used, precautions must be taken to avoid alteration of the microstructure in the area of interest. Laboratory abrasive cutting is recommended to establish the desired plane of polish. In the case of relatively brittle materials, sectioning may be accomplished by fracturing the specimen at the desired location.

Abrasive Cutting

The most commonly used sectioning device in the metallographic laboratory is the abrasive cutter, Figure 1.1. All sectioning should be performed wet. An ample flow of coolant, with an additive for corrosion protection and lubrication, should be directed into the cut. Wet cutting will produce a smoother surface finish than a comparable dry cut and, most importantly, will guard against excessive surface damage caused by overheating. Abrasive wheels should be selected according to the manufacturer's recommendations. Table 1.1 summarizes the Buehler recommendations for our machines. Samples must be fixtured securely during cutting, and cutting pressure should be applied carefully to prevent wheel breakage. Some materials, such as commercial purity (CP) titanium, Figure 1.2, are quite prone to sectioning damage.

Wheels consist of abrasive particles, chiefly alumina or silicon carbide, and filler in a binder material that

Figure 1.1. Delta Orbital Large
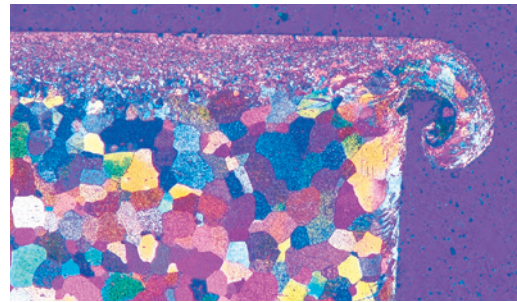
Figure 1.2. Cutting damage (top) and a "burr" after sectioning of an annealed CP titanium specimen (mod. Weck's reagent, 100X, polarized light plus sensitive tint).

may be a resin, rubber, or a mixture of resin and rubber. Alumina (aluminum oxide) is the preferred abrasive for ferrous alloys and silicon carbide is the preferred abrasive for nonferrous metals and minerals. Wheels have different bond strengths and are recommended based on the suitability of their bond strength and abrasive type for the material to be sectioned. In general, as the hardness of a material increases, the abrasives dull more quickly requiring that the binder must break-down and release the dull abrasives. As this occurs, fresh abrasive particles are revealed, allowing the system to maintain cutting speed and efficiency. If the wheel does not wear at the proper rate, dull abrasives will rub against the region being cut generating heat and possibly altering the existing true microstructure. If this heat becomes excessive, it can lead to grain or particle coarsening, softening or phase transformations, and in extreme case, to burning or melting. Different materials have different sensitivities to this problem. But, the need to balance the wheel break-down rate with the hardness of the piece being sectioned, produces the various recommendations listed for cutting different materials and metals with different hardnesses, such as steels.

The size of the abrasive cutter also affects wheel parameters. As the diameter of the wheel is increased, to permit sectioning of larger specimens, the wheel thickness is generally increased. Even so, for a given diameter wheel, there may be a range of thicknesses available. Thicker wheels are stronger but remove more material in the cut. This may, or may not, be a problem. But, thicker wheels do generate more heat during cutting than a thinner wheel, everything else being held constant. Consequently, for cases where the kerf loss or heat generation must be minimized, select the thinnest available wheel of the proper bond strength and abrasive. The AcuThin Wheels offer the thinnest possible for delicate abrasive sectioning. Buehler

SECTIONING

Table 1.1. Buehler's Abrasive Cutting Recommendations

Recommended Use	Bond	Abrasive	Available Diameters (Inches)	(mm)
General Usage Blades				
Tools, Steels, 60HRC+ Carburized Steels	Rubber Resin	Al_2O_3	9, 10, 12, 14, 16, 18	230, 254, 305, 356, 406, 455
Hard Steel 50HRC	Rubber Resin	Al_2O_3	9, 10, 12, 14, 16, 18	230, 254, 305, 356, 406, 455
Medium Hard Steel 35-50HRC	Rubber Resin	Al_2O_3	9, 10, 12, 14, 16, 18	230, 254, 305, 356, 406, 455
Soft or Annealed Steel 15-35HRC, 46-90HRB	Rubber	Al_2O_3	9, 10, 12, 14, 16, 18	230, 254, 305, 356, 406, 455
Medium Hard Nonferrous Materials, Uranium, Titamium, Zirconium	Rubber	SiC	9, 10, 12, 14, 16, 18	230, 254, 305, 356, 406, 455
Soft Nonferrous Materials, Aluminum, Brass, etc.	Rubber	SiC	9, 10, 12, 14	230, 254, 305, 356
Superalloys	Rubber	Al_2O_3	9, 10, 12, 14, 16, 18	254, 305, 356, 230, 406, 455
Thin Blades to Minimize Kerf Loss and Cutting Deformation				
Tool, Hard Steel, $\leq$ 45RC	Rubber	Al_2O_3	5, 9, 10, 12, 14	127, 230, 254, 305, 356
Tool, Hard Steel, $\geq$ 45RC	Rubber	Al_2O_3	5, 7, 9, 10, 12, 14	127, 178, 230, 254, 305, 356
Hard or Soft Nonferrous Materials	Rubber	SiC	7	178

Refer to the Buehler Product Catalogue for ordering information and exact dimensions of arbor sizes, outer diameter and thickness.

also offers diamond and CBN blades with resin bonded or Rimlock[1] metal bonded blade forms in sizes from 8in [203mm] to 16in [406mm] diameters. Resin-bonded diamond blades are ideal for cutting very hard cemented carbide specimens; Rimlock or continuous rim blades are recommended for cutting rocks and petrographic specimens.

Historically, the most common cutter design has been the so-called "chop" cutter. Basically, the blade is attached to a motor and the operator pulls a handle to drive the blade downward into the work piece. Because of the design, the blade moves through an arc as it is pulled downward, Figure 1.3a. For efficient cutting, the piece must be oriented to minimize the contact area with the wheel. For small parts, this is generally easy to do, but for larger parts, it may not be possible to orient the sample properly for optimal cutting. When a round wheel is used to cut a round bar in chop mode, the contact area is initially very small. As the cut progresses, the cut widens until the maximum diameter is reached. After this, the contact area decreases until sectioning is completed. Under application of a constant load, the pressure on the abrasive particles in the cut decreases as the contact area increases. If the pressure

applied to the grains is inadequate for cutting, then heat is generated which may not be dissipated by the coolant, which causes deformation damage, phase changes and possibly burning or melting.

A chop cutter may be set up to pulse as the load is applied, that is, the wheel is fed into the sample, and then feeding is halted momentarily, Figure 1.3b. Pulsing causes the wheel to be stress shocked which removes both dull and sharp abrasive from the wheel as new abrasive is exposed to the cut. While a better cut may be obtained, cutting time and wheel wear are increased. For small parts, a Y-feed cut (see Figure 1.3c) can provide a faster cutting time.

Optimal cutting, with the least damage to the sample, is obtained by keeping the pressure on each abrasive particle in the wheel constant and as low as possible by keeping the contact area between wheel and sample constant and low. This idea is called the minimum area of contact cutting (MACC) method. This can be achieved with several different cut types.

Several other options for MACC are the "planar"

[1]Rimlock is a registered trademark of Felker Operations.

or "saw" cut type, Figures 1.3d and 1.3e. A strip of material is removed by moving either the wheel or the work piece perpendicular to the cut direction. After this, the cutter must move back to the side where the cut began. The wheel is dropped down a fixed increment and another strip is removed. In this approach, the wheel must move to the end of the stroke, return to the starting point, and then be moved an increment downwards in each cycle. Consequently, cut times are longer, but may not be possible otherwise. The wheel traverse length must be greater than the specimen width. This machine must be programmed for each size part to obtain cutting. The primary difference in these cut types is that in planar, the cut is performed only in the forward direction while in travese and increment or

Figure 1.3. Illustration of cutter types and the wheel path during sectioning

CUTTING STYLE AND WHEEL PATH

Wheel Path

Sample

1.3a Chop Cutting
The traditional form of machine operation. Wheel contact arc is governed by sample size. Generally a struggle with large/difficult parts.

1.3b Chop Cutting with Pulsing
Wheel contact still governed by sample size. The pulsing action gives a shock to the wheel causing abrasives to break away. Wheel wear is generally high with this method.

1.3c Y-Feed Cut
The abrasive wheel is stationary and the cutting table moves forward completing a one time cut into the sample. Wheel contact arc is governed by sample size.

1.3d Planar Cut
The abrasive wheel lowers into the sample at an incremental rate before the cutting table moves in the forward motion. Wheel contact arc is determined by cut depth.

1.3e Saw Cut
The wheel contact arc can be precisely controlled via depth increment. The traverse stroke must always exceed the part length to avoid a change in wheel contact arc area. Machine needs to be set for each part.

1.3f Orbital
Similar in action to traverse and increment but on a curved path. Simpler and quicker in operation. Part size is irrelevant as the orbital action produces a minimum contact arc area during cutting.

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saw, the cut is performed in both directions

Orbital cutting, Figure 1.3f, combines the best features of chop, oscillation and traverse-and-increment cutters, while minimizing cutting time. As the wheel is fed into the sample, the arbor is also rotated in a small ellipse planar with the wheel permitting the wheel to cut a small strip of material from the sample. But no time is lost, as with the traverse and increment cutter, as the wheel is ready to cut on the original side. With the elliptical orbit and a controlled feed, the conditions for minimum area of contact cutting are obtained, irrespective of work piece size. Special programming is not needed. The orbital cutting concept is available in many of the Delta cutters from Buehler. These also feature SmartCut, which will sense motor overloading during a cut, if it occurs, and reduce the feed rate automatically. When it is safe to return to the original feed rate, SmartCut will do so.

Precision Sectioning

Precision cutters, Figure 1.4, are commonly used in metallographic preparation to section materials that are small, delicate, friable, extremely hard, where the cut must be made as close as possible to a feature of interest, or where the cut width and material loss must be minimal. As the name implies, this type of saw is designed to make very precise cuts. They are smaller in size than the usual laboratory abrasive cut-off saw and use much smaller blades, typically from 3 - 8in [76 - 203mm] in diameter. These blades can be made of copper-based alloys or copper plated steel with diamond or cubic boron nitride abrasive bonded to the periphery of the blade; or, they can be an abrasive wheel using alumina or silicon carbide abrasives with a rubber-based bond. Blades for the precision saws are much thinner than the abrasive wheels used in an abrasive cutter and the load applied during cutting is much less. Consequently, less heat is generated during cutting and damage depths are reduced. While pieces with a small section size, that would normally be sectioned with an abrasive cutter can be cut with a precision saw, the cutting time will be appreciably greater but the depth of damage will be much less. Precision saws are widely used for sectioning sintered carbides, ceramic materials, thermally sprayed coatings, printed circuit boards, electronic components, bone, teeth, etc. Table 1.2 lists selection criteria for precision saw blades. Blades for precision saws are available

with different abrasive sizes and bonds to provide optimum cutting for a wide variety of applications.
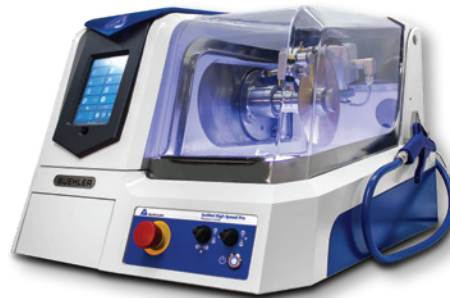
Figure 1.4. IsoMet High Speed Precision Cutter

HELPFUL HINTS FOR SECTIONING

When cutting a difficult specimen with the recommended consumable abrasive wheel, if the cutting action has become very slow, pulse the applied force. This will help break down the abrasive bonding, exposing fresh, sharp abrasives to enhance the cutting action. However, if you are using a resin-bonded diamond blade to cut cemented carbides, or other very hard materials, do not pulse the applied force, as this will shorten wheel life.

Table 1.2. Selection Criteria for Precision Blades

BLADE SERIES	3 x 0.007in [76 x 0.15mm]	4 x 0.012in [102 x 0.3mm]	5 x 0.015in [127 x 0.4mm]	6 x 0.015in [152 x 0.5mm]	7 x 0.025in [178 x 0.6mm]	8 x 0.035in [203 x 0.9mm]
IsoMet Diamond Wafering Blades						
Series 30HC, polymers, rubber, soft gummy materials			X**		X**	X
Series 20HC, aggressive sectioning of metals			X*		X	X
Series 15HC, metal matrix composites, PCBs, bone, Ti, TSC	X	X	X	X	X	X
Series 20LC, hard tough materials, structural ceramics			X*		X	X
Series 15LC, hard brittle materials, glass, Al_2O_3, Zr_2O_3, concrete	X	X	X	X	X	X
Series 10LC, medium to soft ceramics, glass fiber reinforced composites	X		X		X*	X
Series 5LC, soft friable ceramics, composites with fine reinforcing, CaF_2, MgF_2, carbon composites	X		X			
IsoCut CBN Wafering Blades						
Low Concentration, Fe, Co, Ni based alloys and superalloys	X	X	X	X	X	X
High Concentration Fe, Co, Ni based alloys and superalloys		X	X	X	X	X

General Usage and AcuThin Abrasive Wheels For sectioning small, delicate specimens or where minimal deformation and kerf loss is the primary concern

	Bond/ Abrasive	5 x 0.019in [127 x 0.5mm]	7 x 0.03in [178 x 0.8mm]
Tool Steel, hard Steel ≥ HRC45	R/Al_2O_3	X	
Medium hard, soft steel ≤ HRC45	R/Al_2O_3	X	
Soft materials	R/Al_2O_3		X
Tough materials or general use	R/SiC		X

*Alternate blade thickness of 0.020in [0.5mm] **Alternate blade thickness of 0.030in [0.8mm]*
For a complete listing of Buehler consumable supplies for use with the IsoMet Precision Cutters, please refer to the Buehler Product Catalogue.

MOUNTING

The primary purpose of mounting metallographic samples is for convenience in handling samples of difficult shapes or sizes during the subsequent steps of metallographic preparation and examination. A secondary purpose is to protect and preserve extreme edges or surface defects during metallographic preparation. The method of mounting should in no way be injurious to the microstructure of the specimen. Pressure and heat are the most likely sources of injurious effects.

Phenolic plastics were introduced to metallography in 1928 for encapsulating specimens by hot compression mounting. Prior to that time, samples were prepared unmounted, or mounted in flours of sulfur, wax or low-melting point alloys, such as Wood's metal. These "mounting compounds" were not without their problems. Introduction of polymers was a big improvement over these methods. Subsequently, many polymers were evaluated for use as mounting compounds, as they were introduced to the market. Development of castable resins in the 1950's added new resins to the metallographer's tool chest. The simplicity of mounting without using a press, and their low curing temperatures, made castable resins an attractive alternative.

Clamp Mounting

Clamps have been used for many years to mount cross sections of thin sheet specimens. Several samples can be clamped conveniently in sandwich form making this a quick, convenient method for mounting thin sheet specimenss. When done properly, edge retention is excellent, and seepage of fluids from crevices between specimens does not occur. The outer clamp edges should be beveled to minimize damage to polishing cloths. If clamps are improperly used so that gaps exist between specimens, fluids and abrasives can become entrapped and will seep out obscuring edges and can cause cross contamination. This problem can be minimized by proper tightening of clamps, by use of plastic spacers between specimens, or by coating specimen surfaces with epoxy before tightening.

Compression Mounting

The most common mounting method uses pressure and heat to encapsulate the sample with a thermosetting or thermoplastic mounting material. Common thermosetting compounds include phenolic (PhenoCure), diallyl phthalate and epoxy (EpoMet) while methyl methacrylate (TransOptic) is the most commonly used thermoplastic mounting compounds. Table 2.1 lists the characteristics of the compression mounting compounds. Both thermosetting and thermoplastic materials require heat and pressure during the molding cycle; but, after curing, mounts made of thermoplastic compounds must be cooled under pressure to at least 158°F [70°C] while mounts made of thermosetting materials may be ejected from the mold at the maximum molding temperature. However, cooling thermosetting compounds under pressure to near ambient temperature before ejection will significantly reduce shrinkage gap formation. Never rapidly cool a thermosetting mount with water after hot ejection from the molding temperature. This causes the metal to pull away from the mounting

Materials	General Purpose	Best Edge Retention; Very Low Shrinkage; Fine Particle Size; Fills Smallest Crevices	Best Edge Retention; Very Low Shrinkage	Near Zero Electrical Resistance SEM - EDS/WDS	Clear
Table 2.1: Characteristics of Compression Mounting Compounds					
Ceramics	PhenoCure	EpoMet F	EpoMet G	ProbeMet	TransOptic
Steels	PhenoCure	EpoMet F	EpoMet G	ProbeMet	TransOptic
Plated Layers	PhenoCure	EpoMet F	EpoMet G	ProbeMet	TransOptic
Aluminum	PhenoCure			ProbeMet	TransOptic
Copper/ Brass	PhenoCure			ProbeMet	TransOptic
Color	Black, Red or Green	Black	Black	Copper	Transparent
Temperature	300°F [150°C]	300°F [150°C]	300°F [150°C]	300°F [150°C]	350°F [177°C]
Pressure	4200 psi [290 bar]	4200 psi [290 bar]	4200 psi [290 bar]	4200 psi [290 bar]	2100 psi [145 bar]

[1]Rimlock is a registered trademark of Felker Operations.

compound producing shrinkage gaps that promote poor edge retention, see Figure 2.1, because of the different rates of thermal contraction. EpoMet, a thermosetting epoxy, provides the best edge retention, Figure 2.2, of these compounds and is virtually unaffected by hot or boiling etchants while phenolics are badly damaged.
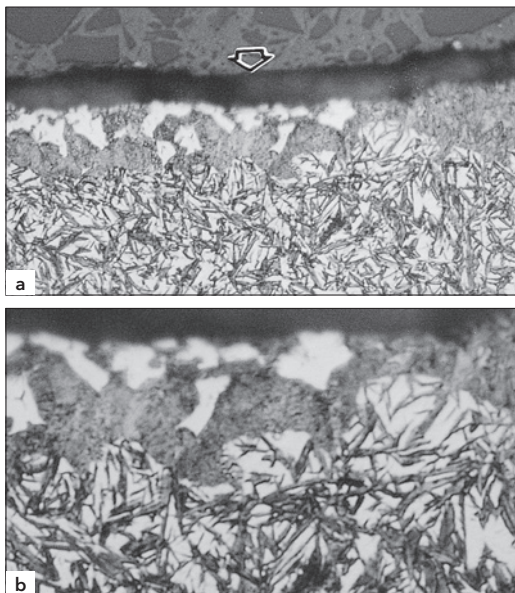

Figure 2.1. Edge retention of this improperly carburized 8620 alloy steel was degraded by a shrinkage gap between the specimen and the phenolic mount: a) 500X; b) 1000X (2% nital).

Mounting presses vary from simple laboratory jacks with a heater and mold assembly to full automated devices that include the SmartCool intelligent cooling system that ends the process as soon as the specimen is safe to handle, as shown in Figure 2.3. An advantage of compression mounting is production of a mount of a predicable, convenient size and shape. Further, considerable information can be engraved on the backside – this is always more difficult with unmounted specimens. Manual, or hand, polishing is simplified, as the specimens are easy to hold.
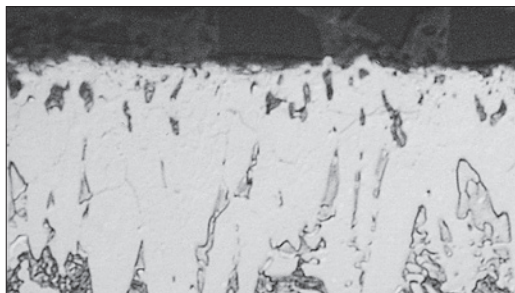
Figure 2.2. Excellent edge retention of a borided 42CrMo4 alloy steel specimen mounted in EpoMet resin (1000X, 2% nital).

Also, placing a number of mounted specimens in a holder for semi- or fully-automated grinding and polishing is easier with standard mounts than for unmounted specimens. Mounted specimens

Figure 2.3. SimpliMet 4000 Automatic Mounting Press

are easier on the grinding/polishing surfaces than unmounted specimens.

Castable Mounting Systems

Most castable mounting systems require neither pressure nor external heat and are recommended for mounting specimens that are sensitive to heat and/or pressure. Acrylic systems, such as VariDur and SamplKwick, are the most widely used castable systems due to their low cost and short curing time. However, shrinkage can be a problem with acrylics. Epoxy systems, although more expensive than acrylics, are commonly used because epoxy will physically adhere to specimens, have low shrinkage, and can be drawn into cracks and pores, particularly if a vacuum impregnation chamber, as shown in Figure 2.4, is employed and a low viscosity epoxy, such as EpoThin 2, is used. Epoxies are very suitable for mounting fragile or friable specimens and corrosion or oxidation specimens. Dyes or fluorescent agents may be added

Figure 2.4. Vacuum Impregnation Equipment

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to epoxies for the study of porous specimens such as thermal spray coated specimens. Most epoxies are cured at room temperature, and curing times can vary from 1 to 9 hours. Some can be cured at slightly elevated temperatures in less time, as long as the higher temperature does not adversely affect the specimen. Table 2.2 lists the characteristics of castable systems.

Cast epoxy systems provide better edge retention than cast acrylic systems, mainly due to the better adhesion of epoxy to the specimen and their lower shrinkage. Acrylics usually do not bond to the specimen and a gap is sometimes formed between specimen and mount, with VariDur 3003 being the exception. When a shrinkage gap is present, edge retention is usually poor. To improve edge retention with castable mounts, the specimen can be plated with electroless nickel, or Flat Edge Filler particles can be added to the system. To obtain electrical conductivity, Con-ductive Filler particles can be added to the castable systems, though the viscosity increases.

When preparing castable resin mounts, particularly epoxy mounts by manual "hand" methods, the metallographer will observe that the surface tension between the mount and the working surface is much higher than with a compression mount. This can make holding the mount more challenging. If automated devices are used, the metallographer may hear "chatter," or noise, during rough grinding due to the greater surface tension. The chatter can be reduced or stopped by changing to contra mode (head and platen rotate in opposite directions).

Acrylics, and some epoxies, do generate considerable heat during curing and this can be strongly influenced by the molding technique used. Nelson [4] measured the exotherm produced by polymerizing an acrylic system using two procedures: a glass mold on a glass plate (insulative) and an aluminum mold on an aluminum plate (conductive).

Table 2.1: Characteristics of Compression Mounting Compounds						
Name	Type	Peak Temperature	Shore D Cure Hardness*	Time	Recommended Mold	Comments
EpoThin 2	Epoxy	<149°F [65°C]	78	9 hours	Any	Clear, very low viscosity, low shrinkage, best for vacuum impregnation
EpoxiCure 2	Epoxy	<104°F [40°C]	80	6 hours	Any	Clear, general pupose epoxy system, low shrinkage
EpoKwick FC	Epoxy	<250°F [121°C]	82	2 hours	Any	Clear, fast curing epoxy system, low shrinkage, very low viscosity
EpoHeat CLR	Epoxy	<324°F [162°C]	82	1 hour	Any	Clear epoxy system, long pot life allows product to be mixed in large batches
VariDur 3003	Acrylic	<252°F [122°C]	90	15-30 minutes	Any	Very low shrinkage with high hardness
SamplKwick	Acrylic	<179°F [81°C]	85	5-8 minutes	Any	Translucent, general purpose system, some shrinkage
VariKleer	Acrylic	<212°F [100°C]	84	5-15 minutes	SamplKup	Clear, general purpose acrylic system, requires pressure vessel for clear mounts, minimal shrinkage. To acheive crystal clear samples, reusable silicon or EPDM molds should not be used.
VariDur 10	Acrylic	<212°F [100°C]	80	8 minutes	Any	Semi transparent, low odor system, low shrinkage, high viscosity
VariDur 200	Acrylic	<212°F [100°C]	85	5-8 minutes	Any	Dark blue, low odor system, low shrinkage, high viscosity

Hardness differences appear negligible but abrasion resistance has a significant effect on edge rounding

Polymerization produced a maximum exotherm of 270°F [132°C] using the insulative approach but only 108°F [42°C] using the conductive approach. Note that 270°F [132°C] is not much less than the 302°F [150°C] temperature used in compression mounting. Nelson also measured the exotherm produced when an epoxy system was cured in a phenolic ring form placed on a pasteboard base. Although this was an insulative approach, the maximum temperature during polymerization was only 45°F [7°C], a vast improvement over the acrylics.

Nelson's work applies to specific acrylic and epoxy resins molded upon specific conditions. While the epoxy that he used exhibited a low exotherm, this does not imply that all epoxy systems will exhibit such low exotherms in polymerization. Epoxy systems that cure in short time periods develop much higher exotherms, that can even exceed that of acrylic systems. In addition to the speed of curing of the epoxy system, other factors do influence the magnitude of the exotherm during polymerization. The larger the mass of epoxy in the mount, the faster it will set and the greater the exotherm. Indeed, very large mounts can generate enough heat to crack extensively. Heating the system makes it less viscous and speeds up curing, also generating more heat during polymerization. The mold material also can influence curing time and temperature. For example, EpoxiCure 2 cures fastest in SamplKup plastic molds, slower in ring forms, and still slower in the reuseable EPDM mounting cups. Consequently, the exotherm will be greater when using the SamplKup type mold and lowest when using the EPDM mounting cups. All of these factors must be considered if the exotherm must be minimized.

Edge Preservation

Edge preservation is a classic preparation problem and many "tricks" have been promoted (most pertaining to mounting, but some to grinding and polishing) to enhance edge flatness. These methods [2] include the use of backup material in the mount, the application of coatings to the surfaces before mounting or the addition of a filler material to the mounting system. Plating [2] of a compatible metal on the surface to be protected (electroless nickel has been widely used) is generally considered to be the most effective procedure. However, image contrast at an interface between a specimen and the electroless nickel may be inadequate for certain evaluations. Figure 2.5 shows the surface of a specimen of 1215 free-machining steel that was salt bath nitrided. One specimen was plated with electroless nickel; both were mounted in EpoMet G. It is hard to tell where the nitrided layer stops for the plated specimen, Figure 2.5a, which exhibits poor image contrast between the nickel and the nitrided surface. This is not a problem for the non-plated specimen, Figure 2.5b.
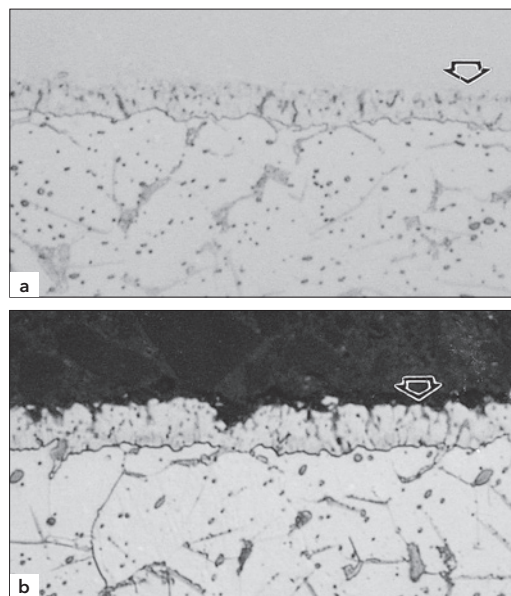
Figure 2.5. Example of a) poor coating edge visibility due to a lack of contrast between the protective nickel plating and the salt bath nitrided surface (arrow) of 1215 free-machining carbon steel; and, b) good contrast and visibility between EpoMet resin and nitrided surface (arrow) plus excellent edge retention (1000X, 2% nital).

Introduction of new technology has greatly reduced edge preservation problems [5,6]. Gaps that form between specimen and mount media are a major contributor to edge rounding, as shown in Figure 2.1. Staining at shrinkage gaps may also be a problem, as demonstrated in Figure 2.6. Use of semi-automatic and automatic grinding/polishing equipment, rather than manual preparation, increases surface flatness and edge retention. To achieve the best results, however, the position of the specimen holder, relative to the platen, must be adjusted so that the outer edge of the specimen holder rotates out over the edge of the surface on the platen during grinding and polishing, particularly for 8in [203mm] diameter platens. The use of harder, woven or non-woven, napless surfaces for

polishing with diamond abrasives (rather than softer cloths such as canvas, billiard and felt) maintains flatness. Final polishing with low nap cloths for short times introduces very little rounding compared to use of higher nap, softer cloths.

Figure 2.6. Staining (thin arrows) due to etchant bleed out from a shrinkage gap (wide arrows) between the phenolic mount and the M2 high speed steel specimen (500X, Vilella's reagent).

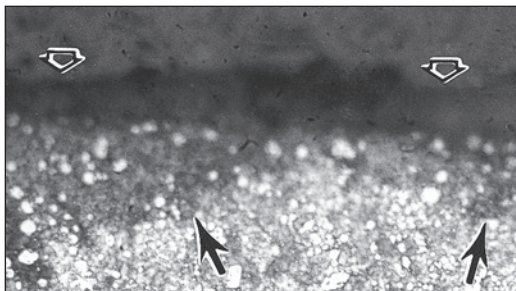These procedures will produce better edge retention with all thermosetting and thermoplastic mounting compounds. Nevertheless, there are still differences among the polymeric materials used for mounting. Thermosetting compounds provide better edge retention than thermoplastic compounds. Of the thermosetting compounds, diallyl phthalate provides little improvement over the much less expensive phenolic compounds. The best results are obtained with EpoMet G or EpoMet F an epoxy based thermosetting compounds that contains a filler material. For comparison, Figure 2.7 shows micrographs of a nitrided 1215 specimen mounted in a phenolic (Figure 2.7a), and in methyl methacrylate (Figure 2.7b), at 1000X. These specimens were prepared in the same specimen holder as those shown in Figure 2.5, but neither displays acceptable edge retention at 1000X. Figure 2.8 shows examples of perfect edge retention, as also demonstrated in Figure 2.5.

Very fine aluminum oxide spheres have been added to castable epoxy systems to improve edge retention, but this is really not a satisfactory solution as the particles are extremely hard (~2000 HV) and their grinding-polishing characteristics are incompatible with softer metals placed inside the mount. Howver, use of a soft ceramic shot (~775 HV) has grinding/polishing characteristics compatible with metallic specimens placed in the mount. Figure 2.9 shows an example of edge retention with the Flat Edge Filler soft ceramic shot in an epoxy system.
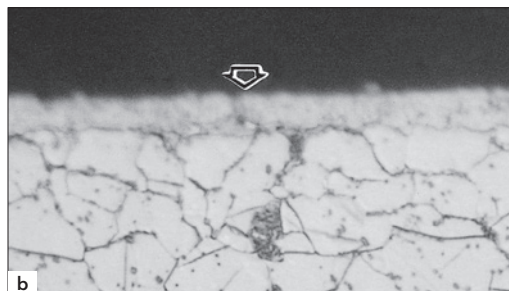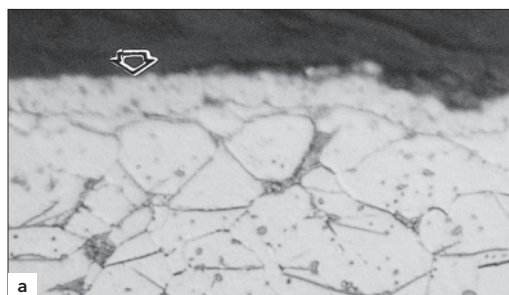
Figure 2.7. Poor edge retention (arrows) at the salt bath nitrided surface of 1215 free-machining carbon steel mounted in a) phenolic resin and in b) methyl methacrylate and polished in the same holder as those shown in Figure 2.5 (1000X, 2% nital).
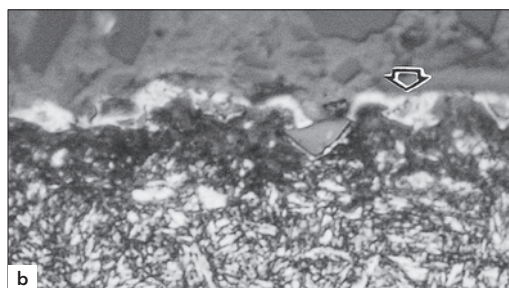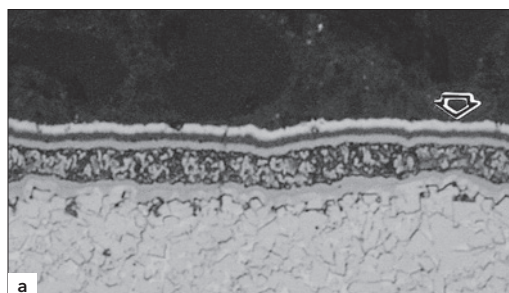
Figure 2.8. Excellent edge retention for a) complex coated (arrow) sintered carbide insert (1000X, Murakami's reagent) and for b) iron nitrided (arrow points to a white iron nitride layer; an embedded shot blasting particle is to the left of the arrow) H13 hot work die steel specimen (1000X, 2% nital). EpoMet was used in both cases.

Following are general guidelines for obtaining the best possible edge retention. All of these factors contribute to the overall success, although some are more critical than others.
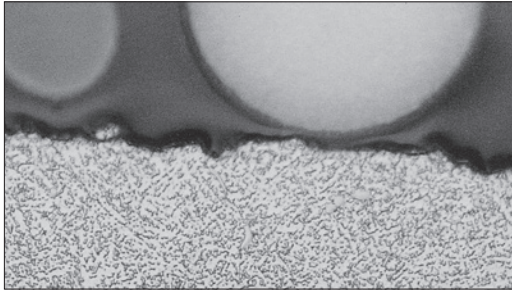
Figure 2.9. Flat Edge Filler shot was added to Epoxicure 2 resin to improve the edge retention of this annealed H13 hot work die steel specimen (500X, 4% picral).

- Properly mounted specimens yield better edge retention than unmounted specimens, as rounding is difficult, if not impossible, to prevent at a free edge. Compression mounts yield better edge preservation than castable systems.

- Electrolytic or electroless Ni plating of the surface of interest provides excellent edge retention. If the compression mount is cooled too quickly after polymerization, the plating may be pulled away from the specimen leaving a gap. When this happens, the plating is ineffective for edge retention.

- Thermoplastic compression mounting compounds are less effective than thermosetting compounds. The best thermosetting compound for edge retention is EpoMet G, an epoxy-based resin containing a hard filler material.

- Never hot eject a thermosetting compound after polymerization and cool it quickly to ambient (e.g., by cooling it in water) as a gap will form between specimen and mount due to the differences in thermal contraction rates. Fully automated mounting presses cool the mounted specimen to near ambient temperature under pressure and this greatly minimizes gap formation due to shrinkage.

- Automated grinding-polishing equipment produces flatter specimens than manual preparation.

- Use the central force mode (defined later in

the text) with an automated grinder-polisher as this method provides better flatness than single, or individual, pressure mode (defined later in the text).

- Orient the position of the smaller diameter specimen holder so that, as it rotates, its periphery slightly overlaps the periphery of the larger diameter platen.

- Use PSA-backed SiC grinding paper (if SiC is used), rather than plain backed paper on the platen and a peripheral hold-down ring, and PSA-backed or magnetic polishing cloths rather than stretched cloths.

- Apex DGD and UltraPrep diamond grinding discs produce excellent flat surfaces for a wide variety of materials.

- Use "hard" napless surfaces for rough polishing (until the final polishing step), such as TexMet C, UltraPol or UltraPad cloths, and fine polishing, such as a TriDent cloth. Use a napless, or a low- to medium-nap cloth, depending upon the material being prepared, for the final step and keep the polishing time brief.

- Rigid grinding discs, such as the Apex Hercules H and S discs, produce excellent flatness and edge retention and should be used whenever possible.

HELPFUL HINTS FOR MOUNTING

Epoxy is the only resin that will physically adhere to a specimen. If its viscosity is low, epoxy can be drawn into pores and cracks by vacuum impregnation. Acrylics are not ideal for vacuum impregnation due to short cure times and generally have high viscosity.

Castable resins are sensitive to shelf life. It is a good practice to date your containers when you get them.

GRINDING & POLISHING

Grinding

Grinding should commence with the finest grit size that will establish an initially flat surface and remove the effects of sectioning within a few minutes. An abrasive grit size of 180-240 [P180-P280] is coarse enough to use on specimen surfaces sectioned by an abrasive wheel. Hack-sawed, bandsawed, or other rough surfaces usually require abrasive grit sizes in the range of 120-180 grit [P120-P180}. The abrasive used for each succeeding grinding operation should be one or two grit sizes smaller than that used in the preceding step. A satisfactory fine grinding sequence might involve SiC papers with grit sizes of 240, 320, 400, and 600 grit [P280, P400, P800 and P1200]. This sequence is used in the "traditional" approach.

As with abrasive cutting, all grinding steps should be performed wet provided that water has no adverse effects on any constituents of the microstructure. Wet grinding minimizes specimen heating, and prevents the abrasive surface from becoming loaded with metal removed from the specimen being prepared.

Each grinding step, while producing damage itself, must remove the damage from the previous step. The depth of damage decreases with the abrasive size but so does the metal removal rate. For a given abrasive size, the depth of damage introduced is greater for soft materials than for hard materials.

For automated preparation using a multiple-specimen holder, the intital step is called planar grinding. This step must remove the damage from sectioning while establishing a common plane for all of the specimens in the holder, so that each specimen is affected equally in subsequent steps. Silicon carbide and alumina abrasive papers are commonly used for the planar grinding step and are very effective. Besides these papers, there are a number of other options available. One option is to planar grind the specimens with a conventional alumina or Silicon carbide grinding stone. This requires a special purpose machine, as the stone must rotate at a high speed, ≥1500 rpm, to cut effectively. The stone must be dressed regularly with a diamond tool to maintain flatness and refresh the abrasive surface for effective grinding.

Other materials have also been used both for the planar grinding stage or, afterwards, to replace SiC paper. For very hard materials such as ceramics and sintered carbides, one, or more, metal-bonded or resin-bonded diamond discs (the traditional type) with grit sizes from about 240 to 9µm can be used. The traditional metal- or resin-bonded diamond disc has diamond spread uniformly over its entire surface. Apex DGD and Apex DGD Color, ranging in grit size from 320 to 0.5µm, are also available as resin bonded diamond grinding discs covering a variety of material applications. An alternate type of disc, the UltraPrep disc, has diamond particles applied in small spots to the disk surface, so that surface tension is lessened. UltraPrep metal-bonded discs are available in six diamond sizes from 125 to 6µm while UltraPrep resin bonded discs are available in three diamond sizes from 30 to 3µm. Once planar surfaces have been obtained, there are several single-step procedures available for avoiding the finer SiC papers. These include the use of platens, thick woven polyester cloths, silk, or rigid grinding discs. With each of these, an intermediate diamond size, generally 9 to 3µm, is used.

Grinding Media

The grinding abrasives commonly used in materials preparation are silicon carbide (SiC), aluminum oxide (Al_2O_3), emery (Al_2O_3 - Fe_3O_4), composite ceramics and diamond. Emery paper is rarely used today in materials preparation due to its low cutting efficiency. SiC is more readily available as waterproof paper than aluminum oxide. These abrasives are bonded to paper, polymeric or cloth backing materials in the form of discs and belts of various sizes. Limited use is made of standard grinding wheels with abrasives embedded in a bonding material. The abrasives may be used also in powder form by charging the grinding surfaces with the abrasive in a premixed slurry or suspension. SiC particles, particularly with the finer size papers, embed easily when grinding soft metals, such as Pb, Sn, Cd and Bi (see Figure 3.1). Embedding of diamond abrasive is also a problem with these soft metals and with aluminum, but mainly with slurries when napless cloths are used, see Figure 3.2.

Silicon Carbide paper manufactured in the United States is typically made according to the ANSI/CAMI standard (B74. 18-1996) while paper manufactured in Europe are made according to the FEPA standard (43-GB-1984, R 1993). Both
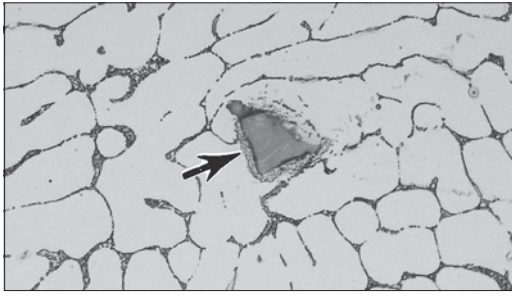
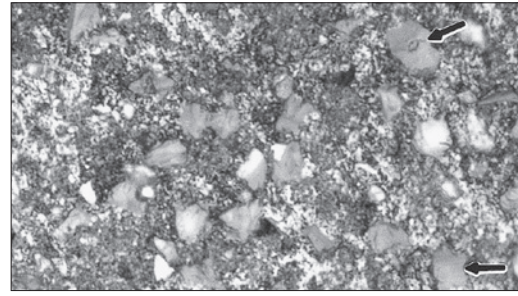Figure 3.1. SiC grit particle (arrow) embedded in a 6061-T6 aluminum weldment (500X, aqueous 0.5% HF).

Figure 3.2. 6μm diamond particles (arrows) embedded in lead (1000X).

standards use the same methods for sizing the abrasives and the same standards to calibrate these devices (sieving for the coarsest grits, sedimentation grading for intermediate grits (240-600 [P280-P1200]), and the electrical resistance method for very fine grit sizes). The grit size numbering systems differ above 180 grit [P180], but equivalent sizes can be determined using Table 3.1. As with many standards, they are not mandatory and manufacturers can, and do, make some of their papers to different mean particle sizes than defined in these specifications. There is a philosophical difference in the two systems. ANSI/CAMI papers use a wider particle size distribution (centered on the mean size) than FEPA papers. A

broader size range allows cutting to begin faster at lower pressures than with a narrower size range, so less heat is generated and less damage results. However, the broader size range does produce a wider range of scratch depths; but, these should be removed by the next step in the preparation sequence. Generation of less damage to the structure is considered to be more important than the surface finish after a particular grinding step, as it is the residual damage in the specimen that may prevent us from seeing the true microstructure at the end of the preparation sequence.

Table 3.1: European/US Equilivancy Guide				
FEPA (Europe)		ANSI/CAMI (US)		
Grit Number	Micron	Grit Number	Micron	Emery Grit
P60	269	60	268	
P80	201	80	188	
P100	162	100	148	
P120	127	120	116	
P180	78	180	78	3
P240	58.5	220	66	2
P280	52.2	240	51.8	
P320	46.2			
P360	40.5	280	42.3	1
P400	35	320	34.3	0
P500	30.2	360	27.3	
P600	25.8			
P800	21.8	400	22.1	00
P1000	18.3	500	18.2	000
P1200	15.3	600	14.5	
P1500	12.6	800	12.2	0000
P2000	10.3	1000	9.2	
P2500	8.4	1200	6.5	
P4000*	5.0*			

The chart shows the midpoints for the size ranges for ANSI/CAMI graded paper according to ANSI standard B74.18-1996 and for FEPA graded paper according to FEPA standard 43-GB-1984 (R1993). The ANSI/CAMI standard lists SiC particles sizes ranges up to 600 grit paper. For finer grit ANSI/CAMI papers, the particles sizes come from the CAMI booklet, Coated Abrasive (1996).

*FEPA grades finer than P2500 are not standardized and are graded at the discretion of the manufacturer. In practice, the above standard values are only guidelines and individual manufactures may work to a different size range and mean value.

GRINDING & POLISHING

Grinding Equipment

A Planar grinding machine, like the PlanarMet 300 shown in Figure 3.3, utilizes a fixed abrasive stone for rapid sample grinding. This type of automated grinder produces planar samples in 1-2 minutes replacing up to 3 traditional grinding steps. Machines like this can be bench-top or floor standing and typically utilize the same sample holders as grinder-polishers to integrate the transition to the next step. Stationary grinding papers, often used by students, but uncommon in industrial use, are supplied in strips or rolls. The specimen is rubbed against the paper from top to bottom. Grinding in one direction is usually better for maintaining flatness than grinding in both directions. This procedure can be done dry for certain delicate materials, but water is usually added to keep the specimen surface cool and to carry away the grinding debris.

Figure 3.3. PlanarMet 300 Planar Grinder

Belt grinders are usually present in most laboratories. These types of devices use coarse abrasive papers from 60 [P60] to 240 [P280] grit, and are mainly used for removing burrs from sectioning, for rounding edges that need not be preserved for examination, for flattening cut surfaces to be macroetched, or for removing sectioning damage.

Lapping is an abrasive technique in which the abrasive particles roll freely on the surface of a carrier disc. During the lapping process, the disc is charged with small amounts of a hard abrasive such as diamond or silicon carbide. Lapping discs can be made of many different materials; cast iron and plastic are used most commonly. Lapping produces a flatter specimen surface than grinding, but it does not remove metal in the same manner

as grinding. Some platens, referred to as laps, are charged with diamond abrasive in a carrier, such as paste, oil based suspension or water based suspension. Initially the diamond particles roll over the lap surface (just as with other grinding surfaces), but they soon become embedded and cut the surface producing microchips.

Figure 3.4. EcoMet 250 Grinder-Polisher

Polishing

Polishing is the final step, or steps, in producing a deformation-free surface that is flat, scratch free, and mirror-like in appearance. Such a surface is necessary to observe the true microstructure for subsequent interpretation, testing or analysis, both qualitative and quantitative. The polishing technique used should not introduce extraneous structures such as disturbed metal (Figure 3.5), pitting (Figure 3.6), dragging out of inclusions, "comet tailing" (Figure 3.7), staining (Figure 3.8) or relief
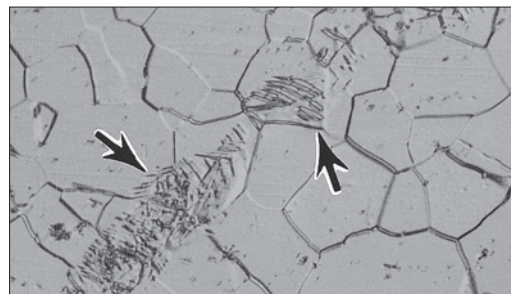

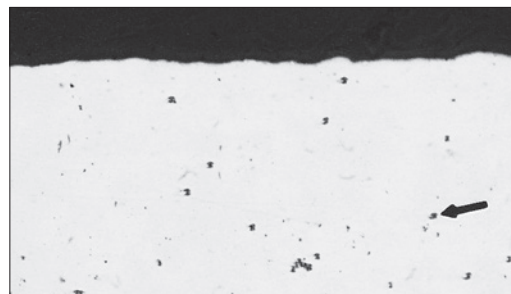*Figure 3.5. Preparation damage (arrows) in annealed CP titanium (500X, DIC, Kroll's reagent).*

Figure 3.6. Pitting (arrow) on the surface of a cold-drawn brass (Cu-20% Zn) specimen (100X)
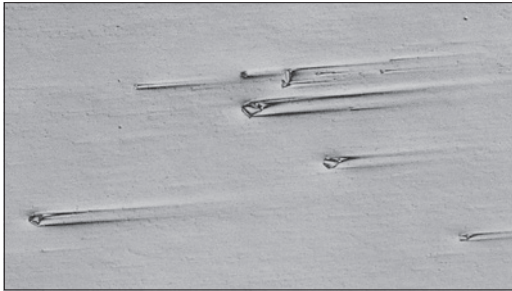
Figure 3.7. "Comet tails" at large nitrides in an annealed H13 hot work die steel specimen (200X, DIC).
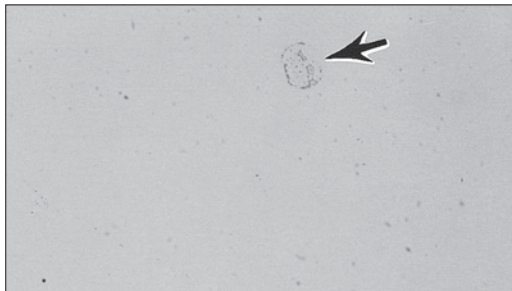
Figure 3.8. Staining (arrow) on the surface of a of Ti-6% Al-2% Sn-4% Zr-2% Mo prepared specimen (200X).

(height differences between different constituents, or between holes and constituents (Figures 3.9 and 3.10). Polishing usually is conducted in several stages. Traditionally, coarse polishing generally was conducted with 6 or 3µm diamond abrasives charged onto napless or low-nap cloths. For hard materials, such as through hardened steels, ceramics and cemented carbides, an additional coarse polishing step may be required. The initial coarse
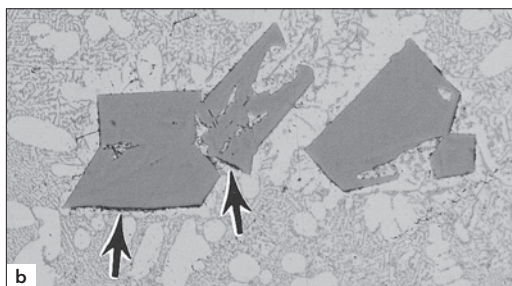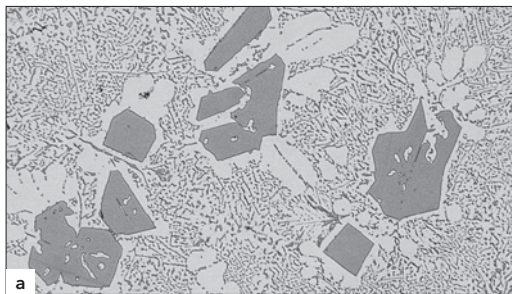
Figure 3.9. Examples of freedom from relief a) and minor relief b) at the edges of large primary hypereutectic silicon particles in an as-cast Al-19.85% Si specimen (200X, aqueous 0.5% HF).

polishing step may be followed by polishing with 1µm diamond on a napless, low nap, or medium nap cloth. A compatible lubricant should be used sparingly to prevent overheating or deformation of the surface. Intermediate polishing should be performed thoroughly so that final polishing may be of minimal duration. Manual, or hand, polishing, is usually conducted using a rotating wheel where the operator rotates the specimen in a circular path counter to the wheel rotation direction.
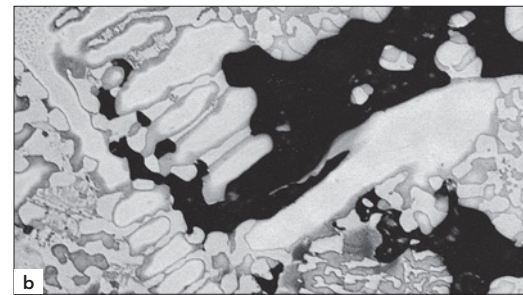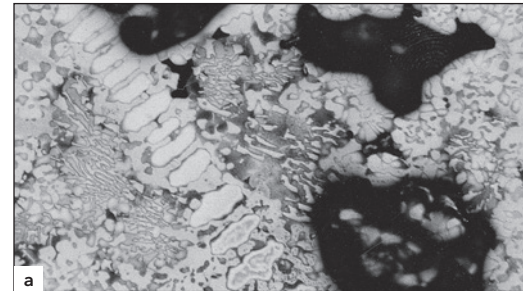
Figure 3.10. Differences in relief control in a brazed specimen containing shrinkage cavities: a) poor control; and, b) good control (100X, glyceregia).

Mechanical Polishing

The term "mechanical polishing" is frequently used to describe the various polishing procedures involving the use of fine abrasives on cloth. The cloth may be attached to a rotating wheel or a vibratory polisher bowl. Historically, cloths have been either stretched over the wheel and held in place with an adjustable clamp on the platen periphery, or held in place with a pressure sensitive adhesive (PSA), magnetic or high friction surface-bonded to the back of the cloth. If a stretched cloth moves under the applied pressure during polishing, cutting will be less effective. If an automated polishing head is used, stretched cloths are more likely to rip, especially if unmounted specimens are being prepared. In mechanical polishing, the specimens are held by hand, held mechanically in a fixture, or merely confined within the polishing area, as with the VibroMet 2 polisher.

GRINDING & POLISHING

Manual "Hand" Polishing

Aside from the use of improved polishing cloths and abrasives, hand-polishing techniques still follow the basic practice established many years ago:

1. Specimen Movement. The specimen is held with one or both hands, depending on the operator's preference, and is rotated in a direction counter to the rotation of the polishing wheel. In addition, the specimen is continually moved back and forth between the center and the edge of the wheel, thereby ensuring even distribution of the abrasive and uniform wear of the polishing cloth. (Some use a small wrist rotation while moving the specimen from the center to the edge of one side of the wheel.) After each step, the specimen is rotated 45 to 90° so that the abrasion is not unidirectional.

2. Polishing Pressure. The correct amount of applied pressure must be determined by experience. In general, a firm hand pressure is applied to the specimen.

3. Washing and Drying. The specimen is washed by swabbing with a liquid detergent solution, rinsed in warm running water, then with ethanol, and dried in a stream of warm air. Alcohol usually can be used for washing when the abrasive carrier is not soluble in water or if the specimen cannot tolerate water. Ultrasonic cleaning may be needed if the specimens are porous or cracked.

4. Cleanness. The precautions for cleanness, as previously mentioned, must be strictly observed to avoid contamination problems. This involves the specimen, the user's hands, and the equipment.

Automated Polishing

Mechanical polishing can be automated to a high degree using a wide variety of devices ranging from relatively simple systems, Figure 3.11, to rather sophisticated, programmable or even touch screen operated devices. Units also vary in capacity from a single specimen to a half dozen or more at a time and can be used for all grinding and polishing steps. These devices enable the operator to prepare a large number of specimens per day with a higher degree of quality than hand polishing and at reduced consumable costs. Automatic polishing

Figure 3.11. EcoMet 300 Grinder-Polisher and AutoMet 300 Power Head

devices produce the best surface flatness and edge retention. There are two approaches for handling specimens. Central force utilizes a specimen holder with each specimen held in place rigidly. The holder is pressed downward against the preparation surface with the force applied to the entire holder. Central force yields the best edge retention and specimen flatness. If the results after etching are inadequate, the specimens must be placed back in the holder and the entire preparation sequence must be repeated. Instead of doing this, most technicians will repeat the final step manually and then re-etch the specimen.

The second method utilizes a specimen holder where the specimens are held in place loosely. Force is applied to each specimen by a piston, hence the term "individual or single force" for this approach. This method provides convenience in examining individual specimens during the preparation cycle, without the problem of regaining planarity for all specimens in the holder on the next step. Also, if the etch results are deemed inadequate, the specimen can be replaced in the holder to repeat the last step, as planarity is achieved individually rather than collectively. The drawback to this method is that slight rocking of the specimen may occur, especially if the specimen height is too great, which degrades edge retention and flatness.

Polishing Cloths

The requirements of a good polishing cloth include the ability to hold the abrasive media, long life, absence of any foreign material that may cause scratches, and absence of any processing chemical (such as dye or sizing) that may react with the specimen. Many cloths of

different fabrics, weaves, or naps are available for specimen polishing. Napless or low nap cloths are recommended for coarse polishing with diamond abrasive compounds. Napless, low, medium, and occasionally high nap cloths are used for final polishing. This step should be brief to minimize relief. Table 3.2 lists current polishing cloths, their characteristics and applications.

Polishing Abrasives

Polishing usually involves the use of one or more of the following abrasives: diamond, aluminum oxide (Al_2O_3), and amorphous silicon dioxide (SiO_2) in collidal suspension. For certain materials, cerium oxide, chromium oxide, magnesium oxide or iron
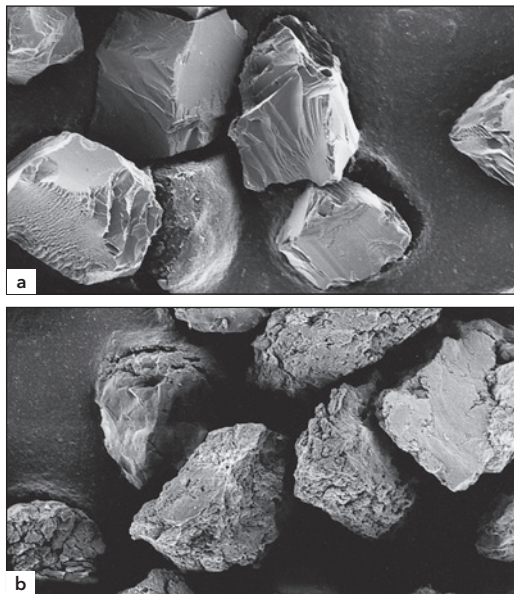
Figure 3.13. Comparison of monocrystalline a) and polycrystalline b) synthetic diamond grain shapes (SEM, 450X).

oxide may be used, although these the late 1920s, as Hoyt [7] mentions a visit to the Carboloy plant in West Lynn, Massachusetts, where he saw sapphire bearings being polished with diamond dust in an oil carrier. He used some of this material to prepare sintered carbides and published this work in 1930. Diamond abrasives were first introduced in a carrier paste but later aerosol and slurry forms were introduced. Virgin natural diamond was used initially, and is still available as MetaDi diamond paste. Later, synthetic diamond was introduced, first of the monocrystalline form, similar in morphology to natural diamond, and then in polycrystalline form. MetaDi II diamond pastes and MetaDi suspensions use synthetic monocrystalline diamond while MetaDi Supreme suspensions and MetaDi

Ultra pastes use synthetic polycrystalline diamonds. Figure 3.13 shows the shape differences between monocrystalline and polycrystalline diamonds. Studies have shown that cutting rates are higher for many materials using polycrystalline diamond compared to monocrystalline diamond.

Colloidal silica was first used for polishing wafers of single crystal silicon where all of the damage on the wafer surface must be eliminated before a device can be grown on it. The silica is amorphous and the solution has a basic pH of about ~10. The silica particles are actually nearly spherical in shape,
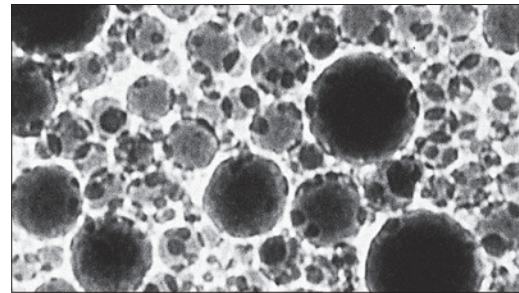
Figure 3.14. Amorphous silica particles in colloidal silica (TEM, 300,000X).

Figure 3.14, the polishing action is slow, and is due to both chemical and mechanical action. Damage-free surfaces can be produced more easily when using colloidal silica than with other abrasives during final polishing. Etchants can respond differently to surfaces polished with colloidal silica. For example, an etchant that produces a grain contrast etch when polished with alumina may instead reveal the grain and twin boundaries with a "flat" etch when polished with colloidal silica. Color etchants frequently respond better when colloidal silica is used producing a more pleasing range of colors and a crisper image. But, cleaning of the specimen is more difficult. For manual work, use a tuft of cotton soaked in a detergent solution. For automated systems, stop adding suspension about 10-15 seconds before the cycle ends and. For the last 10 seconds, flush the cloth surface with running water. Then, cleaning is simpler. Amorphous silica will crystallize if allowed to evaporate. Crystalline silica will scratch specimens, so this must be avoided. When opening a bottle, clean off any crystallized particles than may have formed around the opening. The safest approach is to filter the suspension before use. Additives are used to minimize crystallization, as in MasterMet 2 Colloidal Silica, greatly retarding crystallization.

GRINDING & POLISHING

For routine examinations, a fine diamond abrasive, such as 1µm, may be adequate as the last preparation step. Traditionally, aqueous fine alumina powders and suspensions, such as the MicroPolish II deagglomerated alumina powders and suspensions, have been used for final polishing with medium nap cloths. Alpha alumina (0.3µm size) and gamma alumina (0.05µm size) slurries (or suspensions) are popular for final polishing, either in sequence or singularly. MasterPrep alumina suspension utilizes alumina made by the sol-gel process, and it produces better surface finishes than alumina abrasives made by the traditional calcination process. Calcined alumina abrasives always exhibit some degree of agglomeration, regardless of the efforts to deagglomerate them, while sol-gel alumina is free of this problem. MasterMet colloidal silica suspensions (~10pH) are newer final polishing abrasives that produce a combination of mechanical and chemical action which is particularly beneficial for difficult to prepare materials. Vibratory polishers, Figure 3.15, are often used for final polishing, particularly with more difficult to prepare materials, for image analysis studies, or for publication of

Table 3.2: Polishing Cloth Selection Guide

	Cloth	Recommended Micron Size	Abrasive Type	Cloth Characteristics	Usage Guide	Applications
COARSE	UltraPad	6µm & Up	Diamond	Hard woven, no nap with high material removal	Used to replace multiple SiC grinding steps	Ferrous materials and thermal spray coatings
	UltraPol*	6µm & Up	Diamond	Hard woven, nonaggressive silk cloth	Excellent surface finished used to retain flatness in medium to hard specimens	Minerals, coals, ceramics, inclusion retention in steels, and refractory metals
	TexMet P	6µm & Up	Diamond	Hardest perforated woven cloth for hign material removal	Used for material removal and flatness of hard specimens	Ceramics, carbides, petrographic, hard metals, glass, and metal matrix composites
INTERMEDIATE	Nylon*	6µm & Up	Diamond	Oil resistant, medium hard woven, no nap	Used to retain flatness and hard phases	Ferrous materials, sintered carbides and cast irons
	TexMet C	15-0.02µm	Diamond Al_2O_3, SiO_2	Non-woven pressed cloth used for durability	Used for harder specimens and increased flatness	Ferrous and nonferrous metals, ceramics, electronic packages, PCB's, thermal spray coatings, cast irons, cermets, minerals, composites, plastics
	TriDent*	15-0.02µm	Diamond Al_2O_3	Soft durable, woven synthetic, no nap	Used to maximize flatness and retain phases while provideing excellent surface finish	Ferrous and nonferrous metals, microelectronics, coatings
	VerduTex*	9-1µm	Diamond	Durable, medium hard synthetic silk cloth	Used for rough and final polishing	Ferrous and nonferrous metals, microelectronics, coatings
	VelTex	9-1µm	Diamond	Shot-napped, synthetic velvet cloth	Ideal for final polishing	Steel, nonferrous and soft metals
	WhiteFelt	6-0.02µm	Diamond Al_2O_3, SiO_2	Soft and durable matted wool cloth	General usage for intermediate to fine steps	Ferrous and nonferrous metals
	PoliCloth	6-1µm	Diamond	Medium hard, woven wool cloth	General usage for intermediate steps	Cast iron, copper and aluminum alloys, coal and other soft metals
FINE	MicroCloth	5-0.02µm	Diamond Al_2O_3, SiO_2	Soft, versatile, long napped synthetic rayon cloth	Most popular, general usage final polishing cloth	Ferrous and nonferrous metals, ceramics, composites, PCBs, cast irons, cermets, plastics, electronics
	MicroFloc	3µm-0.02µm	Diamond Al_2O_3, SiO_2	Soft, long napped cloth	General usage for final polishing	Ferrous and nonferrous materials
	MasterTex	1-0.05µm	Al_2O_3, SiO_2	Soft synthetic velvet with low nap	Softer final polishing cloth	Soft nonferrous and microelectronic packages
	ChemoMet	1-0.02µm	Al_2O_3, SiO_2	Soft, porous, chemically resistant, synthetic cloth	General usage pad that removes smear metal from tough materials during chemomechanical polishing	Titanium, stainless steel, lead/tin, solders, electronic packages, soft nonferrous metals, plastics

Interchangable in many of the following methods

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quality work.

Vibratory Polishing

Similarly to electro-polishing, vibratory polishing provides excellent surface finish without deformation and with minimal difficulty. The advantage of vibratory polishing over electro-polishing, however, is that it requires no hazardous chemicals and is suited to any material or mix of materials. The VibroMet 2 polisher generates a high frequency, variable amplitude vibrational motion, which is almost 100% horizontal without vertical movement. The sample
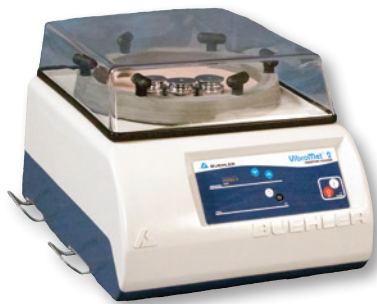
Figure 3.15. VibroMet 2 Vibratory Polisher

is polished stress-free. After a few minutes to a few hours with no further intervention, samples are polished with excellent surface finish and sharply defined edges.

Polishing is typically carried out with fine diamond slurries or suspensions of oxides such as MasterMet, MasterPrep or MicroPolish. Vibratory polishing is particularly suitable for the preparation of sensitive or ductile materials. Samples polished using this technique are particularly suitable for high precision analysis techniques such as electron back-scatter detection (EBSD) or nano-indentation hardness testing. Such techniques are highly sensitive to small amounts of surface deformation, which often cannot be directly observed using standard preparation techniques. The Table 3.3 shows the improvement of diffraction band contrast due to a Vibratory polishing step. Figure 3.16 shows a vibratory polished ferritic steel.

Preparation Methodology

Before vibratory polishing, the samples must be properly prepared using the methods described

in this book including final polishing. High quality sample preparation is a basic starting point for this technique. As already explained above, vibratory polishing is dedicated to fine diamond or oxide suspensions typically between 1 and 0.02 microns.
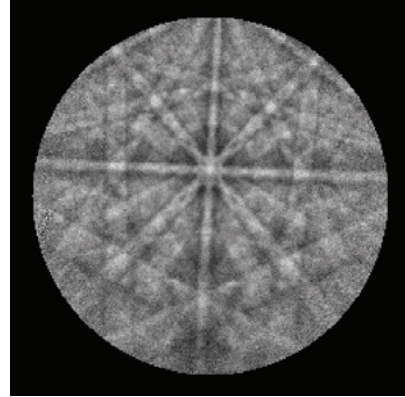
Figure 3.16. Kikuchi lines obtained by EBSD analysis
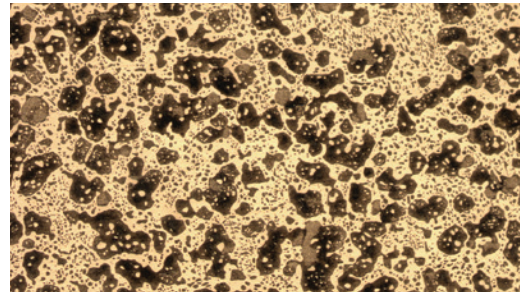
Figure 3.17. Lead alloy prepared by vibratory polishing.

Figure 3.18. Vibratory polished ferritic steel, Electron image overlayed with orientation map normal direction Phase: α - Iron.

Table 3.3: Band Contrast (0 - 255)		
Material	**Standard Polishing**	**Vibratory Polishing**
Mg	161.2	172.25 (+8.7%)
Si-Monocristal	205.75	233.0 (+13.2%)
Ti	134.0	146.6 (+9.1%)
Ni	85.0	102.8 (+20.9%)
Nb	145.6	151.2 (+3.8%)
Pb	No Pattern	108.0

As a recommendation, use the same polishing sus-

Figure 3.19. Sample preparation with their own weight.
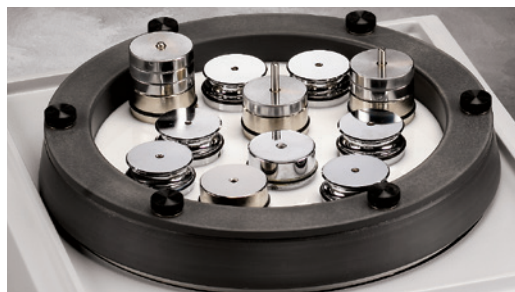
Figure 3.20. Preparation with sample holder and adding weight.

pension as in the final stage of the standard

mechanical preparation method. If the process uses a chemical additive such as hydrogen peroxide or other etchants, the addition will be optional on the VibroMet 2. Vibratory polishing is typically carried out without any additional chemical additives. The suspension should be sufficiently dispersed on the polishing cloth, and when using oxide suspensions particular care should be taken to ensure that the polishing cloth remains well moistened throughout the polishing cycle. If this is not done, then some polishing suspensions can crystallize. A popular compound for vibratory polishing is MasterMet 2 – a 0.02μm colloidal silica suspension that contains an additive to prevent crystallization.

The polishing cloth should be a flocked or napped cloth (such as MicroFloc, MicroCloth, MasterTex or VelTex), as the fibres in such cloths ease sample movement on the platen and help ensure a uniform polishing result.

Sample pressure is applied by the weight of the sample itself or by the addition of extra weights. The samples must be sufficiently rigid and typically should have a minimum total mass of 4 N [0.4 kg]. If the weight is too low, the samples could corrode or tarnish during the polishing.

There is no fixed rule for vibratory polishing time,

as this depends on many factors. One of the main factors is the amplitude strength, which is adjustable on the unit. This value changes the strength or intensity of the vibrations, which has a direct impact on the polishing time. In the case of new or unknown materials, the times can be determined empirically. Typically, if the polishing conditions are correct, a significant improvement can be seen in less than 30 minutes of vibratory polishing. The samples can then be examined under a light microscope or with a scanning electron microscope. If the polishing quality is not sufficiently improved, you can repeat the polishing cycle, or amend the polishing conditions.

Electrolytic Polishing and Etching

Electrolytic polishing or etching is achieved by the completion of an electrical circuit through an electrolyte. The specimen is set as the anode in the circuit. The application of current drives an oxidizing chemical reaction at the anode to achieve dissolution of the metal at the surface of the specimen where it is in contact with the electrolyte.

The conditions under which such reactions occur can significantly affect the outcome of the reaction. These include:

- Current density (related to the area to be polished)

- Voltage

- Time

- Electrolyte composition, viscosity and temperature

- Electrolyte movement ("refreshing" the electrolyte in the area of the reaction)

Figure 3.21 is a schematic diagram showing the relationship between current density and voltage in such a polishing cell. At low voltages, rapid dissolution of the metal will result in etching, as different areas of the material are removed at different rates.

If the voltage is increased, passivation processes change the nature of the reaction and a polishing effect occurs. In this case, peaks on the surface dissolve preferentially, microscopically smoothing the surface. As this is purely a chemical effect, there is no associated mechanical damage from this process. As such, electrolytically polished surfaces are

particularly useful for surface sensitive tests where even small amounts of surface deformation are unacceptable.

Figure 3.22. Electro polished ferritic steel examined with EBSD [Electron image overlayed with orientation map normal direction Phase: α - Iron

Figure 3.23. Aluminum as cast condition with colored dendrites revealed by anodizing with Barker's reagent 40V for 460 seconds (polarized light, 25X)
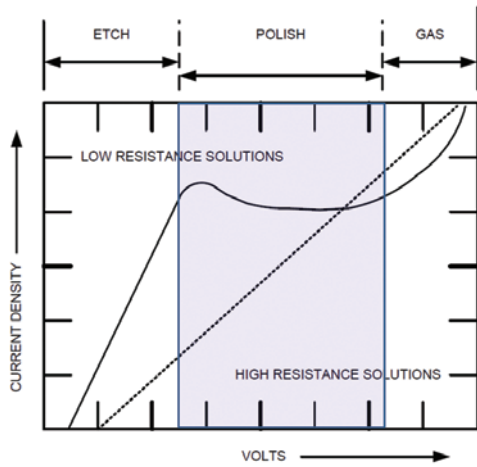
Figure 3.21. Influence of current density and voltage on the sample. The optimum area for polishing is shaded. At low voltages, the sample is etched. Excessive voltage creates voids in the electrolyte and a pitting effect will occur due to the irregular density.

If the voltage is increased further, the passivation layer breaks down and oxygen is evolved, resulting in pitting at the surface of the specimen.

Electrolytic polishing and etching processes can be quick and effective, and highly reproducible when correctly performed. Historically, most applications for electrolytic polishing in metallography have been in process inspection of materials that are difficult to polish and etch with chemical etchants, such as superalloys and stainless steel – although soft materials are also ideal candidates, as mechanical polishing to a high standard can be difficult. The process has seen particular growth in metallography during recent years due to the increasing requirements for crystallographic analyses such as EBSD and surface characterization tests such as nano-indentation hardness testing, both of which require a deformation free surface to attain the best results.

Optimizing the reproducibility of electrolytic polishing and etching processes requires good control of the conditions. This is most readily achieved by using dedicated equipment, consisting of a good quality power supply unit and a polishing or etching cell that effectively controls the movement of the electrolyte as well as the area of the specimen. Figure 3.24 shows a such cell. It is recommended to use equipment that has integrated temperature control and cooling of the electrolyte, as heat can
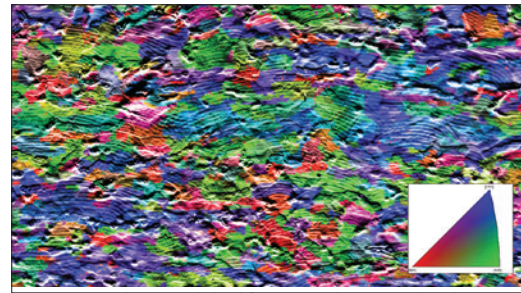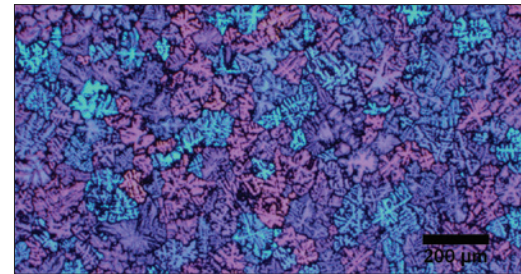
be generated during the process.

One of the common chemicals used in electrolytic polishing is perchloric acid, which can become unstable at higher temperatures (>100°F [38°C]) or higher concentrations or if higher concentrations occur due to excessive evaporation. Avoid the use
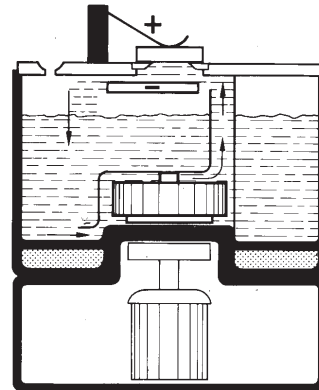
Figure 3.24. The electrolyte is conveyed by a circulation pump between the cathode (-) and the sample (+). By the uniform flow of the electrolyte the material debris will flushed away and the sample is uniform wetted. An integrated cooling ensures that the electrolyte does not get too warm.

of perchloric acid in contact with organic materials, such as mounting media, as unstable perchlorates can form and accumulate in the electrolyte.

GRINDING & POLISHING

Typical sequence for electropolishing of ferritic material:

- Step1: Grinding with 400 grit [P800] and 600 grit [P1200] SiC-Paper on grinding machine (each 60s.)

- Step 2: Electrolytic polishing on electro polishing system ElectroMet 4 (Figure 3.25), at 30Vdc for 60 seconds

Sometimes, electro polished surfaces are wavy and this will cause a problem at higher magnifications. Electropolishing will tend to round edges or pores and wash out non-metallic inclusions. Two or more phase alloys are more difficult to polish, because the phases will preferentially attacked. Consequently,

Figure 3.25. ElectroMet 4 Electrolytic Etcher/Polisher

electrolytic polishing is not recommended for failure analysis, but can be used for a few seconds as a final polishing step to remove the last existing deformation.

Anodizing

The process of anodizing is related to electrolytic polishing and etching, in that the specimen is set as the anode within the electrical circuit. However, in this case the purpose is to grow an oxide layer on the surface. The oxide layer, when viewed under polarized light, will cause interference effects that can introduce contrast and colour between phases or crystallographic orientations. This is a particularly common process for aluminium alloys, but has also been used on other materials such as titanium and

EXAMPLES OF PREPARATION PROCEDURES

The "Traditional" Method

Over the past forty years, a general procedure has been developed that is quite successful for preparing most metals and alloys. This method is based on grinding with silicon carbide waterproof papers through a series of decreasing abrasive sizes, then rough polishing with one or more diamond abrasive sizes, followed by fine polishing with one or more alumina suspensions of different particle size. This procedure will be called the "traditional" method, and is described in Table 4.1.

This procedure is used for manual or automated preparation, although manual control of the force applied to a specimen would not be as consistent. Complementary motion means that the specimen holder is rotated in the same direction as the platen, and does not apply to manual preparation. Some machines can be set so that the specimen holder rotates in the direction opposite to that of the platen, called "contra." This provides a more aggressive action but was not part of the "traditional" approach when automated. The traditional method is not fixed, as it can be modified by substitution or the ommission of one or more of the polishing steps.

Times and pressures could be varied, as well, to suit the needs of the work, or the material being prepared. This is the "art" of materials preparation.

Contemporary Methods

New concepts and preparation materials have been introduced that enable technicians to shorten the process while producing better, more consistent results. Much of this effort has centered upon reducing or eliminating the use of silicon carbide paper in the grinding steps. In all cases, an initial grinding step must be used, but there are a wide range of materials that can be chosen instead of SiC paper. There is nothing wrong with the use of SiC for the first step, except that it has a short life. If an automated device is used that holds a number of specimens rigidly (central force), then the first step must remove the sectioning damage on each specimen and bring all of the specimens in the holder to a common plane. The first step is often called "planar grinding". SiC paper is most often used for this step although more than one sheet may be needed according to the surface area to be planed down and how much material needs to be ground back. If it is such that more than a few

Table 4.1: The Traditional Procedure for Preparing Most Metals and Alloys					
Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	120 [P120] grit SiC water cooled	6 [27]	240-300	⬤ ✦	Until Plane
CarbiMet	220 [P240] or 240 [P280] grit SiC water cooled	6 [27]	240-300	⬤ ✦	1:00
CarbiMet	320 [P400] grit SiC water cooled	6 [27]	240-300	⬤ ✦	1:00
CarbiMet	400 [P600] grit SiC water cooled	6 [27]	240-300	⬤ ✦	1:00
CarbiMet	600 [P1200] grit SiC water cooled	6 [27]	240-300	⬤ ✦	1:00
Canvas Cloth	6μm MetaDi Diamond Paste with MetaDi Fluid Extender	6 [27]	120-150	⬤ ✦	2:00
Billiard or Felt cloths	1μm MetaDi Diamond Paste with MetaDi Fluid Extender	6 [27]	120-150	⬤ ✦	2:00
MicroCloth	Aqueous 0.3μm α – alumina MicroPolish slurry	6 [27]	120-150	⬤ ✦	2:00
MicroCloth	Aqueous 0.05μm γ – alumina MicroPolish slurry	6 [27]	120-150	⬤ ✦	2:00

⬤ = Platen ✦ = Specimen Holder

sheets of SiC paper are needed, then the use of a more aggressive and more durable surface such as ZirMet paper or diamond grinding discs (Apex or UltraPrep Diamond Discs) may be used. With this huge array of products to choose from, how can the lab technician decide what to use? Each of these products has advantages and disadvantages, and this is only the first step.

One or more steps using diamond abrasives on napless surfaces usually follow planar grinding. PSA-backed silk, nylon or polyester cloths are widely used. These give good cutting rates, maintain flatness and minimize relief. Silk cloths, such as the UltraPol cloth, provide the best flatness and excellent surface finishes relative to the diamond size used. UltraPad cloth, a thicker, hard, woven cloth, is more aggressive, gives nearly as good a surface finish, similar excellent flatness, and longer life than an UltraPol cloth. Synthetic chemotextile surfaces, such as TexMet C, give excellent flatness and are more aggressive than silk. They are excellent for retaining second phase particles and inclusions. Diamond suspensions are most popular with automated polishers as they can be added easily during polishing; although it is still best to charge the cloth initially with diamond paste of the same size to get polishing started quickly. Final polishing could be performed with a very fine diamond size, such as 0.1µm diamond, depending upon the material, your needs and personal preferences. Otherwise, final polishing is performed with MasterMet colloidal silica or with MicroPolish or MasterPrep alumina suspensions using napless, or low to medium nap cloths. For some materials, such as titanium and zirconium alloys, an attack polishing solution is added to the slurry to enhance deformation and scratch removal and improve polarized light response.

Contra rotation, where the polishing head moves in the direction opposite to the platen, is preferred as the abrasive suspension stays on the cloth better, although this will not work if the head rotates at a high rpm. Examples of generic contemporary preparation practices follow in Tables 4.2 – 4.4. Specific procedures for a wide range of materials are given in the next section.

The starting SiC abrasive size is chosen based upon the degree of surface roughness and depth of cutting damage and the hardness of the material. Never start with a coarser abrasive than necessary to remove the cutting damage and achieve planar conditions in a reasonable time.

A similar scheme can be developed using rigid grinding discs, such as the Apex Hercules H Disc. These discs are generally restricted to materials above a certain hardness, such as 175 HV, although some softer materials can be prepared using them. This disc can also be used for the planar grinding step. An example of such a practice, applicable to nearly all steels (results are marginal for solution annealed austenitic stainless steels) is given in Table 4.3.

The planar grinding step could also be performed using a 45µm metal bonded, or a 30µm resin bonded, UltraPrep disc or with the Apex Hercules H rigid grinding disc and 15 or 30µm diamond, depending upon the material. Rigid grinding discs contain no abrasive; they must be charged during use and suspensions are the easiest way to do this. Polycrystalline diamond suspensions are favored over monocrystalline synthetic diamond suspensions for most metals and alloys due to their higher cutting rate.

Table 4.2: Generic Contemporary Preparation Procedure for Many Metals and Alloys

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	120 [P120] to 240 [P280] grit SiC water cooled	6 [27]	300		Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	6 [27]	150		5:00
TriDent	3µm MetaDi Supreme Diamond*	6 [27]	150		4:00
ChemoMet	0.02 - 0.06µm MasterMet Colloidal Silica	6 [27]	150		2:00

● = Platen   ⚙ = Specimen Holder   *MetaDi Fluid Extender as desired

EXAMPLES OF PREPARATION PROCEDURES

The Apex Hercules S rigid grinding disc, designed for soft metals and alloys, is used in a similar manner. This disc is quite versatile and can be used to prepare harder materials as well, although its wear rate will be greater than the H disc when used to prepare very hard materials. A generic four step practice is given below for soft metals and alloys.

The planar grinding step can be performed with the 30μm resin bonded diamond disc, or with a second Apex Hercules S disc and 15 or 30μm diamond, depending upon the metal or alloy. For some very difficult metals and alloys, a 1μm diamond step on a TriDent cloth (similar to step 3, but for 3 minutes) could be added, and/or a brief vibratory polish (use the same cloths and abrasives as for step 4) may be needed to produce perfect publication quality images. Four steps may suffice for routine work.

Table 4.3: 4-Step Contemporary Procedure for Steels Using a Rigid Grinding Disc					
Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	120 [P120] to 320 [P400] grit SiC water cooled	6 [27]	300		Until Plane
Apex Hercules H	9μm MetaDi Supreme Diamond*	6 [27]	150		5:00
TriDent	3μm MetaDi Supreme Diamond*	6 [27]	150		4:00
ChemoMet	0.05μm MasterPrep Alumina	6 [27]	150		2:00

● = Platen   ✦ = Specimen Holder   *MetaDi Fluid Extender as desired

Table 4.4: 4-Step Contemporary Procedure for Nonferrous Metals Using a Rigid Grinding Disc					
Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	220 [P280] to 320 [P400] grit SiC water cooled	5 [22]	300		Until Plane
Apex Hercules S	6μm MetaDi Supreme Diamond*	5 [22]	150		5:00
TriDent	3μm MetaDi Supreme Diamond*	5 [22]	150		4:00
ChemoMet	0.02 - 0.06μm MasterMet Colloidal Silica	5 [22]	150		2:00

● = Platen   ✦ = Specimen Holder   *MetaDi Fluid Extender as desired

Following are our recommendations for preparing a wide range of materials arranged, in the case of metals, according to common characteristics, basically in accordance with their classification in the periodic table of the elements. This break down is further modified, especially in the case of iron based alloys, as required due to the wide range of properties that can be experienced.

The elements shown in Figure 5.1 categorizes metallic, nonmetallic and metalloid elements according to similarities in atomic structure and is a good starting point, aided by similarities in physical properties and behavior, for grouping elements and their alloys that have reasonably similar preparation procedures.

Of course, technicians prepare materials other than metals. These have been grouped and sub-divided according to the nature of these materials, as follows:

- Sintered Carbides
- Ceramics
- Composites: Metal Matrix, Polymer Matrix and Ceramic Matrix
- Printed Wiring Boards
- Electronic Materials
- Plastics and Polymers

The procedures discussed in this book were developed and tested extensively using machines with an 8in [203mm] diameter platen with six 1.25in [32mm] diameter specimens in a 5in [127mm] diameter holder. These procedures produced the same results when the specimens were prepared using a 10in [254mm] or a 12in [305mm] diameter platen. When using the 8in [203mm] platen and a 5in [127mm] diameter specimen holder, the machine's head position was adjusted so that the specimens rotate out over the outer periphery of the surface of the platen. This procedure makes maximum use of the working surface and improves edge retention. This alignment practice is less critical with the larger diameter platens when using the same diameter holder. However, if the 7in [178mm] diameter specimen holder is used with the 12in [305mm] diameter platen, the head position should be adjusted so that the specimens rotate out over the platen's edge.

In general, it is difficult to make accurate predictions about variations in time and load when using different format platens and mount sizes. Different mount sizes per se should not be used as the basis for calculations as the area of the specimens within the mount is more important than the mount area itself. For example, the same specimen cross sectional area could be present in 1, 1.25 or 1.5in [25, 32 or 38mm] diameter polymer mounts. Further, the number of specimens in the holder can vary substantially. It is necessary to put at least three

mounts in a central-force holder to obtain a proper weight balance, but, two specimens could be dummy mounts (mounts without specimens). In general, as the area of the specimen in the mount increases, as the diameter and the number of specimens increases, the time and pressure must increase to obtain the same degree of preparation perfection.

If the area of the mounts is used, rather than the area of the embedded specimens, and we assume that we are preparing six 1in [25mm], six 1.25in [32mm] or three 1.5in [38mm] diameter specimens, then we can adjust the pressures relative to the standard values in the tables (for six 1.25in [32mm] diameter specimens) using an 8in [203mm] diameter platen format machine. A comparison of the cross-sectional areas would suggest that for the six 1in [25mm] specimens, the pressures could be reduced to 65% of the standard values while for three 1.5in [38mm] diameter specimens, the pressures and times could be reduced to about 70% of the standard values.

Changing the platen diameter increases the distance that the specimens travel in a given time. For example, for the same rpm values, specimens travel 1.25 and 1.5 times as far using a 10 or 12in [254 or 305mm] diameter platen, respectively, compared to an 8in [203mm] platen. This suggests that the times could be reduced to 80 or 67% of that used for the 8in [203mm] diameter platen when using 10 or 12in [254 or 305mm] diameter platens. While preparation times may be shorter using a larger format machine, consumable cost is greater and larger machines are needed only when the specimen throughput needs are very high or with large specimens.

PROCEDURES FOR SPECIFIC MATERIALS

Light Metals

13 **Al** Aluminium	12 **Mg** Magnesium	4 **Be** Beryllium

Low Melting Point Alloys

30 **Zn** Zinc	48 **Cd** Cadmium	50 **Sn** Tin	51 **Sb** Antimony	82 **Pb** Lead	83 **Bi** Bismuth

Refractory Metals

22 **Ti** Titanium	40 **Zr** Zirconium	72 **Hf** Hafnium	23 **V** Vanadium	24 **Cr** Chromium	41 **Nb** Niobium	42 **Mo** Molybdenum	73 **Ta** Tantalum
74 **W** Tungsten	75 **Re** Rhenium						

Ferrous Metals

26 **Fe** Iron

Copper, Nickel, and Cobalt

29 **Cu** Copper	28 **Ni** Nickel	27 **Co** Cobalt

Precious Metals

44 **Ru** Ruthenium	45 **Rh** Rhodium	46 **Pd** Palladium	47 **Ag** Silver	76 **Os** Osmium	77 **Ir** Iridium	78 **Pt** Platinum	79 **Au** Gold

Other Material Groups

TSC Thermal Spray Coatings	**SC** Sintered Carbides	**C** Ceramics	**M** Minerals	**CM** Composites	**EM** Electronic Materials	**PWB** Printed Wiring Boards	**P** Polymers

Figure 5.1. Providing a recommendation for preparing a specific material is fairly straight-forward for the grinding-polishing procedure. However, there are many more variables to be considered when making recommendations for the other 4 segments of preparation. In the following chapters, you will find additional information on each material for sectioning, mounting, imaging & analysis and hardness testing. The recommendations are based on best practices for the most typical situation.

LIGHT METALS

LIGHT METALS: Al, Mg and Be

13
Al
Aluminium

Aluminum

Aluminum is a soft, ductile metal. Deformation induced damage is a common preparation problem in the purer compositions. After preparation, the surface will form a tight protective oxide layer that makes etching difficult. Commercial grades contain many discrete intermetallic particles with a variety of compositions. These intermetallic particles are usually attacked by etchants before the matrix. Although the response to specific etchants has been used for many years to identify these phases, this procedure requires careful control. Today, energy-dispersive analysis is commonly performed for phase identification due to its greater reliability.

Five and four-step practices for aluminum alloys are presented in Table 6.1 and 6.2 with expected results as in Figure 6.1. While MgO was the preferred final polishing abrasive for aluminum and its alloys, it is a difficult abrasive to use and is not available in very fine sizes. Colloidal silica has replaced magnesia as the preferred abrasive for the final step and is finer in size. For color etching work, and for the most difficult grades of aluminum, a brief vibratory polish

may be needed to completely remove any trace of damage or scratches. The five-step practice is recommended for super pure (SP) and commercially pure (CP) aluminum and for wrought alloys that are difficult to prepare.

Either 240 [P280] or 320 [P400] grit SiC waterproof paper may also be used for the planar grinding step. An UltraPol cloth produces better surface finishes than an UltraPad cloth, but the UltraPad cloth has a longer useful life. A ChemoMet cloth is recommended when edge retention is critical. Pure aluminum and some alloys are susceptible to embedment of fine diamond abrasive particles, especially when suspensions are used. If this occurs, switch to diamond in paste form, which is much less likely to cause embedding. SP and CP aluminum can be given a brief vibratory polish (same products as last step) to improve scratch control, although
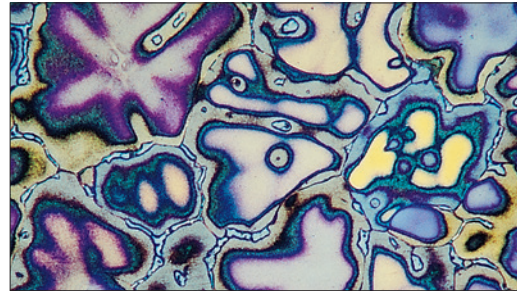
Figure 6.1. Dendritic segregation in as cast 206 aluminum revealed by color etching with Weck's reagent (polarized light, 200X).

Table 6.1: 5-Step Method for Soft Aluminum Alloys

Sectioning	Abrasive Cutter with a wheel recommended for use on non-ferrous materials				
Mounting	Compression or Castable, typically with PhenoCure, EpoxiCure, EpoThin or SamplKwick				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled	5 [22]	300	●◉	Until Plane
TexMet C	9µm MetaDi Supreme Diamond*	5 [22]	150	●◉	5:00
TexMet C	3µm MetaDi Paste*	5 [22]	150	●◉	4:00
TexMet C	1µm MetaDi Paste*	5 [22]	150	●◉	2:00
ChemoMet	0.06µm MasterMet Colloidal Silica	5 [22]	150	●◉	1:30

● = Platen ◉ = Specimen Holder *MetaDi Fluid Extender as desired

Imaging & Analysis	Dendritic spacing, Porosity Assessment, Grain Size (depending on the type of aluminum and its processing)
Hardness Testing	Vickers, Knoop

this is generally not required. MasterPrep alumina suspension has been found to be highly effective as a final polishing abrasive for aluminum alloys, however, the standard alumina abrasives made by the calcination process are unsuitable for aluminum.

For many aluminum alloys, excellent results can be obtained using a four step procedure, such as shown below in Table 6.2 with expected results as shown in Figure 6.2. This procedure retains all of the intermetallic precipitates observed in aluminum and its alloys and minimizes relief. Synthetic napless cloths may be used for the final step with colloidal silica and they will introduce less relief than a low or medium nap cloth, but may not remove fine polishing scratches as well. For very pure aluminum alloys, this procedure could be followed by vibratory polishing to improve the surface finish, as these are quite difficult to prepare totally free of fine polishing scratches.
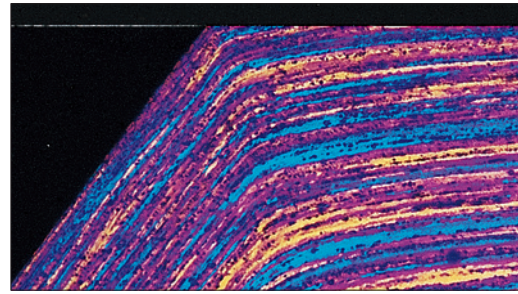
Figure 6.2. Deformed, elongated grain structure of extruded 6061-F aluminum after shearing revealed by anodizing with Barker's reagent (polarized light, 100X).

HELPFUL HINTS FOR ALUMINUM

Many low-alloy aluminum specimens are difficult to polish to a perfect finish using standard methods. A vibratory polisher can be used quite effectively with MasterMet colloidal silica to produce deformation-free, scratch-free surfaces for anodizing or color etching and examination with polarized light or Nomarski DIC.

LIGHT METALS

Table 6.2: 4-Step Method for Aluminum Alloys

Sectioning	Abrasive Cutter with a wheel recommended for use on non-ferrous materials				
Mounting	Compression or Castable, typically with PhenoCure, EpoxiCure, EpoThin or SamplKwick				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled	5 [22]	300	⬤ ⬤	Until Plane
UltraPad	9μm MetaDi Supreme Diamond*	5 [22]	150	⬤ ⬤	5:00
TriDent	3μm MetaDi Supreme Diamond*	5 [22]	150	⬤ ⬤	4:00
ChemoMet	0.02 - 0.06μm Master-Met Colloidal Silica	5 [22]	150	⬤ ⬤	1:30

⬤ = Platen	⬤ = Specimen Holder	*MetaDi Fluid Extender as desired	
Imaging & Analysis	Dendritic spacing, Porosity Assessment, Grain Size (depending on the type of aluminum and its processing)		
Hardness Testing	Vickers, Knoop		

LIGHT METALS

Magnesium

Preparation of magnesium and its alloys is rather difficult due to the low matrix hardness and the higher hardness of precipitate phases that lead to relief problems, and from the reactivity of the metal. Mechanical twinning may result during cutting, grinding, or handling if pressures are excessive. Final polishing and cleaning operations should avoid or minimize the use of water and a variety of solutions have been proposed. Pure magnesium is attacked slowly by water while Mg alloys may exhibit much higher attack rates. Some authors state that water should not be used in any step and they use a 1 to 3 mixture of glycerol to ethanol as the coolant even in the grinding steps. Always grind with a

coolant, as fine Mg dust is a fire hazard. Because of the presence of hard intermetallic phases, relief may be difficult to control, especially if napped cloths are used. Following is a five-step procedure for magnesium and its alloys, see Table 6.3.

MasterPolish is nearly water-free and yields excellent results as the final abrasive (Figures 6.3 - 6.4). After the last step, wash the specimens with ethanol. Cleaning after the last step, without using water, is difficult. Holding the specimen under running water for about a second eased the cleaning problem and did not appear to harm the microstructure. Cosmetic cotton puffs can scratch the surface when swab etching. For best results, etch-polish-etch cycle may be needed. Magnesium has a hexagonal close-packed crystal structure and will respond to polarized light. To enhance the response, use a brief vibratory polish with the materials used in the last step.

Figure 6.3. Mechanical twinning in deformed high-purity magnesium (99.8% Mg) (acetic-picral etch, crossed polarized light plus sensitive tint, 50X).

HELPFUL HINTS FOR LIGHT METALS

For polishing, apply diamond paste and use lapping oil, or use oil-based MetaDi diamond suspension, available down to a 0.05μm size, with a medium nap cloth.

Table 6.3: 5-Step Method for Magnesium Alloys

Sectioning	Precision saw, with no water and a 15HC blade recommended for use onMetal Matrix Composites, PCBs, Bone, TI, TSC				
Mounting	Castable, typically with PhenoCure, EpoxiCure, EpoThin or SamplKwick				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC* water cooled	3 [13]	300	◐◉	Until Plane
TexMet C	9μm MetaDi Oil-based Diamond	3 [13]	150	◐◉	6:00
TexMet C	3μm MetaDi Oil-based Diamond	3 [13]	150	◐◉	5:00
TexMet C	1μm MetaDi Oil-based Diamond	3 [13]	150	◐◉	4:00
ChemoMet	0.05μm MasterPolish	4 [18]	150	◐◉	1:30

● = Platen   ◉ = Specimen Holder   *SiC surfaces were coated with wax to minimize embedment

Imaging & Analysis	Dendritic spacing, Porosity Assessment, Grain Size
Hardness Testing	Brinell

Figure 6.4. As-cast microstructure of a magnesium alloy with 2.5% of rare earth elements, 2.11% Zn and 0.64% Zr showing a film of the rare earth elements in the grain boundaries, alloy segregation within grains ("coring" revealed by color variations within grains) and a few mechanical twins in the grains (acetic-picral etch, crossed polarized light plus sensitive tint, 100X).

Beryllium

Beryllium is also a difficult metal to prepare and presents a health risk to the metallographer. Only those familiar with the toxicology of Be, and are properly equipped to deal with these issues, should work with the metal. The grinding dust is extremely toxic. Wet cutting prevents air contamination but the grit must be disposed of properly. As with Mg, Be is easily damaged in cutting and grinding forming mechanical twins. Light pressures are required. Although some authors claim that water cannot be used, even when grinding Be, others report no difficulties using water. Attack-polishing agents are frequently used when preparing Be and many are recommended [2]. Table 6.4 shows a four-step practice for beryllium with expected results as shown in Figure 6.5.

For the final step, mix hydrogen peroxide (30% concentration – avoid physical contact!) with the MasterMet colloidal silica in a ratio of one part hydrogen peroxide to five parts colloidal silica. Oxalic acid solutions (5% concentration) have also been used with alumina for attack polishing. For optimal polarized light response, follow this with vibratory polishing using a one-to-ten ratio of hydrogen peroxide to colloidal silica.
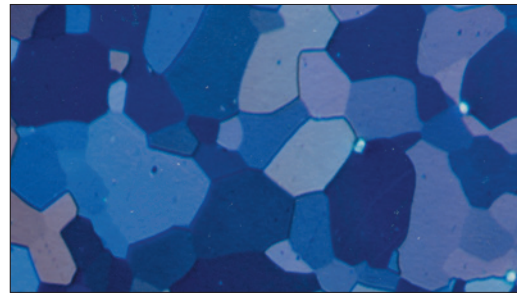
Figure 6.5. Grain structure of wrought, P/M beryllium (unetched, cross-polarized light, 100X).

HELPFUL HINTS FOR LIGHT METALS

Small diamond abrasive sizes are prone to embedment when applied as a suspension or by spraying. Charge the cloth using diamond paste to eliminate embedding. Another technique is to use both a fine diamond paste and MasterPrep alumina suspension – simultaneously – to prevent embedding.

Table 6.4: 4-Step Method for Beryllium

Sectioning	Precision saw, with no water and a 15HC blade recommended for use onMetal Matrix Composites, PCBs, Bone, TI, TSC
Mounting	Castable, typically with PhenoCure, EpoxiCure, EpoThin or SamplKwick

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled	4 [18]	300	⬤ ✦	Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	4 [18]	150	⬤ ✦	5:00
TriDent	3µm MetaDi Supreme Diamond*	4 [18]	150	⬤ ✦	4:00
ChemoMet	0.02 - 0.06µm Master-Met Colloidal Silica	3 [13]	150	⬤ ✦	2:00

⬤ = Platen   ✦ = Specimen Holder   *MetaDi Fluid Extender as desired

Imaging & Analysis	Dendritic spacing, Porosity Assessment, Grain Size
Hardness Testing	Brinell

LOW MELTING POINT METALS

LOW-MELTING POINT METALS: Sb, Bi, Cd, Pb, Sn and Zn

As pure metals, antimony, bismuth, cadmium, lead, tin, and zinc are all very soft and difficult to prepare. Pure antimony is quite brittle, but alloys containing Sb are more common, see Figure 7.1. Bismuth is a soft metal, but brittle, and not difficult to prepare. However, retaining bismuth particles in free machining steels is difficult. Cadmium and zinc, both with hexagonal close-packed crystal structures, are quite prone to mechanical twin formation if sectioning or grinding is performed too aggressively (Figure 7.2). Zinc is harder than tin or lead and tends to be brittle. Zinc is widely used to coat sheet steel (galvanized steel) for corrosion protection, and is a common metallographic subject. Pure zinc is very difficult to prepare. Lead is very soft and ductile and pure specimens are extremely difficult to prepare; however, lead alloys are considerably easier. Tin, which is allotropic with a body-centered tetragonal crystal structure at room temperature, is soft and malleable and less sensitive to twinning. Pure tin, like pure lead, is very difficult to prepare. Due to their low melting points, and low recrystallization temperatures,
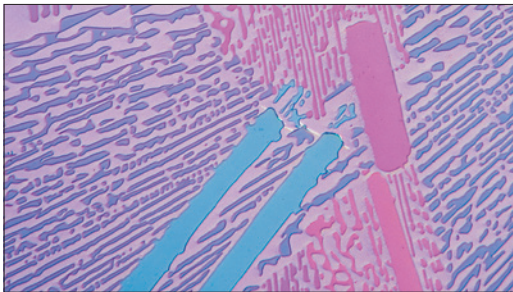
Figure 7.1. Proeutectic dendrites of Cu_2Sb surrounded by a eutectic mixture of antimony and Cu_2Sb, in an as-cast Sb – 30% Cu specimen (unetched, polarized light, 200X).

cold setting resins are usually recommended as recrystallization may occur during hot compression mounting. Some of these metals in the pure, or nearly pure form, will deform under the pressures used in compression mounting. Alloys of these metals are easier to prepare, as they are usually higher in hardness. Heating of surfaces during grinding must be minimized. Grinding of these metals is always difficult, as SiC particles tend to embed heavily.
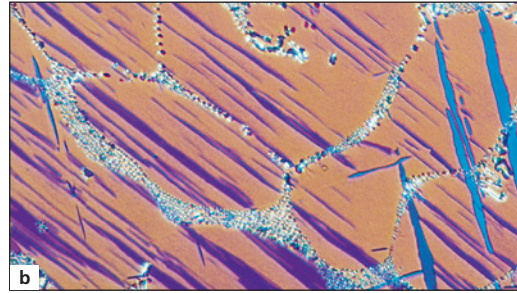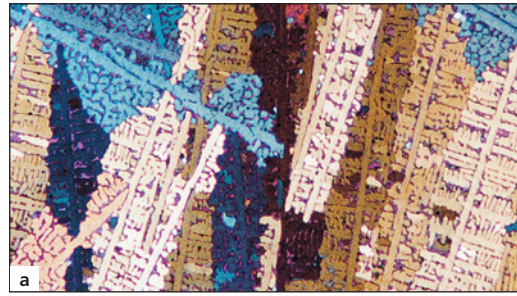
Figure 7.2. a) Cadmium dendrites and a Cd-Bi eutectic in an as-cast Cd – 20% Bi alloy (unetched, crossed polarized light plus sensitive tint, 50X). b) As-cast microstructure of Zn – 0.1% Ti – 0.2% Cu alloy exhibiting mechanical twins and a three-phase eutectic (alpha-Zn, Cu-Zn and $Zn_{15}Ti$) in a cellular pattern (Palmerton reagent, crossed polarized light plus sensitive tint, 200X).

Many authors have recommended coating the SiC paper surface with bees wax, but this does not solve the problem. Paraffin (candle wax) is much better for reducing embedding. Embedding is most common with the finer grit size papers. Diamond is not a very effective abrasive with these metals. Alumina works quite well. Following is a procedure for these alloys, see Table 7.1.

For best results, follow this with a vibratory polish using MasterMet colloidal silica on MicroCloth pad for times up to 1-2 hours. This will improve polarized light response for the hcp Cd and Zn and the rhombohedral Bi. The 1µm alumina step will remove any embedded silicon carbide particles much more effectively than a diamond abrasive step.

Table 7.1: 6-Step Method for Low-Melting Temperature Alloys

Sectioning	Precision saw, with a 30HC blade recommended for use on soft gummy materials
Mounting	Castable, typically with EpoThin

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled (wax coated)*	4 [18]	150		Until Plane
CarbiMet	400 [P800] grit SiC water cooled (wax coated)*	4 [18]	150		1:00
CarbiMet	600 [P1200] grit SiC water cooled (wax coated)*	4 [18]	150		1:00
CarbiMet	1200 [P2500] grit SiC water cooled (wax coated)*	4 [18]	150		1:00
MicroFloc	1μm MicroPolish II Alumina	5 [22]	150		5:00
MicroFloc	0.05μm MasterPrep Alumina**	4 [18]	150		4:00

● = Platen   ✸ = Specimen Holder

*Rub candle was lightly across rotating disc prior to grinding   **See vibratory polish recommendation in text

Imaging & Analysis	Grain Size, Measurement & Analysis applications
Hardness Testing	N/A

HELPFUL HINTS FOR LOW-MELTING POINT METALS

Embedment of fine abrasive particles is a common problem when preparing soft, low-melting point specimens. To reduce embedment of SiC abrasive particles, coat the paper with wax before grinding. Johnson [47] showed that candle wax is far more effective than paraffin. However, if the layer is applied too thickly, the metal-removal rate in grinding will be reduced drastically.

REFRACTORY METALS: Ti, Zr, Hf, Cr, Mo, Nb, Re, Ta, V and W

22 Ti Titanium

Titanium

Pure titanium is soft and ductile, but is very easily damaged by twinning in sectioning and grinding. Preparation of commercially pure titanium, which is a popular grade, is very difficult, while preparation of the alloys is somewhat easier. Some authors have stated that titanium alloys should not be mounted in phenolic resins as the alloys can absorb hydrogen from the resin. Further, it is possible that the heat from mounting could cause hydrides to go into solution. This is also possible with castable resins if the exothermic reaction of polymerization generates excessive heat. If the hydride phase content is a subject of interest, then the specimens must be mounted in a castable resin with a very low exotherm (long curing times favor lower heat generation, and vice versa). Titanium is very difficult to section and has low grinding and polishing rates. The following practice for titanium and its alloys demonstrates the use of an attack-polishing agent added to the final polishing abrasive to obtain the best results, especially for commercially pure titanium, a rather difficult metal to prepare free of deformation for color etching, heat tinting and/or polarized light examination of the grain structure. Attack polishing solutions added to the abrasive slurry or suspension must be treated with great care to avoid burns. Use good, safe laboratory practices and it is advisable to wear protective gloves. This three step practice could be modified to four steps by adding a 3 or 1µm diamond step, but this is usually unnecessary, see Table 7.1 and Figures 8.1 – 8.1 for expected results.

A number of attack polishing agents have been
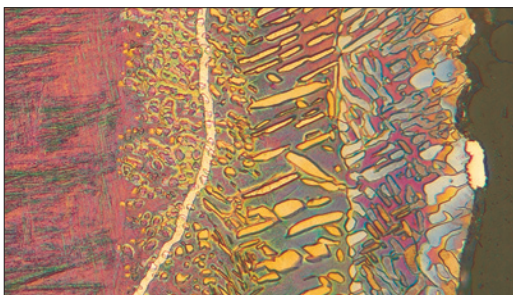
Figure 8.1. Alpha at the surface of heat treated (1038 °C, water quench) Ti – 3% Cr alloy after tint etching with Beraha's reagent (polarized light, 500X).
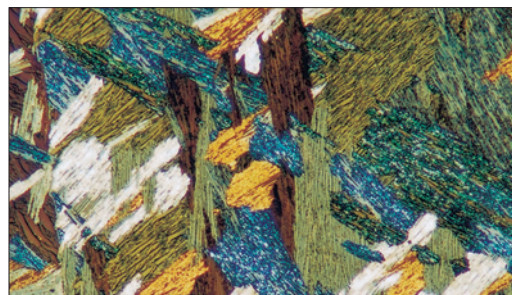
Figure 8.2. Basket-weave alpha-beta structure of as-cast Ti – 6% Al – 4% V revealed by heat tinting (polarized light, 100X).

Table 7.1: 3-Step Method for Ti Alloys					
Sectioning	Abrasive Cutter with a wheel recommended for use on ductile materials				
Mounting	Compression, typically with EpoMet				
Surface	**Abrasive / Size**	**Load - lbs [N] / Specimen**	**Base Speed [rpm]**	**Relative Rotation**	**Time [min:sec]**
CarbiMet	320 [P400] grit SiC water cooled	6 [27]	300	⊙⊙	Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	6 [27]	150	⊙⊙	10:00
ChemoMet	0.02 - 0.06µm Master-Met Colloidal Silica**	5 [22]	150	⊙⊙	10:00

● = Platen   ⊛ = Specimen Holder

* Plus MetaDi Fluid Extender as desired
** Attack polish may be used, 1 part Ammonium Persulfate solutions (10g Ammonium Persulfate per 100mℓ distilled water) or 30% Hydrogen Peroxide to 5 parts Silica.

Imaging & Analysis	Grain Size, Measurement & Analysis Applications
Hardness Testing	Vickers

used. The simplest is a mixture of 10mℓ hydrogen peroxide (30% concentration – avoid skin contact) and 50mℓ colloidal silica. Some metallographers add either a small amount of Kroll's reagent to this mixture, or a few mℓ of nitric and hydrofluoric acids (avoid contact). These latter additions may cause the suspension to gel. In general, these acid additions do little to improve the action of the hydrogen peroxide (the safer 3% concentration is not effective). Polarized light response of CP titanium can be improved by following this procedure with a brief vibratory polish using colloidal silica.
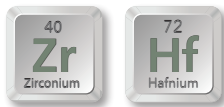
Zirconium and Hafnium

Pure zirconium and pure hafnium are soft, ductile hexagonal close-packed metals that can deform by mechanical twinning if handled aggressively in sectioning and grinding. As with most refractory metals, grinding and polishing removal rates are low and eliminating all polishing scratches and deformation can be difficult. It may even be possible to form mechanical twins in compression mounting. Both can contain very hard phases that make relief control more difficult. To improve polarized light response, it is common practice to chemically polish specimens after mechanical polishing, see Figures 8.3 – 8.4. Alternatively, attack polishing additions can be made to the final polishing abrasive slurry, or
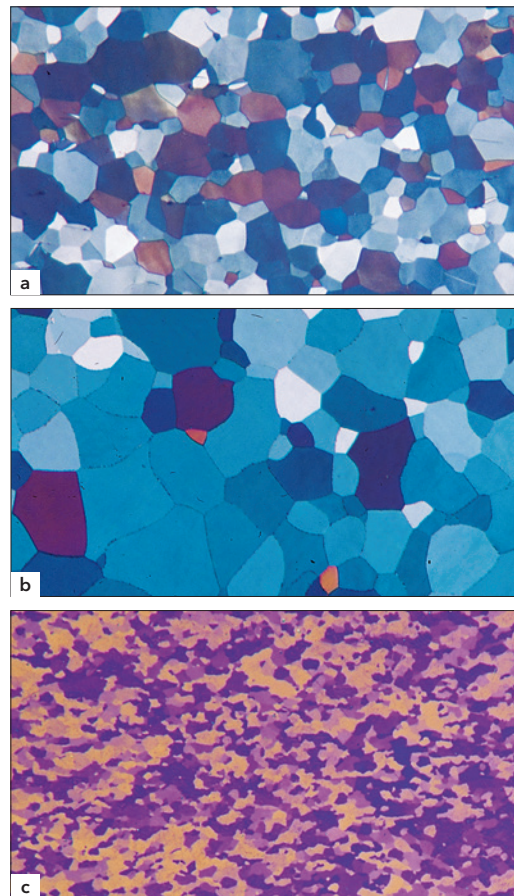
Figure 8.3. a) Equiaxed grain structure of wrought zirconium (unetched, crossed polarized light, 100X). b) Equiaxed grain structure of wrought hafnium (unetched, crossed polarized light plus sensitive tint, 100X). c) Fine grain recrystallized microstructure of wrought Zr - 1.14% Cr strip in the as-polished condition at 200X using polarized light plus sensitive tint.

Table 8.2: 4-Step Method for Zirconium and Hafnium

Sectioning	Precision Saw with a 15LC blade recommended for use on hard brittle materials				
Mounting	Compression, typically with EpoMet				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled*	5 [22]	300	↻ ↻	Until Plane
UltraPad	9μm MetaDi Supreme Diamond**	5 [22]	150	↻ ↻	5:00
TriDent	3μm MetaDi Supreme Diamond**	5 [22]	150	↻ ↻	3:00
ChemoMet	0.02 - 0.06μm Master-Met Colloidal Silica	5 [22]	150	↻ ↻	7:00

● = Platen   ❀ = Specimen Holder

*Rub candle wax lightly across rotating disc prior to grinding   **Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Grain Size, Measurement & Analysis Applications
Hardness Testing	Vickers

vibratory polishing may be employed. Table 8.2 is a four-step procedure that can be followed by either chemical polishing or vibratory polishing.

Several attack polishing agents have been used for Zr and Hf. One is a mixture of 1-2 parts hydrogen peroxide (30% concentration – avoid all skin contact) to 8 or 9 parts colloidal silica. Another is 5mL of a chromium trioxide solution (20g CrO_3 to 100mℓ water) added to 95mℓ colloidal silica or MasterPrep alumina slurry. Additions of oxalic, hydrofluoric or nitric acids have also been used. All of these attack polishing additions must be handled with care as they are strong oxidizers. Skin contact must be avoided. Chemical polishing solutions are reviewed in [2]. Cain's has been popular. Use under a hood and avoid skin contact. Ann Kelly developed an excellent chemical polish for refractory metals, such as Zr, Hf, and Ta. It consists of 25mℓ lactic acid, 15mℓ nitric acid and 5mℓ hydrofluoric acid. Swab vigorously for up to 2 minutes. To prepare ultra-pure Zr and Hf, the above method is unsatisfactory and a procedure, such as in Table 7.1 (add a 5 or 3µm alumina step) is needed, as diamond is ineffective. Use the chromium trioxide attack polish with the alumina and conclude with Kelly's chemical polish.
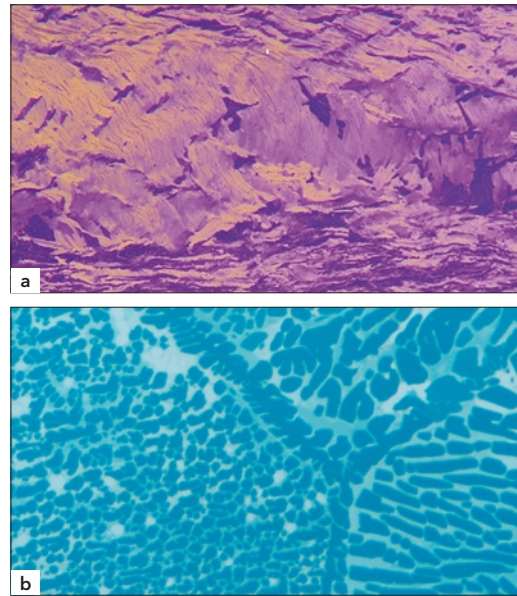
Figure 8.4. a) Cold worked microstructure of wrought zirconium alloy XL (Zr - 3% Sn - 0.8% Nb - 0.8% Mo) in the as-polished condition at 200X with polarized light plus sensitive tint. b) Microstructure (α-Zr + AlZr$_3$) of wrought Zr - 8.6% Al in the as-polished condition at 200X with polarized light.

HELPFUL HINTS FOR REFRACTORY METALS

Due to their very low rate of grinding and polishing, the more aggressive contra rotation gives suitable surfaces in less time.

Refractory metal preparation is aided by using attack-polishing additives. Polarized light response may be improved by following preparation by swabbing with a chemical polishing solution.

OTHER REFRACTORY METALS: Cr, Mo, Nb, Re, Ta, V and W

23 **V** Vanadium	24 **Cr** Chromium	41 **Nb** Niobium	42 **Mo** Molybdenum
73 **Ta** Tantalum	74 **W** Tungsten	75 **Re** Rhenium	
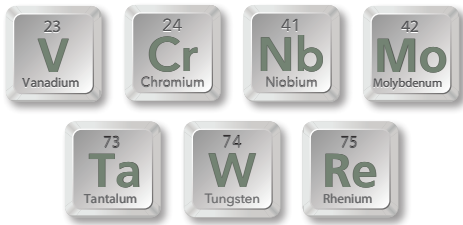
These refractory metals have body-centered cubic crystal structures (except for rhenium which is hexagonal close packed) and are soft and ductile when pure, but some are brittle in commercial form. They can be cold worked easily, although they do not work harden appreciably, so it may be difficult to get completely deformation-free microstructures.
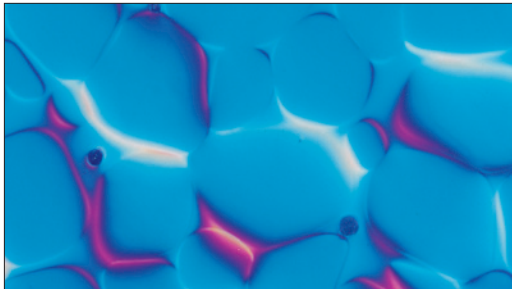
Figure 8.5. Microstructure of powder-made W – 5.6% Ni – 2.4% Fe revealing tungsten grains surrounded by a nickel-iron matrix (unetched, Nomarski DIC, 200X).

Pure chromium is soft and brittle; but, when encountered commercially, for example, as a plated layer, it is hard and brittle. Chromium alloys are relatively easy to prepare, although difficult to etch. Molybdenum (Figures 8.6 and 8.7a) may be tough or brittle

depending upon composition. It is susceptible to deformation damage in sectioning and grinding. Pure niobium (columbium) is soft and ductile and difficult to prepare while its alloys are harder and simpler to prepare. Grinding and polishing rates have been reported to vary with crystallographic orientation. Rhenium is very sensitive to cold work and will form mechanical twins. Tantalum is softer than niobium and more difficult to prepare as it easily forms damaged layers in sectioning and grinding. Tantalum may contain hard phases that promote relief control problems. Vanadium is a soft, ductile metal but may be embrittled by hydrogen; otherwise it can be prepared much like a stainless steel (Figure 8.7b). Tungsten is not too difficult to prepare, although grinding and polishing rates are low. Hard carbides and oxides may be present in these metals that introduce relief control problems, see Figures 8.5 and 8.8.

Mechanical polishing often incorporates an attack-polishing agent in the final step or is followed by vibratory polishing or chemical polishing. Manual preparation of these metals and their alloys tends to be very tedious due to their low grinding and polishing rates. Automated approaches are highly recommended, especially if attack polishing is performed. Following is a generic four step practice suitable for these metals and their alloys, see Table 8.3.

Many attack-polishing additives [2] have been suggested for these metals and their alloys. A good general purpose attack polish consists of a mixture of 5mℓ chromium trioxide solution (20g CrO_3 in 100mℓ water) to 95mℓ MasterMet colloidal silica. Avoid skin contact as this is a strong oxidizing solution. A number of chemical polishing solutions

Table 8.3: 4-Step Method for Refractory Metals

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled	6 [27]	300	◉ ◉	Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	6 [27]	150	◉ ◉	10:00
TriDent	3µm MetaDi Supreme Diamond*	6 [27]	150	◉ ◉	8:00
ChemoMet	0.02 - 0.06µm Master-Met Colloidal Silica plus attack polish agent**	6 [27]	150	◉ ◉	5:00

● = Platen ◉ = Specimen Holder *Plus MetaDi Fluid Extender as desired **See text for agent

have been suggested [2]. For Nb, V and Ta, use a solution consisting of 30mℓ water, 30mℓ nitric acid, 30mℓ hydrochloric acid and 15mℓ hydrofluoric acid. Swab or immerse at room temperature. An alternative chemical polish for Nb, V and Ta consists of 120mℓ water, 6g ferric chloride, 30mℓ hydrochloric acid and 16mℓ hydrofluoric acid. Eary and Johnson recommend immersing the specimen in this solution 1 minute for V, 2 minutes for Nb and 3 minutes for Ta. Vibratory polishing is also very helpful for these metals and their alloys.
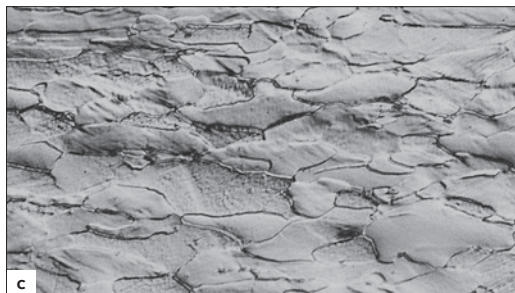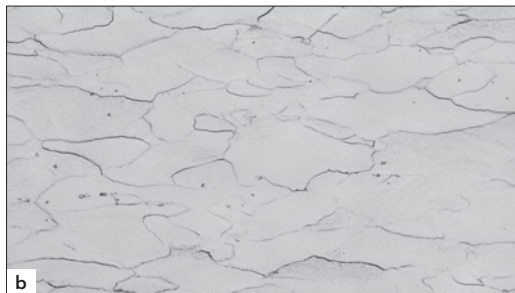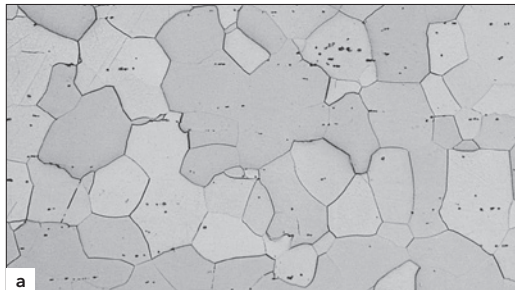
Figure 8.6. a) Alpha grains in Mo – 47.5% Re. The small spots are sigma phase (Murakami's reagent, 200X). Deformed alpha grains in W – 25% Re containing sigma phase (black spots) revealed using b) bright field and c) Nomarski DIC which reveals the cold work much better (Murakami's reagent, 500X).
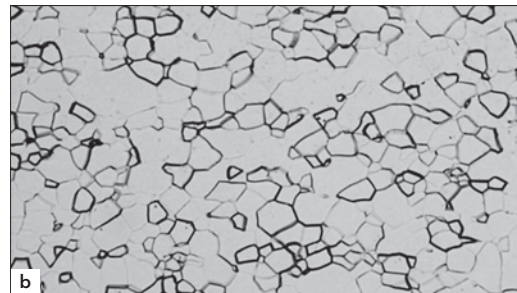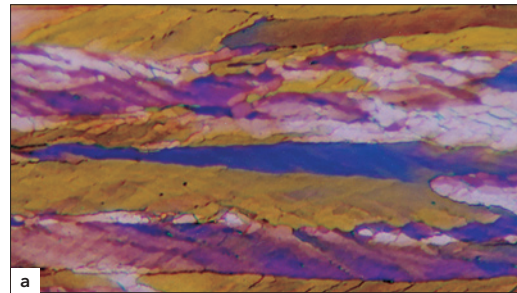
Figure 8.7. a) Non-recrystallized grain structure of wrought pure molybdenum, longitudinal plane (500X, polarized light, etch of water, hydrogen peroxide (30% conc) and sulfuric acid in 7:2:1 ratio). b) Equiaxed alpha grain structure in wrought pure vanadium (200X, etch: glycerol-nitric acid-hydrofluoric acid, 1:1:1 ratio).
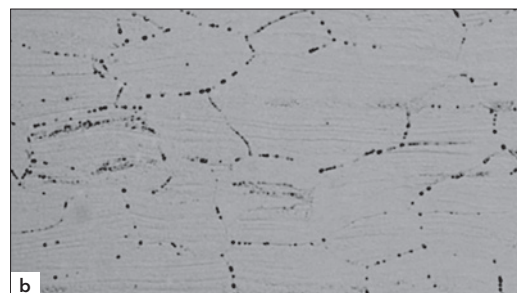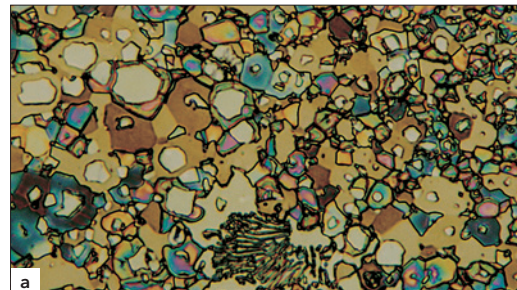
Figure 8.8. a) Wrought tungsten - 10 atomic % Ti containing a small amount of alpha-Ti, beta-Ti-W eutectic and grains of beta-Ti,W of varying composition and crystal orientation (500X, Kroll's reagent/Murakami's reagent at room temperature). b) Fine grain boundary precipitates (not identified) in wrought, cold worked Fan Steel 85-03 alloy (Nb - 28% Ta - 10.5% W - 0.9% Zr), longitudinal plane (500X, etchant: lactic acid-nitric acid-hydrofluoric acid, 30:10:5 ratio).

Ferrous Metals

Iron-based alloys account for a large portion of all metals production. The range of compositions and microstructures of iron-based alloys is far wider than any other system. Pure iron is soft and ductile. Development of scratch-free and deformation-free grain structures is difficult, excellent results are shown in Figure 9.1. Sheet steels present the same problem, which can be complicated by protective coatings of zinc, aluminum or Zn-Al mixtures. In general, harder alloys are much easier to prepare. Cast irons may contain graphite, which must be retained in preparation. Inclusions are frequently evaluated and quantified. Volume fractions can vary from nearly 2% in a free machining grade to levels barely detectable in a premium, double vacuum melt alloy. A wide range of inclusion, carbide and nitride phases has been identified in steels. Addition of 12 or more percent chromium dramatically reduces the corrosion rate of steels, producing a wide range of stainless steel alloys. Tool steels cover a wide range of compositions and can attain very high hardnesses. Preparation of ferrous metals and alloys is quite straightforward using the contemporary methods. Edge retention (see guidelines on pages 13-15) and inclusion retention are excellent, especially if automated equipment is used. The following procedures are recommended and are adequate for the vast majority of ferrous metals and alloys. Table 4.3 presented a variation of the method given in Table 9.1. This practice is also useful for cast iron specimens including graphitic cast irons, however, due to their high silicon content and the potential for staining problems, it is best to use the MasterPrep alumina suspension for the final polishing step.

For softer steels, use the 320 [P400] grit SiC paper, for step 1. The Apex Hercules S disc can be used but this is not usually necessary. However, if substituted, use 6µm MetaDi Supreme for the second step instead of 9µm, as shown in Table 9.2.

This practice is well suited for solution annealed austenitic stainless steels and for soft sheet steels. UltraPol or UltraPad cloths could be substitute for the rigid grinding discs, if desired. For perfect publication quality images, or for color etching, follow this practice with a brief vibratory polish using the cloths and abrasives in the last step.
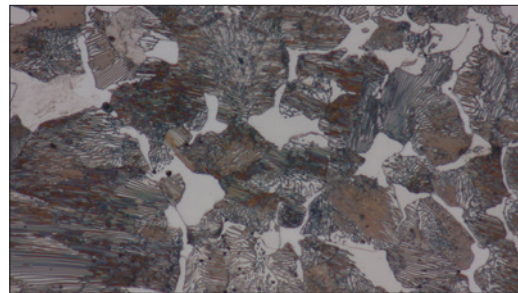
Figure 9.1. Normalized carbon steel shows coarse ferritic-pearlitic (grains with lamellar cementite) structure. Etched with 3% Nital (200X).

Table 9.1: 4-Step Method for Hard Steel

Sectioning	Abrasive Cutter with a wheel recommended for use on ferrous materials HRC35-60				
Mounting	Compression or Castable, typically with EpoMet or VariDur 3000				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex DGD Red	75µm Diamond water cooled	6 [27]	300	⬤ ✸	Until Plane
Apex Hercules H	9µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ✸	5:00
TriDent	3µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ✸	3:00
MicroCloth	0.05µm MasterPrep Alumina	6 [27]	150	⬤ ✸	2:00

⬤ = Platen   ✸ = Specimen Holder   *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Grain Size, Phase Area Percent, Object Measurement, Measurement & Analysis Applications
Hardness Testing	Vickers, Knoop, Rockwell

FERROUS METALS

Table 9.2: 4-Step Method for Soft Steel

Sectioning	Abrasive Cutter with a wheel recommended for use on ferrous materials HRC15-35				
Mounting	Compression or Castable, typically with EpoMet or VariDur 200				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320grit [P400] SiC water cooled	6 [27]	300	⚫ ⚙	Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	6 [27]	150	⚫ ⚙	5:00
VerduTex	3µm MetaDi Supreme Diamond*	6 [27]	150	⚫ ⚙	3:00
MicroCloth	0.05µm MasterPrep Alumina	6 [27]	150	⚫ ⚙	2:00

⚫ = *Platen*   ⚙ = *Specimen Holder*   *Plus MetaDi Fluid Extender as desired*

Imaging & Analysis	Grain Size, Object Measurement, Measurement & Analysis Applications
Hardness Testing	Vickers, Knoop

Many steels, particularly the harder steels, can be prepared in three steps with excellent results. A recommended practice is given in Table 9.3. Instead of the diamond grinding disc, SiC can be used if preferred. For soft alloys, use 240 or 320 [P280 or P400] grit SiC paper; harder alloys use 120 [P120], 180 [P180], or 240 [P280] grit SiC paper, depending upon the starting surface finish and the hardness of the alloy.   The UltraPad cloth can also be used for the second step for steels of any hardness.

The following practice is recommended for stainless steels and maraging steels. For solution annealed austenitic grades and for ferritic stainless grades and for annealed maraging grades, use the Apex Hercules S disc or the UltraPol cloth and a 30um MetaDi Supreme polycrystalline diamond supsension for best results. Start with 120 grit [P120] SiC paper only if it is a very hard martensitic stainless steel, such as type 440C. The solution annealed austenitic stainless steels and the fully ferritic stainless steels (Figure 9.2) are the most difficult to prepare. It may be helpful to add a 1µm diamond step on a TriDent cloth before the last step, or to follow the last step with a brief vibratory polish using colloidal silica on MicroCloth or a ChemoMet cloth, see Table 9.4.

Table 9.3: 3-Step Method for Heat-Treated Steel

Sectioning	Abrasive Cutter with a wheel recommended for use on ferrous materials HRC35-60				
Mounting	Compression or Castable, typically with EpoMet or VariDur 3000				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex DGD Red	75µm Diamond water cooled	6 [27]	300	⚫ ⚙	Until Plane
Apex Hercules S	9µm MetaDi Supreme Diamond*	6 [27]	150	⚫ ⚙	5:00
MicroFloc	3µm MetaDi Supreme Diamond*	6 [27]	150	⚫ ⚙	3:00

⚫ = *Platen*   ⚙ = *Specimen Holder*   *Plus MetaDi Fluid Extender as desired*

Imaging & Analysis	Case Depth, Decarburization Depth Assessment, Grain Size, Phase Area Percent, Object Measurement, Measurement & Analysis Applications
Hardness Testing	Vickers, Knoop, Rockwell

Cast Iron

Cast iron is a ferrous material with high carbon content (> 2%), Silicon (> 1.5%) and other components such as manganese, chromium or nickel. In general there is a distinction between gray cast iron (carbon is in the form of graphite) and white cast iron (carbon is in the form of cementite). The simplest type of cast iron is grey cast iron (ASTM A 48), also called "GJL" according to the European Norm. Grey cast iron, or grey iron, has graphite flakes which act as notches when tensile forces are applied, so the tensile strength is relatively low, as in Figuer 9.3. Compared to this the compressive strength is much better. Cast iron "GJS" (DIN EN 1563 cast iron, ductile iron, spherolitic graphite) has better mechanical properties, due to the spheroidal

form of the graphite it contains. The graphite is in the form of spherical nodules rather than flakes, which helps prevent the creation of cracks and provides improved ductility as shown in Figure 9.4. This form is achieved by desulphurization of the melt and the addition of small amounts of magnesium, cerium or calcium prior to casting. Preparation artifacts can occur due to insufficient time or too low material removal rates. The effects are visible as pull outs of

Figure 9.3. Pearlitic gray cast iron, etched with Beraha's CdS tint (1300X).

Figure 9.2. Microstructure of a duplex (ferrite tan, austenite white) stainless steel held at 816° C for 48 hours which forms sigma phase (orange particles) revealed by electrolytic etching with aqueous 20% NaOH at 3 V DC, 9 seconds (500X).
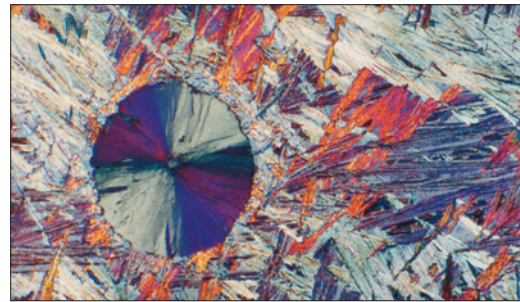
Figure 9.4. Nodular graphite, lower bainite and retained austenite in austempered ductile iron, etched with modified Beraha-martensite reagent (~1300X).

Table 9.4: 4-Step Method for Stainless and Maraging Steel					
Sectioning	Abrasive Cutter with a wheel recommended for use on ferrous materials HRC35-50				
Mounting	Compression, typically with EpoMet				
Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	120 [P120] to 320grit [P400] SiC water cooled	6 [27]	300	⬤ ✸	Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ✸	5:00
TriDent	3µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ✸	3:00
ChemoMet	0.05µm MasterPrep Alumina	6 [27]	150	⬤ ✸	2:00
⬤ = Platen ✸ = Specimen Holder *Plus MetaDi Fluid Extender as desired					
Imaging & Analysis	Grain Size, Phase Area Percent, Object Measurement, Measurement & Analysis Applications				
Hardness Testing	Vickers, Knoop, Rockwell				

FERROUS METALS

Table 9.5 shows a good practice to prepare cast iron.

Sectioning	Abrasive cutter with a wheel recommended for use on ferrous materials HRC35-50				
Mounting	Compression or Castable				
Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320grit [P400] SiC water cooled	5 [22]	300		Until Plane
TexMet C	9µm MetaDi Diamond Supreme*	5 [22]	150		5:00
TriDent	3µm MetaDi Diamond Supreme*	5 [22]	150		4:00
MicroCloth	0.05µm MasterPrep Alumina	5 [22]	150		2:00

⬤ = *Platen*   ✹ = *Specimen Holder*

Imaging & Analysis	Graphite in Cast Iron
Hardness Testing	Brinell

Figure 10.1. Alpha grains containing annealing twins in phosphorous-deoxidized arsenical bronze that was annealed and lightly cold drawn (Klemm's I reagent, polarized light, 50X).

Copper

Pure copper is extremely ductile and malleable. Copper and its alloys come in a wide range of compositions, including several variants of nearly pure copper for electrical applications to highly alloyed brasses and bronzes and to precipitation hardened high strength alloys. Copper and its alloys can be easily damaged by rough sectioning and grinding practices and the depth of damage can be substantial. Scratch removal, particularly for pure copper and brass alloys, can be very difficult. Following the preparation cycle with a brief vibratory polish using colloidal silica is very helpful for scratch removal. Attack-polish additions have been used in the past to improve scratch removal but usually are not necessary using the contemporary method followed by vibratory polishing, see Table 10.1 and Figure 10.1 for results.

Planar grinding can be performed using the PlanarMet 300 or resin bonded Apex DGD col- or with 35µm for any copper alloy. Avoid the use of metal bonded discs as they introduce too much mechanical stress.

Table 10.1: 5-Step Method for Cu and Cu Alloys

Sectioning	Abrasive Cutter with a wheel recommended for use on non-ferrous materials				
Mounting	Compression or Castable, typically with PhenoCure or VariDur				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	220 [P240] to 320 [P400] grit SiC water cooled	5 [22]	300	◑ ✦	Until Plane
TexMet C	9µm MetaDi Supreme Diamond*	5 [22]	150	◑ ✦	5:00
VerduTex	3µm MetaDi Supreme Diamond*	5 [22]	150	◑ ✦	3:00
VerduTex	1µm MetaDi Supreme Diamond*	5 [22]	150	◑ ✦	2:00
ChemoMet	0.02 - 0.06µm Master-Met Colloidal Silica**	5 [22]	150	◑ ✦	1:30

● = Platen ✦ = Specimen Holder   *Plus MetaDi Fluid Extender as desired   **Plus attack polishing reagent (see text for details)

Imaging & Analysis	Grain Size, Coating Thickness Layer, Measurement & Analysis Applications
Hardness Testing	Vickers, Knoop

![28 Ni Nickel]

Nickel

Nickel and its alloys have face-centered cubic crystal structures and are prepared in basically the same way as austenitic stainless steels. Pure nickel is more difficult to prepare than the alloys. The Ni-Fe magnetic alloys are rather difficult to prepare scratch free unless vibratory polishing is used. The Monel (Ni-Cu) and the highly corrosion resistant (Ni-Cr-Fe) alloys are more difficult to prepare than the nickel-based superalloys. Solution annealed superalloys are always more difficult to prepare than age hardened superalloys (Figure 10.2). Age hardened superalloys can be prepared using the Apex Hercules H disc; for all other nickel alloys, use the Apex Hercules S disc for best results. The following practice works well for nickel based superalloys (and Fe-Ni based super alloys) and the highly corrosion resistant Ni-Cr-Fe alloys, see Table 10.2.

If color etching is to be performed, follow the last step with a brief vibratory polish using the same materials as in the last step. This step is also helpful for the most difficult to prepare solution annealed alloys. Alternatively, for the most difficult specimens, or when color etching is being performed, a 1µm diamond step on a Trident cloth can be added before the final step.

For pure nickel, nickel-copper and nickel-iron alloys, a five step practice is preferred, as given below. The planar grinding step can be performed using either the 30µm resin-bonded UltraPrep diamond disc or with 240 [P280] or 320 [P400] grit SiC papers with equal success, see Table 10.3.

Attack-polishing agents are not often used with these alloys to eliminate fine polishing scratches or residual damage. If this is a problem, and some of these grades are very difficult to get perfectly free of scratches and deformation damage, a brief vibratory polish, using the same materials as in the last step, will provide the needed improvement. MasterPrep alumina may give better results than colloidal silica for the more pure nickel compositions.
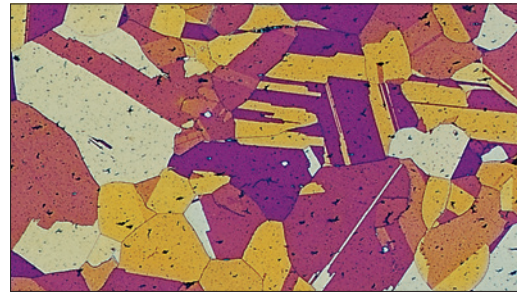
Figure 10.2. Alpha grains containing annealing twins of solution annealed and double aged Waspaloy nickel-based superalloy (Beraha's reagent, 100X).

Table 10.2: 4-Step Method for Ni-Based Superalloys					
Sectioning	Abrasive Cutter with a wheel recommended for use on SuperAlloys				
Mounting	Compression, typically with EpoMet				
Surface	**Abrasive / Size**	**Load - lbs [N] / Specimen**	**Base Speed [rpm]**	**Relative Rotation**	**Time [min:sec]**
CarbiMet	240 [P280] grit SiC water cooled	6 [27]	300	⟳ ⟳	Until Plane
Apex Hercules H or S	9µm MetaDi Supreme Diamond*	6 [27]	150	⟳ ⟳	5:00
TriDent	3µm MetaDi Supreme Diamond*	6 [27]	150	⟳ ⟳	5:00
ChemoMet	0.02 - 0.06µm Master-Met Colloidal Silica**	6 [27]	150	⟳ ⟳	2:00

● = Platen ✸ = Specimen Holder   *Plus MetaDi Fluid Extender as desired   **Plus attack polishing reagent (see text for details)

Imaging & Analysis	Grain Size, Porosity Assesment, Measurement & Analysis Applications
Hardness Testing	Vickers, Knoop

Table 10.3: 5-Step Method for Ni, Ni-Cu and Ni-Fe Alloys

Sectioning	Abrasive Cutter with a wheel recommended for use on SuperAlloys				
Mounting	Compression, typically with EpoMet				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	240 [P280] grit SiC water cooled	6 [27]	300	↻ ↻	Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	6 [27]	150	↻ ↻	5:00
TriDent	3µm MetaDi Supreme Diamond*	6 [27]	150	↻ ↻	3:00
TriDent	1µm MetaDi Supreme Diamond*	6 [27]	150	↻ ↻	2:00
ChemoMet	0.02 - 0.06µm Master-Met Colloidal Silica**	6 [27]	150	↻ ↻	2:00

● = Platen ❇ = Specimen Holder   *Plus MetaDi Fluid Extender as desired   **Plus attack polishing reagent (see text for details)

Imaging & Analysis	Grain Size, Porosity Assesment, Measurement & Analysis Applications
Hardness Testing	Vickers, Knoop

27 Co Cobal t

Cobalt

Cobalt and its alloys are more difficult to prepare than nickel and its alloys. Cobalt is a tough metal with a hexagonal close-packed crystal structure and is sensitive to deformation damage by mechanical twinning. Grinding and polishing rates are lower for Co than for Ni, Cu or Fe. Preparation of cobalt and its alloys is somewhat similar to that of refractory metals. Despite its HCP crystal structure, crossed polarized light is not very useful for examining cobalt alloys compared to other HCP metals and alloys. Following is a practice for preparing Co and its alloys, see Table 10.4 and Figure 10.3 for results.

Two steps of SiC paper may be needed to get the specimens co-planar. If the cut surface is of good quality, start with 320 [P400] grit paper. Cobalt and its alloys are more difficult to cut than most steels, regardless of their hardness. Attack polishing has not been reported but chemical polishing has been used after mechanical polishing. Morral (2) has recommended two chemical polishing solutions: equal parts of acetic and nitric acids (immerse) or 40mℓ lactic acid, 30mℓ hydrochloric acid and 5mℓ nitric acid (immerse). A wide variety of Co-based alloys have been prepared with the method Table 10.4 without need for chemical polishing. The 1µm diamond step could be eliminated for routine work.
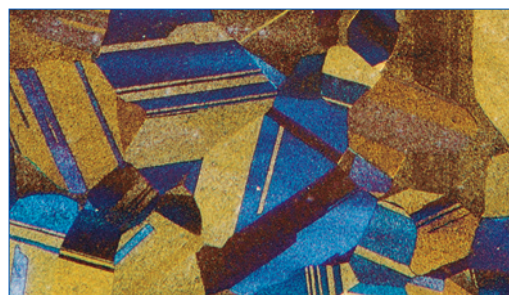
Figure 10.3. Equiaxed grain structure of Elgiloy (Co – 20% Cr – 15% Fe – 15% Ni – 7% Mo – 2% Mn – 0.15% C – 0.05% Be) after hot rolling and annealing revealing annealing twins (Beraha's reagent, crossed polarized light plus sensitive tint, 100X).

COPPER, NICKEL, COBALT

Table 10.4: 5-Step Method for Co					
Sectioning	Abrasive Cutter with a wheel recommended for use on SuperAlloys				
Mounting	Compression, typically with EpoMet				
Surface	**Abrasive / Size**	**Load - lbs [N] / Specimen**	**Base Speed [rpm]**	**Relative Rotation**	**Time [min:sec]**
CarbiMet	320 [P400] grit SiC water cooled	6 [27]	300	⬤ ⬤	Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ⬤	5:00
TexMet C	3µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ⬤	5:00
TexMet C	1µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ⬤	3:00
ChemoMet	0.02 - 0.06µm MasterPrep Alumina	6 [27]	150	⬤ ⬤	2:00

⬤ = Platen ⬤ = Specimen Holder   *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Grain Size, Porosity Assesment, Measurement & Analysis Applications
Hardness Testing	Vickers, Knoop

PRECIOUS METALS: Au, Ag, Ir, Os, Pd, Pt, Rh and Ru

Relatively few metallographers work with precious metals, other than those used in electronic devices. Preparing precious metals within an integrated circuit is discussed later (see Electronic Materials). The precious metals are very soft and ductile, deform and smear easily, and are quite challenging to prepare. Pure gold is very soft and the most malleable metal known. Alloys, which are more commonly encountered, are harder and somewhat easier to prepare. Gold is difficult to etch. Silver is very soft and ductile and prone to surface damage from deformation, see Figure 11.1. Embedding of abrasives is a common problem with both gold and silver and their alloys. Iridium is much harder and more easily prepared. Osmium is rarely encountered in its pure form, even its alloys are infrequent subjects for metallographers. Damaged surface layers are easily produced and grinding and polishing rates are low. It is quite difficult to prepare. Palladium is malleable and not as difficult to prepare as most of the precious metals. Platinum is soft and malleable. Its alloys are more commonly encountered. Abrasive embedment is a problem with Pt and its alloys. Rhodium is a hard metal and is relatively easy to prepare. Rh is sensitive to surface damage in sectioning and grinding. Ruthenium is a hard, brittle metal that is not too difficult to prepare.

Stewart (Tech-Notes, Vol. 2, Issue 5) has described a method for preparing jewelry alloys. Invariably, these are small pieces, due to their cost, and must be mounted. Stewart uses EpoMet G resin for most specimens. If transparency is need, he uses TransOptic resin. Fragile beads and balls are mounted in castable resins. Grinding and polishing was conducted at 400 rpm. His practice with equivalent current materials is shown in Table 11.1.
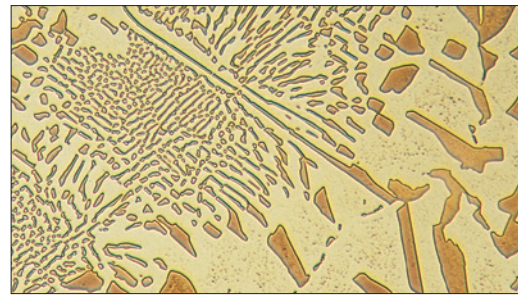
Figure 11.1. Eutectic microstructure of Ag – 28% Cu where Klemm's reagent has colored the copper particles and the silver phase is uncolored (500X).

Table 11.1: Stewart's Manual Procedure for Precious Metals

Sectioning	Precision Cutter with 30HC blade recommended for use on soft gummy materials			
Mounting	Castable, typically with EpoThin			

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Time [min:sec]
CarbiMet	240 [P280] grit SiC water cooled (wax coated)*	Moderate	400	Until Plane
CarbiMet	320 [P400] grit SiC water cooled (wax coated)*	Moderate	400	1:00
CarbiMet	400 [P600] grit SiC water cooled (wax coated)*	Moderate	400	1:00
CarbiMet	600 [P1200] grit SiC water cooled (wax coated)*	Moderate	400	1:00
TriDent	6µm MetaDi Supreme Diamond**	Moderate	400	2:00
MasterTex	1µm MetaDi Supreme Diamond**	Moderate	400	2:00

*Rub candle was lightly across rotating disc prior to grinding   **Plus MetaDi Fluid Extender as desired*

Imaging & Analysis	Porosity Assessment, OmniMet Object Measurements, Measurement & Analysis Applications
Hardness Testing	Vickers, Knoop

This can be followed by a brief vibratory polish using colloidal silica on MasterTex, ChemoMet or Micro-Cloth surfaces to further enhance the quality of the preparation; but, a 1µm diamond finish is adequate for most work. Attack-polishing has been used to prepare gold and its alloys and chemical polishing has been performed after mechanical polishing of silver, but neither practice is commonly performed. Alternate etch-polish cycles may be needed to remove fine polishing scratches, especially for annealed specimens.

A procedure for an automated system was developed after experimentation with a number of precious metal specimens. Most of these metals and alloys are quite soft, unless they have been cold worked, and they are susceptible to embedding of abrasives. In this method, only one silicon carbide step is used. The TexMet C cloth is used for the diamond steps, as it will hold the abrasive in its surface well, which minimizes embedding. Only diamond paste is used, as slurries will be more prone to embedding, as we observed in high gold alloys. Use only a small amount of distilled water as the lubricant. Do not get the cloth excessively wet. Final polishing is with a ChemoMet cloth and MasterPrep alumina. Due to their excellent corrosion resistance, colloidal silica is not effective as an abrasive for precious metals. The ChemoMet cloth has many fine pores to hold the abrasive. The cycle is given in Table 11.2.

For 18 karat gold (Figure 11.2) and higher (≥75% Au), it is necessary to use an attack polish agent in the final step. An aqueous solution of 5g CrO_3 in 100mℓ water works well. Mix 10mℓ of the attack polish agent with 50mℓ of MasterPrep suspension. This will thicken, so add about 20-30mℓ water to make it thinner. A 3 to 6 minute attack polish step will remove the fine polishing scratches. Wear protective gloves as the chromium trioxide solution is a strong oxidizer.
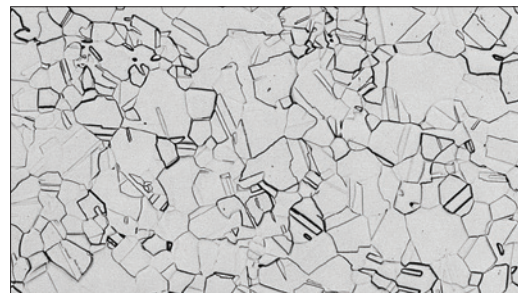
Figure 11.2. Equiaxed grain structure of cold rolled and annealed 18-karat gold (Neyoro 28A: 75% Au – 22% Ag – 3% Ni) revealing annealing twins (equal parts 10% NaCN and 30% H_2O_2, 50X).

Table 11.2: 5-Step Method for Precious Metals

Sectioning	Precision Saw with a blade recommended for use on soft gummer materials				
Mounting	Castable, typically with EpoThin				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled	3 [13]	150	⬤ ✸	Until Plane
TexMet C	9µm MetaDi Supreme Diamond*	3 [13]	150	⬤ ✸	5:00
TexMet C	3µm MetaDi Supreme Diamond*	3 [13]	150	⬤ ✸	3:00
TexMet C	1µm MetaDi Supreme Diamond*	3 [13]	150	⬤ ✸	2:00
ChemoMet	0.05µm MasterPrep Alumina	2 [9]	150	⬤ ✸	2:00

⬤ = Platen ✸ = Specimen Holder *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Porosity Assesment, OmniMet Object Measurements, Measurement & Analysis Applications
Hardness Testing	Vickers, Knoop

THERMAL SPRAY COATINGS

Thermal spray coatings (TSC) and thermal barrier coatings (TBC) are widely used on many metal substrates. Invariably, these coatings are not 100% dense but contain several types of voids, such as porosity and linear detachments. Hot compression mounting is not recommended as the molding pressure can collapse the voids. Use a low-viscosity castable epoxy with vacuum infiltration to fill the connected voids with epoxy. Fluorescent dyes may be added to the epoxy. When viewed with fluorescent illumination, the epoxy-filled voids appear bright yellow-green. This makes it easy to discriminate between dark holes and dark oxides, as would be seen with bright field illumination. Filling the pores with epoxy also makes it easier to keep the pore walls flat to the edge during preparation. Aside from this mounting requirement, TSC and TBC specimens are prepared using all of the factors needed for good edge retention (see pages 14-17). A variety of procedures can be used. The Apex Hercules H rigid grinding disc produces exceptional edge flatness for these specimens. Two four-step procedures for TSC and TBC specimens for specimens with metallic coatings and one procedure for specimens with ceramic coatings are given, see Tables 12.1–12.3.

Table 12.1: 4-Step Method for TSC and TBC Specimens with Metallic Coatings

Sectioning	Precision Saw with 15HC blade recommend for thermal spray coatings
Mounting	Castable, typically with epoxy drawn under vacuum

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex DGD Yellow	35µm Diamond water cooled	6 [27]	300		Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	6 [27]	150		5:00
TriDent	3µm MetaDi Supreme Diamond*	6 [27]	150		5:00
ChemoMet	0.02 - 0.06µm MasterMet Colloidal Silica	4 [18]	150		2:00

● = Platen ✸ = Specimen Holder *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Porosity Assessment, Coating Layer Thickness, Particle Sizing, Phase Area Percent
Hardness Testing	Vickers, Knoop

Table 12.2: Alternate 4-Step Method for TSC and TBC Specimens with Metallic Coatings

Sectioning	Precision Saw with 15HC blade recommend for thermal spray coatings
Mounting	Castable, typically with epoxy drawn under vacuum

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex Hercules H	30µm MetaDi Diamond*	6 [27]	300		Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	6 [27]	150		4:00
TriDent	3µm MetaDi Supreme Diamond*	6 [27]	150		3:00
ChemoMet	0.02 - 0.06µm MasterMet Colloidal Silica	4 [18]	150		2:00

● = Platen ✸ = Specimen Holder *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Porosity Assessment, Coating Layer Thickness, Particle Sizing, Phase Area Percent
Hardness Testing	Vickers, Knoop

THERMAL SPRAY COATINGS

The 30µm resin bonded, or the 45µm metal bonded UltraPrep diamond discs, can be substituted for the planar grinding step. (Tables 12.1 and 12.2).

UltraPol or UltraPad cloths can be used in the second step (Tables 12.1 and 12.3) with results shown in Figures 12.1 and 12.2.
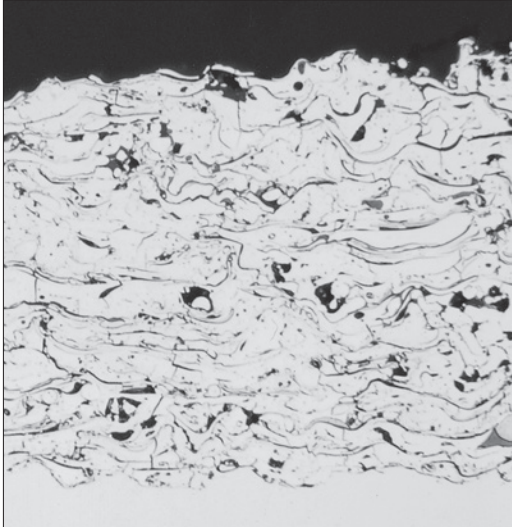
Figure 12.1. NiCrAlY thermally-spray coated steel specimen revealing a small amount of porosity (black spots), linear detachments (elongated black lines), and inclusions (gray particles) (unetched, 100X).
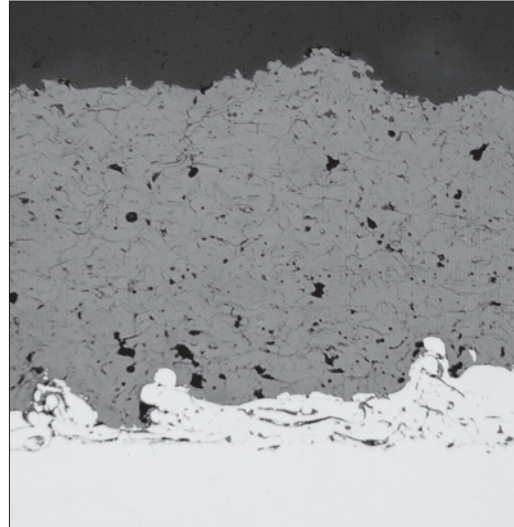
Figure 12.2. Microstructure of a steel substrate covered by two thermally-sprayed layers, a NiAl bond coat and yittria-zirconia top coat (unetched, 100X). The bond coat contains pores, linear detachments and inclusions while the top coat is quite porous.

Table 12.3: 4-Step Method for Ceramic Thermal Spray Coatings

Sectioning	Precision Saw with 15HC blade recommend for thermal spray coatings				
Mounting	Castable, typically with epoxy drawn under vacuum				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex DGD Yellow	35µm Diamond water cooled	5 [22]	300	⬤ ⚙	Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	5 [22]	150	⬤ ⚙	5:00
TriDent	3µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ⚙	5:00
ChemoMet	0.02 - 0.06µm Master-Met Colloidal Silica	6 [27]	150	⬤ ⚙	2:00

⬤ = Platen   ⚙ = Specimen Holder   *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Porosity Assessment, Coating Layer Thickness, Particle Sizing, Phase Area Percent
Hardness Testing	Vickers, Knoop

Sintered carbides are very hard materials made by the powder metallurgy process and may by reinforced with several types of MC-type carbides besides the usual tungsten carbide (WC) as shown in Figure 13.1 – 13.3. The binder phase is normally cobalt although minor use is made of nickel. Modern cutting tools are frequently coated with a variety of very hard phases, such as alumina, titanium carbide, titanium nitride and titanium carbonitride. Sectioning is normally performed with a precision saw, so surfaces are very good and rough abrasives are not usually required. Listed below are two alternate procedures for preparing sintered carbides using Apex DGD metal-bonded UltraPrep discs or the Apex Hercules H rigid grinding discs. A further option would be the use of the traditional metal-bonded diamond discs.

If a greater amount of material must be removed in the planar grinding step, use either the 30µm resin bonded or the 45µm metal-bonded UltraPrep disc for step one, in Table 13.1.

The final step in these two procedures, Tables 13.1 and 13.2, employs either MasterMet colloidal silica or MasterPrep alumina suspensions as they will produce the best definition of the structure, particularly if the surfaces have a complex series of coatings for improved wear resistance, as used in some coated carbide inserts. However, if such a coating is not present, the matrix grain structure can be observed quite clearly after the 3µm diamond
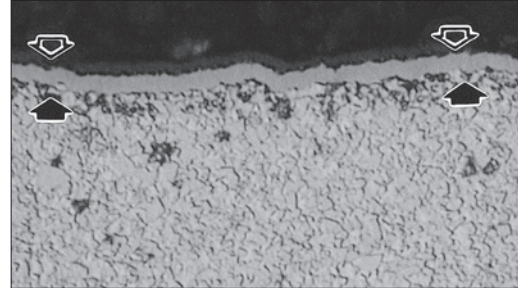
Figure 13.1. Surface of a WC – 11.5% Co cutting tool enriched with 1.9% Ta and 0.4% Nb (form complex carbides, the dark regions in the matrix) and coated with alumina (arrows) for enhanced tool life (Murakami's reagent, 1000X).

HELPFUL HINTS FOR SINTERED CARBIDES

The ability to see the boundaries between the WC particles and cobalt binder in the as-polished condition depends on the surface of the polishing cloth used in the last step. To see no boundaries, which makes it easier to see graphite or eta phase, use a napless surface, such as TriDent, TexMet or UltraPol cloths. To see the phase boundaries between WC particles and the cobalt binder, use a medium nap surface, such as MicroCloth, MasterTex cloths.

Table 13.1: 4-Step Method for Sintered Carbides					
Sectioning	Precision Saw with 20LC blade recommended for hard tough materials				
Mounting	Castable, typically epoxy with added flat-edge filler				
Surface	**Abrasive / Size**	**Load - lbs [N] / Specimen**	**Base Speed [rpm]**	**Relative Rotation**	**Time [min:sec]**
UltraPrep Metal-Bonded	45µm Diamond water cooled	6 [27]	300	◉ ◉	5:00†
TexMet P	9µm MetaDi Supreme Diamond*	6 [27]	150	◉ ◉	4:00
VerduTex	3µm MetaDi Supreme Diamond*	6 [27]	150	◉ ◉	3:00
ChemoMet	0.02 - 0.06µm MasterMet Colloidal Silica	6 [27]	150	◉ ◉	2:00
● = Platen ◉ = Specimen Holder *Plus MetaDi Fluid Extender as desired					
Imaging & Analysis	Porosity Assessment, Particle Sizing, Coating Layer Thickness				
Hardness Testing	N/A				
†Grind longer than "until plane"					

Table 13.2: 4-Step Method for Sintered Carbides

Sectioning	Precision Saw with 20LC blade recommended for hard tough materials				
Mounting	Castable, typically epoxy with added flat-edge filler				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex Hercules H	30µm MetaDi Supreme Diamond*	6 [27]	150-200	⚫ ⚙	5:00†
Apex Hercules S	9µm MetaDi Supreme Diamond*	6 [27]	150	⚫ ⚙	4:00
VerduTex	3µm MetaDi Supreme Diamond*	6 [27]	150	⚫ ⚙	3:00
ChemoMet	0.02 - 0.06µm Master-Met Colloidal Silica	6 [27]	150	⚫ ⚙	2:00

⚫ = Platen   ⚙ = Specimen Holder   *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Porosity Assessment, Particle Sizing, Coating Layer Thickness
Hardness Testing	Vickers

†Grind longer than "until plane"
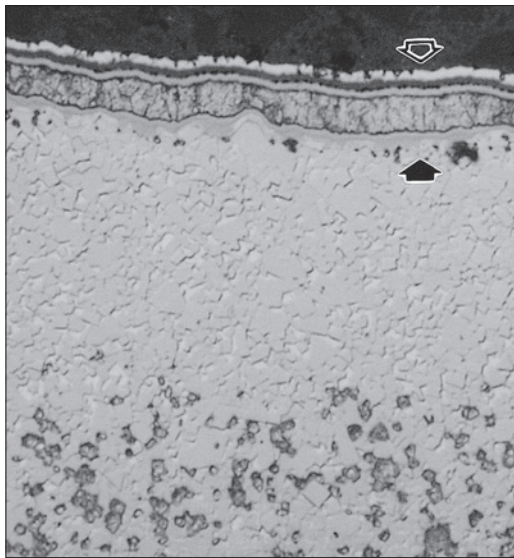
Figure 13.2. Microstructure at the surface of a multilayered, CVD-coated WC - 8% Co cutting tool. The arrows point to the CVD layers of TiCN, TiN, TiC and alumina. The region below the coatings is made higher in Co to improve crack resistance and complex carbide forming elements (Ta, Ti and Nb) are added to the matrix (dark spots at bottom) for wear resistance (Murakami's reagent, 1000X).
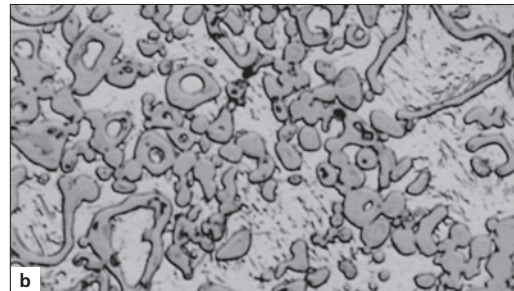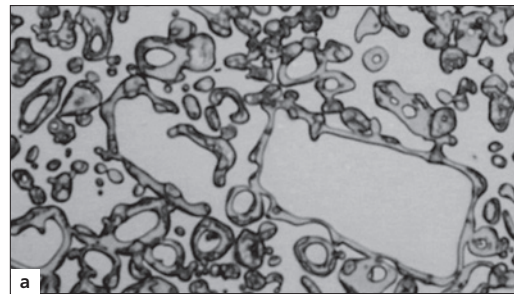
Figure 13.3. Microstructure of cold isostatically pressed and age hardened Ferro-Titanit Nikro 128 (Fe - 13.5% Cr - 9% Co - 4% Ni - 30% TiC) cutting tool; a) etched with Murakami's reagent at room temperature to darken the TiC; and b) electrolytic etched with aqueous 1% chromium trioxide at 2 V DC, 30 seconds to reveal the martensitic matrix (1000X).

step. For routine examination there is little need to go beyond the third step. It is also possible to use a 1µm diamond step, similar to step three, but for 2 minutes, as an alternative fourth step.

Ceramic materials are extremely hard and brittle and may contain pores. Sectioning must be performed using diamond blades. If the specimen is to be thermally etched, then it must be mounted in a resin that permits easy demounting, and vacuum infiltration of epoxy into the voids should not be done. Deformation and smearing are not problems with ceramics due to their inherent characteristics. But, it is possible to break out grains or produce cracking during preparation.

Pullouts are a major problem to control as they can be misinterpreted as porosity. Mechanical preparation has been done successfully with laps, metal-bonded diamond discs, rigid grinding discs, or hard cloths. SiC paper is rather ineffective with most ceramics, as they are nearly as hard or as hard as the SiC abrasive. Consequently, diamond is commonly used for nearly all preparation steps. Automated preparation is highly recommended when preparing ceramic materials as very high forces arise between the specimen and the working surface, often too high for manual preparation. Listed are two generic procedures for preparing ceramics using different approaches, see Tables 14.1 and 14.2 also Figures 14.1 and 14.2 for results.

MasterPolish 2 suspension is specifically formulated for final polishing ceramic materials and this step can me added for a more thorough examination as it will yield a better surface finish than 1μm diamond.
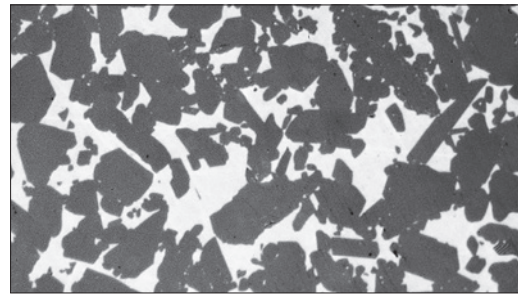
Figure 14.2. Microstructure of hot-pressed silicon nitride (binder unknown) (unetched, 500X).

HELPFUL HINTS FOR CERAMICS

Cut specimens as close as possible to the desired examination plane using a saw and blade that produces the least possible amount of damage, producing the best possible surface for subsequent preparation. Rough grinding is slow and tedious and does considerable damage to the structure and should be avoided as much as possible.

Coarse abrasives for grinding may be needed at times. However, severe grain pull-outs can result from use of coarse abrasives and it is recommended to use the finest possible coarse abrasive for grinding.
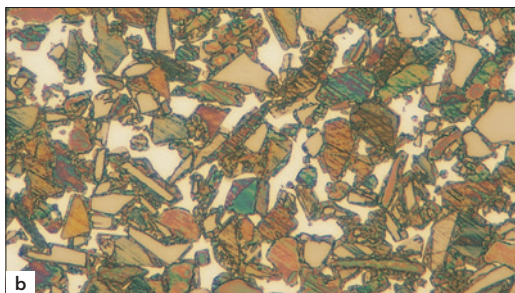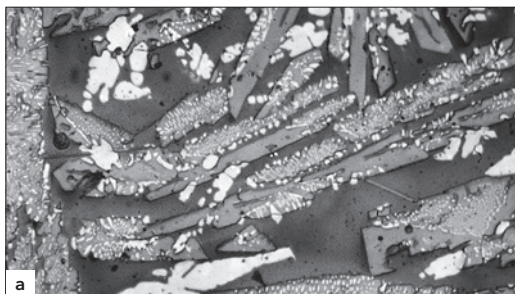
Figure 14.1. a) Microstructure of an alumina (matrix), zirconia (gray), silica (white) refractory material (unetched, 200X). b) Silicon - SiC ceramic etched electrolytically with 10% oxalic acid (500X).

CERAMICS

CERAMICS

Table 14.1: 4-Step Method for Ceramic

Sectioning	Precision Saw with 10LC or 20LC blade recommended for medium to soft ceramics or structural ceramics
Mounting	Castable, typically epoxy with flat edge filler if for very hard ceramic applications

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
UltraPrep Metal-Bonded	45µm Diamond water cooled	8 [36]	150	⬤ ⚙	Until Plane
Apex Hercules H	9µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ⚙	5:00
VerduTex	3µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ⚙	5:00
TexMet C	1µm MetaDi Supreme Diamond*	10 [44]	150	⬤ ⚙	5:00

⬤ = Platen ⚙ = Specimen Holder *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Grain Size, Porosity Assessment
Hardness Testing	Vickers

Table 14.2: 4-Step Method for Ceramic

Sectioning	Precision Saw with 10LC or 20LC blade recommended for medium to soft ceramics or structural ceramics
Mounting	Castable, typically epoxy with flat edge filler if for very hard ceramic applications

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex DGD Red	75µm Diamond water cooled	8 [36]	150	⬤ ⚙	Until Plane
TexMet P	15µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ⚙	6:00
VerduTex	6µm MetaDi Supreme Diamond*	6 [27]	150	⬤ ⚙	4:00
TexMet C	3µm MetaDi Supreme Diamond*, MasterMet Colloidal Silica (50:50 Simultaneously)	6 [27]	150	⬤ ⚙	4:00

⬤ = Platen ⚙ = Specimen Holder *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Grain Size, Porosity Assessment
Hardness Testing	Vickers

A mineral is comprised of solid substance with a defined chemical composition that formed naturally with the aid of varying geological conditions. A rock is a solid aggregate of minerals. Some of these mineral groups can be seen in Table 15.1.

There are two commonly used techniques for preparing geological specimens; thin section and bulk mount. Thin sections are typically prepared at 30μm for transmitted polarized light analysis for mineralogy and optical petrology. Bulk mounts are typically prepared for reflected light microscopy and is primarily used to examine opaque specimens that such as ore microscopy, coal and opaque minerals, see example in Figure 15.1.

Most geological specimens can be grouped into categories based on the variation in hardness and the water sensitivity. The most common scale of measurement is the Mohs hardness scale (Table 15.2). Which is defined by ten indicators (a mineral with a higher index scratches those below it). The scale ranges from talc to diamond.

Care should be taken when working with water sensitive minerals as they can eroded (soils), swell (clays), react (salts), accelerate reactions (cements) among other issues. Table 15.3 describes a generic preparation method for most non-water sensitive minerals of varying hardness. In case of water sensitive materials preparation would be similar with the replacement of water with either AutoMet Lapping Oil or glycol for the grinding steps. Diamonds paste (with Lapping Oil as extender) or Oil based suspensions can be used for the 9μm and 3μm polishing step. A final polish with MicroCloth and MicroPolish II 0.05μm alumina polishing powder combined with a 50:50 mixture of propylene glycol and alcohol is recommended.

Table 15.2: Mohs Hardness Scale

Mohs Hardness	Mineral	Chemical Formula
1	Talc	$Mg_3Si_4O_{10}(OH)_2$
2	Gypsum	$CaSO_4 \bullet 2H_2O$
3	Calcite	$CaCO_3$
4	Flurite	CaF_2
5	Apatite	$Ca_5(PO_4)_3(OH,Cl,F)$
6	Orthoclase	$KAlSi_3O_8$
7	Quartz	SiO_2
8	Topaz	$Al_2SiO_4(OH,F)_2$
9	Corundum	Al_2O_3
10	Diamond	C
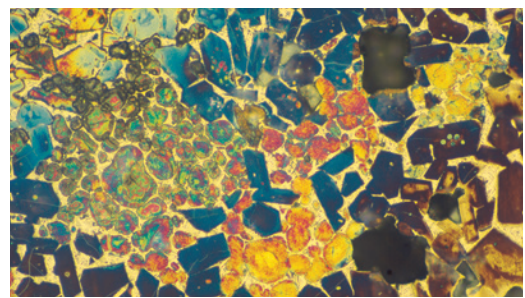
Figure 15.1. Microstructure of a cement clinker (Portland cement used for construction), showing various phases such as alite and belite.The specimen has been etched with 2% nital, ~260x. Reflected light.

Table 15.1: Minerals Groups

Minerals Group	Example
Sulfides	Cobaltite, Pyrite, Realgar
Oxides and Hydroxides	Microlite, Spinel
Halides	Cryolite, Fluorite, Halite, Prosopite
Nitrates, Carbonates, Borates	Magnesite, Malachite, Mother of Pearl, Rhodozite
Sulfates	Crocoite, Chromates Gypsum
Phosphates, Arsenates, Vanadates	Lazulite, Purpurite, Pyromorphite, Variscite, Zenotime
Silicates	Analcime, Smaragdite, Smoky Quartz Zektzerite, Zircon
Non-minerals	Amber, Coal, Opal, Pearl, Vulcanite

MINERALS

Table 15.3: Four Step Procedure for Preparing Bulk Mounts

Sectioning	Precision Saw with 15LC blade recommended for hard brittle materials
Mounting	Castable, typically EpoThin or EpoHeat

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex DGD	45µm Diamond water cooled	5 [22]	300	⬤ ⊛	3:00
TriDent	9µm MetaDi Supreme Diamond Suspension	3 [13]	150	⬤ ⊛	4:00
TriDent	3µm MetaDi Supreme Diamond Suspension	3 [13]	150	⬤ ⊛	4:00
TexMet	0.05µm MasterPrep Alumina Suspension	4 [18]	150	⬤ ⊛	2:00

⬤ = Platen ⊛ = Specimen Holder

Imaging & Analysis	Phase Area Percent
Hardness Testing	N/A

Glass

Glass preparation was historically prepared using silicon carbide followed by multiple steps of cerium oxide. The traditional preparation sequence (Table 15.4) can be used for a wide range of glass but the method can lead to the risk of edge chipping as the abrasive rolls along the leading edge of the unmounted sample. More recent advancements in fixed abrasive has led to the use of diamond grinding discs for glass preparation, such as in Table 15.5, as it has become critical for flat scratch free surfaces for testing including FTIR and interferometers, results shown in in Figure 15.2.

Figure 15.2. Quartz tube after 6µm polish.

Table 15.4: Traditional Procedure for Glass

Sectioning	Precision Saw with 15LC blade recommended for hard brittle materials
Mounting	Castable, typically EpoThin or EpoHeat

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled	5[22]	300	⬤ ⊛	Until Plane
CarbiMet	400 [P800] grit SiC water cooled	5[22]	300	⬤ ⊛	1:00
TexMet P	MiroMet (CeO) 1µm	5[22]	150	⬤ ⊛	5:00
MicroCloth	MiroMet (CeO) 1µm	5[22]	150	⬤ ⊛	2:00

⬤ = Platen ⊛ = Specimen Holder

Imaging & Analysis	Measurement & Analysis Applications
Hardness Testing	N/A

Table 15.5: Five Step Procedure for Glass

Sectioning	Precision Saw with 15LC blade recommended for glass
Mounting	Castable

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex DGD	45µm Diamond water cooled	5[22]	300	⬤ ✦	5:00
Apex DGD	15µm Diamond water cooled	5[22]	300	⬤ ✦	5:00
Apex DGD	3µm Diamond water cooled	5[22]	300	⬤ ✦	5:00
TexMet C	3µm MetaDi Supreme Diamond Suspension	5[22]	150	⬤ ✦	4:00
MasterTex	MasterPolish 2	5[22]	150	⬤ ✦	4:00

⬤ = Platen ✦ = Specimen Holder

Imaging & Analysis	Measurement & Analysis Applications
Hardness Testing	N/A

COMPOSITES

The diverse and highly adjustable characteristics of composite materials have caused a rapid increase in their use worldwide. From light weight golf clubs to high-performance airplane parts, reinforced composite materials are becoming more popular due to their unique physical properties and the capability to be manufactured readily into complex geometries. The term composites covers an extremely wide range of materials, generally grouped under the subcategories polymer-matrix composites (PMC), metal-matrix composites (MMC), and ceramic-matrix composites (CMC). Consequently, preparation schemes can be quite difficult to generalize.

The differences in hardness and grinding/polishing characteristics from this range of materials mean that relief control is one of the major concerns during preparation. Pull out and fracture problems are also very common. Sectioning frequently produces considerable damage that must be removed in subsequent preparation steps. Mounting with castable epoxy resin along with vacuum impregnation is frequently performed.

Polymer Matrix Composites

The most common analysis requirement for polymer matrix composites, as in Figure 16.1, is the determination of void content and fibre characteristics such as percentage area and size distribution. For all of these analyses, relief control is paramount for retaining accuracy. The investigation of failure mechanisms and microstructural analysis of features such as matrix modifiers and fillers can also be required.

There are many types of thermosetting and thermoplastic materials that are used as matrices in fibre-reinforced composites. The matrix can also contain dispersed phases known as modifiers that can range in size from tens of microns to nano scale. Materials used for the reinforcing phase include carbon, glass, plastic, natural fibres and boron.

Interface characteristics are important to the overall properties of these materials and examination of features such as inter-ply delamination are important to the understanding of failure mechanisms in many polymer matrix composites. Honeycomb and foam materials are also being incorporated into composite structures, to improve physical characteristics against weight – particularly important in the aerospace industry
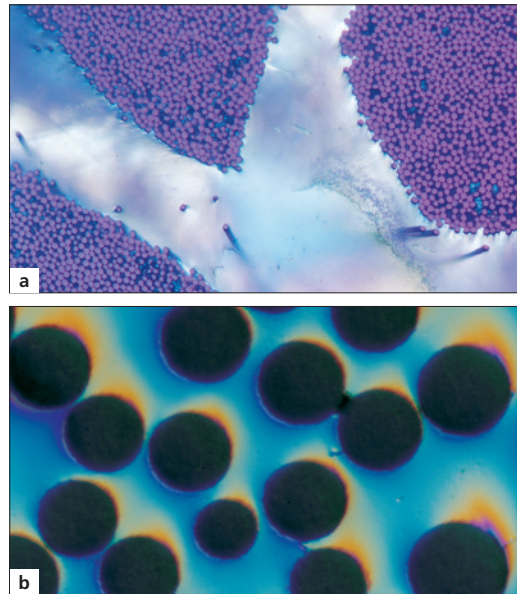
Figure 16.1. a) Microstructure of a polymer-matrix composite, graphite fabric-reinforced polysulfone (unetched, polarized light, 100X). b) Microstructure of alumina fibers in an aluminum - lithium alloy matrix (500X, as-polished, Nomarski DIC).

Table 16.1 shows a useful general approach for the preparation of many polymer matrix composites. However, some reinforcing materials – especially those that are smaller than ~10μm in size – can be prone to brittle fracture when diamond based preparation is used. In such cases, an alternative approach is to replace the 3μm diamond stage with 0.3μm alumina on a TexMet C cloth. For many analyses (such as void counting, fiber analysis and delamination examinations) this provides a suitable finish and limits possible relief effects. A shortened polishing stage can then be used to remove any remaining artifacts in the softer matrix material.
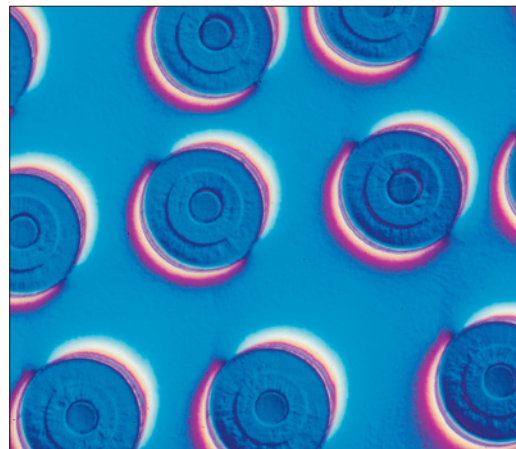
Figure 16.2. Microstructure of a metal-matrix composite, SiC in a CP titanium matrix (unetched, Nomarski DIC, 100X).

Table 16.1: 4-Step Method for Polymer-Matrix Composites					
Sectioning	Precision Saw with 15HC blade recommended for metal matrix composites				
Mounting	Castable, typically epoxy				
Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled	6 [27]	300	⏺ ✺	Until Plane
TexMet P	9µm MetaDi Supreme Diamond*	6 [27]	150	⏺ ✺	5:00
VerduTex	3µm MetaDi Supreme Diamond*	6 [27]	150	⏺ ✺	5:00
MicroCloth	0.05µm MasterPrep Alumina	6 [27]	150	⏺ ✺	1:30

⏺ = Platen ✺ = Specimen Holder *Plus MetaDi Fluid Extender as desired

Imaging & Analysis — Phase Area Percent, Porosity Assessment, OmniMet Object Measurements

Hardness Testing — N/A

Metal Matrix Composites

MMCs have been commercially available for many years. They consist of a metal matrix component, together with a reinforcing component that can be in various forms (e.g. particle or fibre). The metal matrix is generally formed from conventional light metal alloys (aluminium and titanium based) but can also be special alloys produced primarily for this type of application.

The analysis of metal matrix alloys includes both details of metallurgical microstructure and also interface properties. Examination of any chemical interactions and reactions between the matrix and the reinforcement component are instrumental in defining the characteristics of the material. As such, the dominant concerns during preparation of these materials will be the prevention of relief and the recovery of damage at interfaces during initial sectioning and grinding stages. Table 16.2 provides a good standard approach to the preparation of this material group. If the sectioning technique used is more damaging, or more material needs to be removed, then a more aggressive grinding stage may be preferred (for instance, replacing the ApexDGD Color disc with an UltraPrep disc).

Ceramic Matrix Composites

Ceramic Matrix Composites see Figure 16.3, typically consist of a ceramic matrix, such as alumina or silicon carbide, combined with a dispersed phase such as silicon carbide, zirconium oxide, aluminium nitride or titanium boride. In these systems, the
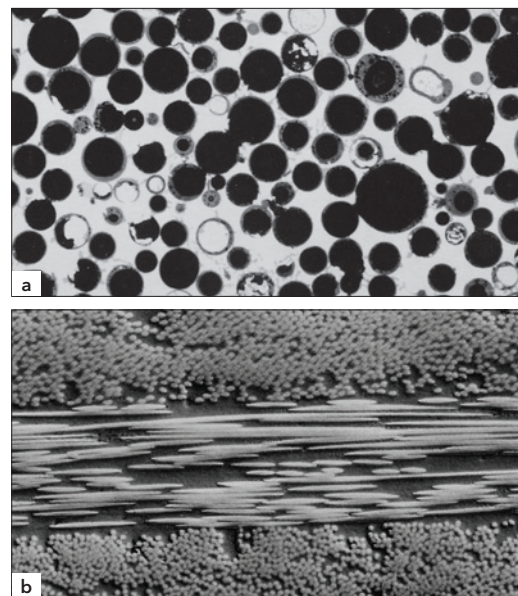
Figure 16.3. a) Synthetic foam made with hollow ceramic spheres added to 7075 aluminum (100X original magnification). Etched with aqueous 0.5% HF. b) Carbon-reinforced polymer composite (original at 100X). Specimen is as-polished and viewed with Nomarski DIC illumination.

preparation characteristics of the different phases do not necessarily lead to relief problems. The brittle nature of many of the component materials can lead to relatively deep damage if inappropriate sectioning or grinding techniques are used. Combined with the high wear resistance of these materials, recovery of such damage can be very slow. The primary concern is normally, therefore, to section as closely as possible to the area of interest, with the lowest

COMPOSITES

Table 16.2: 4-Step Method for Metal-Matrix Composites

Sectioning	Precision Saw with 15HC blade recommended for metal matrix composites
Mounting	Castable, typically epoxy

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
Apex DGD Yellow	35µm Diamond water cooled	5 [22]	300		Until Plane
UltraPad	9µm MetaDi Supreme Diamond*	5 [22]	150		4:00
VerduTex	3µm MetaDi Supreme Diamond*	6 [27]	150		3:00
ChemoMet	0.02 - 0.06µm Master-Met Colloidal Silica	6 [27]	150		1:30

● = Platen   ✪ = Specimen Holder   *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Fiber density, Phase Area Percent, OmniMet Object Measurements
Hardness Testing	N/A

Table 16.3: 4-Step Method for Ceramic-Matrix Composites

Sectioning	Precision Saw with 15HC blade recommended for metal matrix composites
Mounting	Castable, typically epoxy

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
UltraPrep	45µm Diamond water cooled	6 [27]	300		Until Plane
UltraPad	15µm MetaDi Supreme Diamond*	6 [27]	150		4:00
VerduTex	6µm MetaDi Supreme Diamond*	6 [27]	150		3:00
VerduTex	1µm MetaDi Supreme Diamond*	6 [27]	150		2:00

● = Platen   ✪ = Specimen Holder   *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Phase Area Percent, Pororsity Assesment, OmniMet Object Measurements
Hardness Testing	N/A

damage method available (Table 16.3), usually using a diamond blade on a precision saw. An alternative to the UltraPrep metal bonded disc would be the use of the Apex DGD fixed diamond disc with metadi fluid as a lubricant. As with pure ceramics, the addition of MasterMet colloidal silica in even proportion with the diamond slurry in the final stage can enhance the finish.

HELPFUL HINTS FOR COMPOSITES

Sectioning damage can propagate down the fibers and can be difficult to remove. Prevent damage by using precision saw sectioning and diamond wafering blades with a very fine abrasive, specifically like 5LC and 10LC Series. Thinner blades produce less damage. Section as close as possible to the plane of interest.

Composites can be damaged by compression mounting. Castable mounting resins with low peak temperature are preferred.

Avoid aggressive grinding steps by using longer times and finer abrasives.

PRINTED WIRING BOARDS

The vast majority of printed wiring boards (PWB's) are of a rigid variety, the bulk of which are composed of layers of woven glass fiber cloth in a polymeric matrix. Flex circuits, which are becoming quite common, do not typically contain glass fiber, but instead, their bulk is often composed of layers of polyimide. The circuitry in both types of boards is composed of plated and/or foil metal. The metal used is generally copper, while in a few cases, gold and/or nickel plating may be present, as shown in figure 17.1. Furthermore, depending upon whether the boards have undergone assembly or shock testing, solders of various compositions might also be present.

Luckily for the metallographer, the variety of materials present in PWB's generally do not complicate the preparation methods due to the fact that extremely hard and brittle materials are not commonly found in the boards. This changes, however, when 'packed' boards with ceramic or semiconductor components must be sectioned. Please refer to
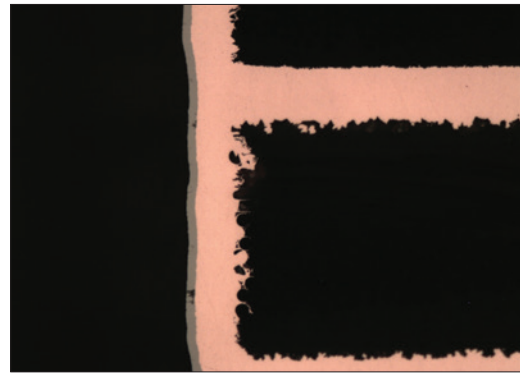
Figure 17.1. Nickel plating on the Plated through Hole of the Back Panel PWB, 500x

Table Table 17.1: 5-Step Method for Printed Wiring Boards (target preparation)					
Sectioning	Precision Saw with 15HC blade recommended for PWBs				
Mounting	Castable, typically a fast cure system like SamplKwick				
Surface	**Abrasive / Size**	**Load - lbs [N] / Specimen**	**Base Speed [rpm]**	**Relative Rotation**	**Time [min:sec]**
CarbiMet	320 [P400] grit SiC water cooled	3 [13]	150	⊙⊛	Hit edge of the target
CarbiMet	600 [P1200] grit SiC water cooled	3 [13]	150	⊙⊛	Hit the level of interest
TriDent	9µm MetaDi Supreme Diamond*	5 [22]	150	⊙⊛	3:00
TriDent	3µm MetaDi Supreme Diamond*	5 [22]	150	⊙⊛	3:00
ChemoMet	0.05µm MasterPrep Alumina + 0.06µm MasterMet Colloidal Silica (50:50)	4 [18]	150	⊙⊛	1:15

● = Platen   ⊛ = Specimen Holder   *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Manual Interactive Thickness, Porosity Assessment
Hardness Testing	Knoop

methods of preparation for ceramics or microelec-tronic materials for information on these cases. A generalized method for non-packed PWB's of both the rigid and flex varieties is presented in Table 17.1. A 1μm step may be added if needed. This method is best used to perform targeted grind-ing on multiple specimens using semi-automic equipment.

Quality control of manufactured PWB's often demands statistical analysis based on plating thickness measurements taken from the centers of plated through-holes. However, to generate enough data for a representative sampling, nu-merous specimens must be prepared. This is easiest when coupons are taken from PWB's and are ganged together in precise alignment. In this man-ner, the through holes in all of the coupons can be sectioned at one time, with one set of consumables. The PC-Met Precision High Volume Printed Wiring Board and the PWB Met Small Hole Accessories allow this type of atuomation.

HELPFUL HINTS FOR PWBS

To improve penetration of acrylic mounting material into the through-holes of a PWB specimen, dip the "pinned" PWB coupons in liquid hardener immediately prior to mounting in acrylic. This will "wick" the acrylic into the through-holes and minimize the presence of problematic bubbles in these critical locations.

ELECTRONIC MATERIALS

The term, 'electronic materials' encompasses an extremely wide range of materials. This is due to the fact that most microelectronic devices are composites, containing any number of individual components. For example, present day microprocessor failure analysis might require the metallographer to precisely cross section through a silicon chip plated with multiple thin-film layers of oxides, polymers, ductile metals such as copper or aluminum, and refractory metals such as tungsten and/or titanium-tungsten. In addition, the packaging of such a device might contain materials of such varying mechanical properties as toughened aluminum oxide and solder. The solder materials may have compositions ranging up to 97% lead. With such a vast number of materials incorporated into a single device, and with these materials having such highly disparate mechanical properties, it is virtually impossible to develop a general method for achieving perfect metallographic results. Instead,
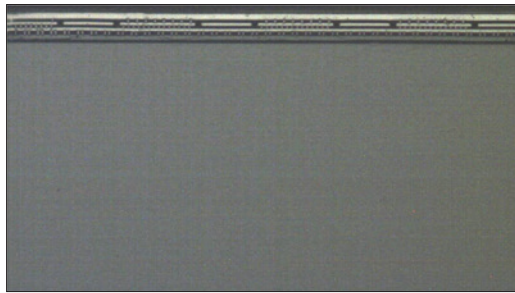

Figure 18.1. Aluminum circuitry on silicon substrate Heatsink Small Outline Package (HSOP), 500x

we must focus on a few individual materials, and develop a philosophy of preparation in which we give our attention specifically to the materials of interest.

First and foremost in the class of electronic materials is silicon. Silicon is a relatively hard, brittle material, which does not respond well to grinding with large silicon carbide abrasives. Silicon carbide papers contain strongly bonded abrasive particles which, when they collide with the leading edge of a piece of silicon, create significant impact damage. In addition, they create tensile stresses on the trailing edge of silicon, which results in deep and destructive cracking. Cutting close to the target area is preferable to grinding, but to accurately approach the target area within a silicon device, fine grinding is still necessary. This is the point at which silicon preparation divides into two distinct classes. The first is preparation through traditional metallographic techniques. The second is preparation of unencapsulated silicon chips (or die), using special fixturing and abrasives. This second category is a specialized field of metallography, which is discussed in other texts [8] and is beyond the scope of this book. Therefore, we will focus on standard metallographic techniques throughout this discussion.

Standard preparation of epoxy encapsulated silicon is similar to general metallographic preparation, with the exception that only very fine grades of silicon carbide paper are used.

Table 18.1: 5-Step Procedure for Preparing Silicon in Microelectronic Devices					
Sectioning	Precision Saw with blade recommeded for microelectronic devices				
Mounting	Castable, typically EpoThin				
Surface	**Abrasive / Size**	**Load - lbs [N] / Specimen**	**Base Speed [rpm]**	**Relative Rotation**	**Time [min:sec]**
CarbiMet	600 [P1200] grit SiC water cooled	3 [13]	100	⚫🔘	Until Plane
VerduTex	6µm MetaDi Supreme Diamond*	5 [22]	100	⚫🔘	3:00
VerduTex	3µm MetaDi Supreme Diamond*	5 [22]	100	⚫🔘	3:00
VerduTex	1µm MetaDi Supreme Diamond*	5 [22]	100	⚫🔘	3:00
ChemoMet	0.06µm MasterMet Colloidal Silica	2 [9]	100	⚫🔘	2:00
⚫ = Platen 🔘 = Specimen Holder *Plus MetaDi Fluid Extender as desired					
Imaging & Analysis	Measurement & Analysis Applications, Manual Interactive Thickness				
Hardness Testing	N/A				

Table 18.1 illustrates a typical process of preparation with results shown in Figure 18.1.

Using silicon carbide grinding paper with abrasive sizes coarser than 600 [P1200] grit may cause serious damage to the silicon. Therefore, this method often requires precision cutting or cleaving near to the target area of the specimen prior to the initial grinding step. Care must be taken to assure that only precision saw blades designed especially for microelectronic materials are used when cutting.

Silicon responds very well to the chemomechanical polishing effects of colloidal silica, but not as well to aluminum oxide. However, there are instances when an aluminum oxide product, such as the MasterPrep suspension, should be used for final polishing. This relates back to the philosophy of preparation mentioned earlier. An example of the appropriate
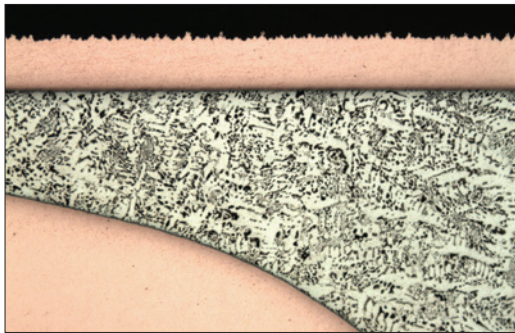

Figure 18.2. Microsection of the Pb Solder Connection, 200x

use of MasterPrep alumina would be: when a silicon die is attached to a lead frame material such as nickel-plated copper, and the lead frame and die attach materials are the materials of interest. In this case, we are not so concerned with the surface finish of the silicon, but instead, we want to be sure that the nickel doesn't smear, and that we are able to discern the true microstructure of the lead frame materials (refer to the preparation methods for copper and nickel on pages 47-48).

When preparing a silicon device for the purpose of inspecting the metalized, thin film circuitry, the techniques would be the same as those listed above. Again, the choice of final polishing agent would be determined entirely by the features of interest. For instance, aluminum circuitry responds extremely well to the chemomechanical effects of colloidal silica, but tungsten and titanium-tungsten are not as effectively polished with colloidal silica as are the materials by which they are surrounded. Therefore, colloidal silica often causes such refractory metals to stand in relief against a well polished background. This results in edge rounding of the refractory metals and can make interface analysis quite difficult. An alternative is to use an extremely fine diamond suspension for final polishing in order to reduce these effects.

When lead alloy solders are the materials of interest in a microelectronic package (Figure 18.2), we can

Table 18.2: 5-Step Procedure for General Electronic Components					
Sectioning	Precision Saw with blade recommeded for microelectronic devices				
Mounting	Castable, typically EpoThin				
Surface	**Abrasive / Size**	**Load - lbs [N] / Specimen**	**Base Speed [rpm]**	**Relative Rotation**	**Time [min:sec]**
CarbiMet	320 [P400] grit SiC water cooled	3 [13]	150	◉ ◉	Hit the edge of the target
TexMet P	9µm MetaDi Supreme Diamond*	5 [22]	150	◉ ◉	5:00
VerduTex	3µm MetaDi Supreme Diamond*	5 [22]	150	◉ ◉	3:00
VerduTex	1µm MetaDi Supreme Diamond*	5 [22]	150	◉ ◉	3:00
ChemoMet	0.05µm MasterPrep Alumina	3 [13]	150	◉ ◉	1:30

● = Platen ❀ = Specimen Holder   *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Measurement & Analysis Applications, Coating Layer Thickness
Hardness Testing	N/A

often separate them into two general categories. The first is the eutectic, or near-eutectic solders. These tend to be ductile, but they respond well to the same general preparation listed in Table 18.2. During the 3µm polishing step, the best results are obtained by using diamond paste.

Pastes contain a waxy base that reduces diamond

Figure 18.3. Pb-Free Solder in Plated Through Hole, 200x.

embedding in ductile metals. Limit the use of extenders in such cases. A few drops of water lubrication does not break down the waxy base, and therefore it retains the desired properties of diamond paste.

The second category of lead solders is the high temperature solders (in the range of 90% to 97% lead). These are difficult to prepare, and require special consideration. This is especially true when they are incorporated into a device that contains a ceramic package. The mechanical properties of these toughened ceramics require aggressive grinding techniques, which cause ceramic debris (and often, the abrasive used for the grinding operation) to became embedded in the solder. Silicon carbide abrasives are a poor choice for grinding in such cases, as silicon carbide is only slightly harder (and significantly more brittle) than the typical packaging material they are intended to grind. As the silicon carbide abrasive fractures, it produces elongated shards, which embed deeply into high-temperature solders. In addition, they do not grind the ceramic package effectively, and therefore produce extreme edge rounding at the interface with the solder. Diamond grinding produces a more desired result since diamond abrasives are blockier in shape, and are therefore easier to remove after they have embedded themselves. In addition, they are capable of creating a flat surface when ceramic packages are present.

High removal rates and good surface finishes have been obtained on ceramic packages when using diamond paste on the Apex Hercules S discs.

Polishing of high-temperature solders attached to ceramics often produces undesired edge rounding. However, this cannot be completely avoided. The cushioning effects of polishing pads will tend to cause the abrasive to preferentially remove material from ductile materials faster than from hard, brittle materials. By using stiff, flat polishing surfaces, one can reduce this effect, but at the expense of having additional material embedded in the high-temperature solder. One effective preparation technique is to perform a grind-polish-grind-polish type of procedure. In this technique, a polishing step, which utilizes a napped polishing cloth, is used in conjunction with a step-down in abrasive size from the previous grinding step. This process is used to polish out the embedded material. The next step is a grinding process, utilizing another step-down in abrasive size, to flatten the specimen again. This continues to a sufficiently fine polishing stage at which final polishing will produce the desired result. Table 18.2 illustrates an example of such a process.

HELPFUL HINTS FOR ELECTRONIC MATERIALS

Consider what area of the specimen is most critical when choosing a final polishing suspension. For example, the chemomechanical nature of colloidal silicas (such as MasterMet and MasterMet 2) make them ideal for polishing silicon, glass, oxides, aluminum, and to some degree copper. However, polishing nickel platings and gold ball bonds with these agents often results in smearing. For such non-reactive materials, use MasterPrep aluminum oxide final polishing suspension to produce a flat, scratch-free surface.

POLYMERS

Plastics and polymers are normally quite soft. Many different sectioning methods have been used. A sharp razor blade or scalpel, or even a pair of scissors, can be used. Microtomes have been used, and a surface cut after refrigeration in liquid nitrogen requires very little subsequent preparation. A jeweler's saw may be used, although the surface is somewhat rough. The precision saw produces excellent surfaces, while an abrasive cut-off saw yields a bit more damage and a rougher surface. Blades and wheels for sectioning polymers are available from Buehler. Damage from sectioning is quite easy to remove.

Surface quality can be degraded by abrasion from the debris produced during grinding and polishing.
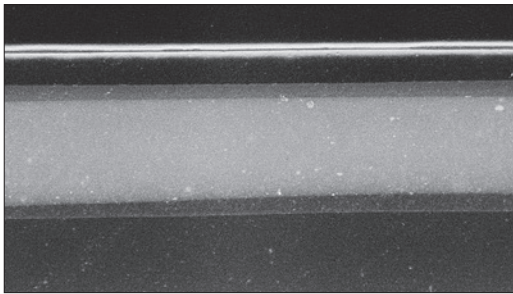
Mounted specimens are much easier to prepare than nonmounted specimens. Castable resins are preferred as the heat from a mounting press may damage or alter the structure of the specimen. However, there may be a visibility problem if a transparent, clear polymeric specimen is mounted in a clear, transparent epoxy. In this case, EpoColor resin, with its deep red color, will produce excellent color contrast between specimen and mount in darkfield or polarized light. Due to their light weight, a specimen may float in the epoxy filled mold. To prevent this, put double sided adhesive tape on the bottom of the mold cup and set the specimen on top of the tape. Always use practices that minimize the exotherm during polymerization.

Figure 19.1. Micrograph showing multi-layered polymer meat casing mounted in epoxy (dark field, 100X).
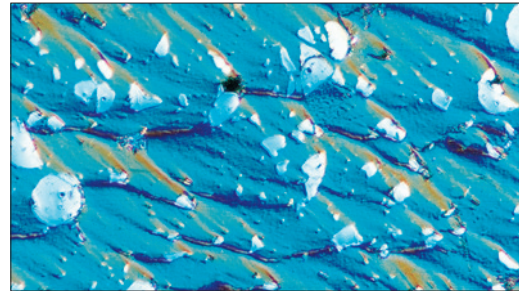
Figure 19.2. Micrograph of high-density polyethylene containing a filler (white particles) (unetched, Nomarski DIC, 100X).

Table 19.1: 6-Step Method for Polymers

Sectioning	Precision Saw with 30HC blade recommnded for polymers				
Mounting	Castable, typically EpoThin				

Surface	Abrasive / Size	Load - lbs [N] / Specimen	Base Speed [rpm]	Relative Rotation	Time [min:sec]
CarbiMet	320 [P400] grit SiC water cooled	4 [18]	300	◓◍	Until Plane
CarbiMet	400 [P600] grit SiC water cooled	4 [18]	300	◓◍	1:00
CarbiMet	600 [P1200] grit SiC water cooled	4 [18]	300	◓◍	1:00
CarbiMet	1200 [P2500] grit SiC water cooled	4 [18]	300	◓◍	1:00
TexMet C	3μm MetaDi II Diamond Paste*	5 [22]	150	◓◍	4:00
MasterTex	0.05μm MasterPrep Alumina	3 [13]	150	◓◍	3:00

● = Platen   ◍ = Specimen Holder   *Plus MetaDi Fluid Extender as desired

Imaging & Analysis	Manual Interactive Thickness
Hardness Testing	N/A

Preparation of plastics and polymers for microstructural examination follows the same basic principles as for other materials. Automated polishing devices are preferred to manual preparation. Rough grinding abrasives are unnecessary, even for the planar grinding step. Pressures should be lighter than used for most metals.

Water is generally used as the coolant, although some plastics and polymers may react with water. In such cases, use a fluid that will not react with the particular plastic or polymer. Embedding can be a problem with plastics and polymers. ASTM E 2015 (Standard Guide for Preparation of Plastics and Polymeric Specimens for Microstructural Examination) describes procedures for preparing several types of plastics and polymers. Table 19.1 contains a generic procedure for preparing many plastic and polymeric materials, see the results in Figures 19.1 and 19.2. Depending upon the material and the surface roughness after sectioning, it may be possible to start grinding with 400 [P800] grit or even 600 [P1200] grit SiC paper.

If flatness is critical, the final step can be altered. Use a TexMet cloth with MasterPrep alumina abrasive slurry running in contra rotation mode at 120-150rpm for 3 minutes.

HELPFUL HINTS FOR POLYMERS

Plastics often have little contrast from the mounting media when viewed under a microscope. This makes thickness measurement and discerning edges difficult. Mounting in EpoColor dye enhanced epoxy will resolve this situation by producing excellent color contrast between the specimens and mount.

Polymers and plastics may react with fluids during sectioning and preparation. Always check your particular polymer and choose the proper fluid, usually a water, or oil based solution to avoid a reaction.

ETCHING

Metallographic etching encompasses all processes used to reveal particular structural characteristics of a metal that are not evident in the as-polished condition. Examination of a properly polished specimen before etching may reveal structural aspects such as porosity, cracks, and nonmetallic inclusions. Indeed, certain constituents are best measured by image analysis without etching, because etching will reveal additional, unwanted detail and make detection difficult or impossible. The classic examples are the measurement of inclusions in steels and graphite in cast iron. Of course, inclusions are present in all metals, not just steels. Many intermetallic precipitates and nitrides can be measured effectively in the as-polished condition.

In certain nonferrous alloys that have non-cubic crystallographic structures (such as beryllium, hafnium, magnesium, titanium, uranium and zirconium), grain size can be revealed adequately in the as polished condition using polarized light. Figure 20.1 shows the microstructure of cold-drawn zirconium viewed in cross-polarized light. This produces grain coloration, rather than a "flat etched" appearance where only the grain boundaries are dark.
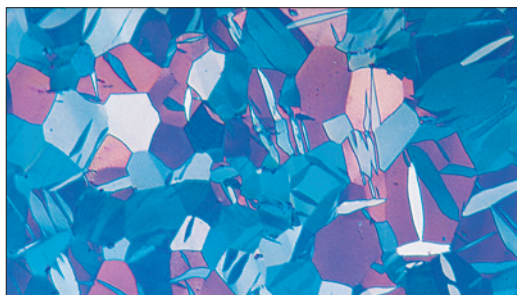
Etching Procedures

Figure 20.1. Mechanical twins at the surface of hot worked and cold drawn high-purity zirconium viewed with polarized light (200X).

Microscopic examination is usually limited to a maximum magnification of 1000X — the approximate useful limit of the light microscope, unless oil immersion objectives are used. Many image analysis systems use relay lenses that yield higher screen magnifications that may make detection of fine structures easier. However, resolution is not improved beyond the limit of 0.2-0.3µm for the light microscope. Microscopic examination of a properly prepared specimen will clearly reveal structural characteristics such as grain size, segregation, and the shape, size, and distribution of the phases and inclusions that are present. Examination of the microstructure will reveal prior mechanical and thermal treatments give the metal. Many of these microstructural features are measured either according to established image analysis procedures, e.g., ASTM standards, or internally developed methods.

Etching is done by immersion or by swabbing (or electrolytically) with a suitable chemical solution that essentially produces selective corrosion. Swabbing is preferred for those metals and alloys that form a tenacious oxide surface layer with atmospheric exposure such as stainless steels, aluminum, nickel, niobium, and titanium and their alloys. It is best to use surgical grade cotton that will not scratch the polished surface. Etch time varies with etch strength and can only be determined by experience. In general, for high magnification examination the etch depth should be shallow; while for low magnification examination a deeper etch yields better image contrast. Some etchants produce selective results in that only one phase will be attacked or colored. A vast number of etchants have been developed; the reader is directed to references 1-3, 9 and ASTM E 407. Pages 82-87 list some of the most commonly used etchants for the materials described in this book.

Etchants that reveal grain boundaries are very important for successful determination of the grain size. Grain boundary etchants are given in [1-3, 9]. Problems associated with grain boundary etching, particularly prior austenite grain boundary etching, are given in [2, 10 and 11]. Measurement of grain size in austenitic or face-centered cubic metals that exhibit annealing twins is a commonly encountered problem. Etchants that will reveal grain boundaries, but not twin boundaries, are reviewed in [2].

Selective Etching

Image analysis work is facilitated if the etchant chosen improves the contrast between the feature of interest and everything else. Thousands of etchants have been developed over the years, but only a small number of these are selective in nature. Although the selection of the best etchant, and its proper use, is a very critical phase of the image analysis process, only a few publications have addressed this problem [12-14]. Selective etchants, that is, etchants that preferentially attack or color a specific phase, are listed in

[1-3, 9, 13 and 14] and illustrated in 13 and 14. Stansbury [15] has described how potentiostatic etching works and has listed many preferential potentiostatic etching methods. The potentiostat offers the ultimate in control over the etching process and is an outstanding tool for this purpose. Many tint etchants act selectively in that they color either the anodic or cathodic constituent in a microstructure. Tint etchants are listed and illustrated in several publications [1-3, 16-21].

A classic example of the different behavior of etchants is given in Figure 20.2 where low-carbon sheet steel has been etched with the standard nital and picral etchants, and also a color tint etch. Etching with 2% nital reveals the ferrite grain boundaries and cementite. Note that many of the ferrite grain boundaries are missing or quite faint; a problem that degrades the accuracy of grain size ratings. Etching with 4% picral reveals the cementite aggregates (one could not call this pearlite as it is too nonlamellar in appearance and some of the cementite

exists as simple grain boundary films) but no ferrite grain boundaries. If one is interested in the amount and nature of the cementite (which can influence formability), then the picral etch is far superior to the nital etch as picral revealed only the cementite. Tint etching with Beraha's solution (Klemm's I could also be used) colored the grains according to their crystallographic orientation. With the development of color image analyzers, this image can now be used quite effectively to provide accurate grain size measurements since all of the grains are colored.

Figure 20.3 shows a somewhat more complex example of selective etching. The micrograph shows the ferrite-cementite-iron phosphide ternary eutectic in gray iron. Etching sequentially with picral and nital revealed the eutectic, Figure 20.3a, surrounded by pearlite. Etching with boiling alkaline sodium picrate, Figure 20.3b, colored the cementite phase only, including in the surrounding pearlite (a higher magnification is required to see the very finely spaced cementite that is more lightly
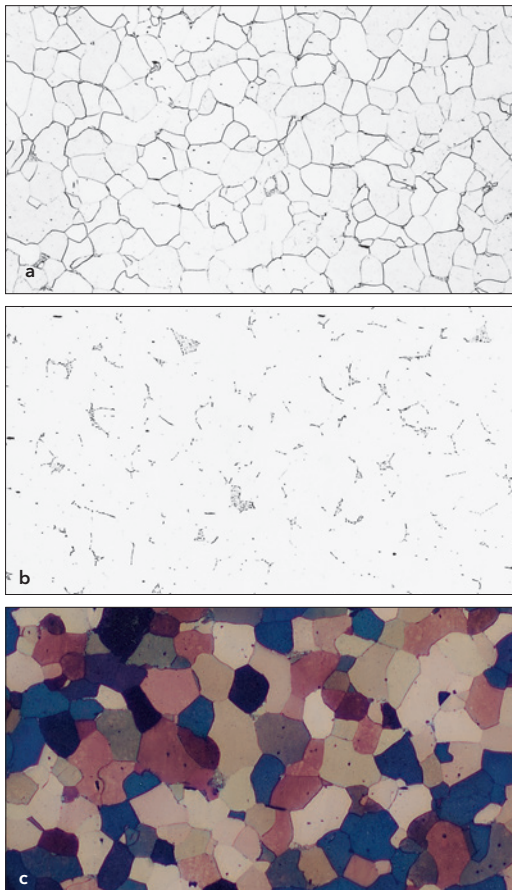
Figure 20.2. Microstructure of low-carbon sheet steel etched with (a, top) 2% nital, (b, middle) 4% picral; and (c, bottom) Beraha's reagent (100mℓ water, 10g $Na_2S_2O_3$ and 3g $K_2S_2O_5$) at 100X.
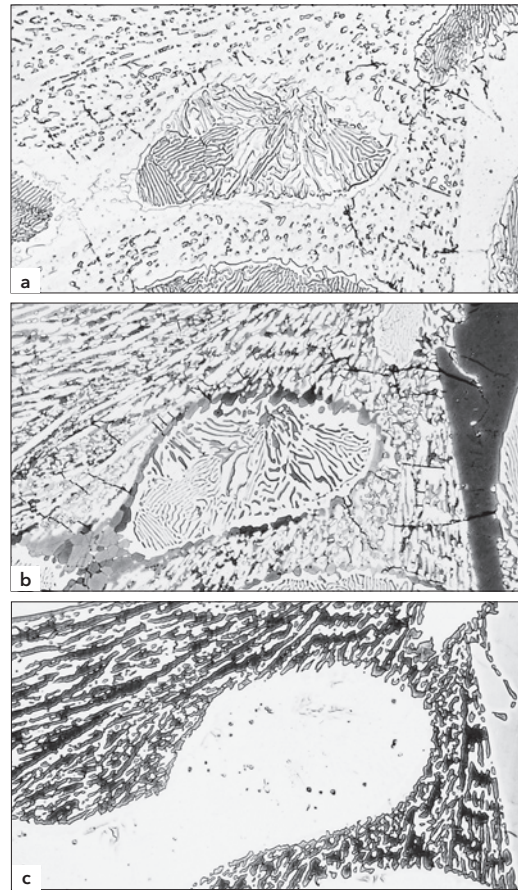

Figure 20.3. The ternary eutectic (α-Fe_3C-Fe_3P) in gray cast iron revealed by etching a) in picral and nital to "outline" the phases b) boiling alkaline sodium picrate to color the cementite. c) boiling Murakami's reagent to color the phosphide (200X).

colored). Etching with boiling Murakami's reagent, Figure 20.3c, colors the iron phosphide darkly and will lightly color the cementite after prolonged etching. The ferrite could be colored preferentially if Klemm's I was used.

Selective etching has been commonly applied to stainless steels for detection, identification and measurement of delta ferrite, ferrite in dual phase grades, and sigma phase. Figure 20.4 shows examples of the use of a number of popular etchants to reveal the microstructure of a dual phase stainless steel in the hot rolled and annealed condition. Figure 20.4a shows a well delineated structure when the specimen was immersed in ethanolic 15% HCl for 30 minutes. All of the phase boundaries are clearly revealed, but there is no discrimination between ferrite and austenite. Twin boundaries in the austenite are not revealed. Glyceregia, a etchant for stainless steels, was not suitable for this grade, Figure 20.4b, as it appears to be rather orientation sensitive. Many electrolytic etchants have been used for etching stainless steels, but only a few have se-

lective characteristics. Of the four shown in Figures 20.4c to f, only aqueous 60% nitric acid produced any gray level discrimination between the phases, and that was weak. All revealed the phase boundaries nicely, however. Two electrolytic reagents, shown in Figures 20.4g and h, are commonly used to color ferrite in dual phase grades and delta ferrite in martensitic grades. Of these, aqueous 20% sodium hydroxide, Figure 20.4g, usually gives more uniform coloring of the ferrite. Murakami's and Groesbeck's reagents have also been used for this purpose. Tint etchants have been developed by Beraha that color the ferrite phase nicely, as demonstrated in Figure 20.4i.

Selective etching techniques are not limited to iron-based alloys, although these have more thoroughly developed than for any other alloy system. Selective etching of beta phase in alpha-beta copper alloys has been a popular subject. Figure 20.5 illustrates coloring of beta phase in Naval Brass (UNS 46400) using Klemm's I reagent. Selective etching has a long historical record for identification of interme-
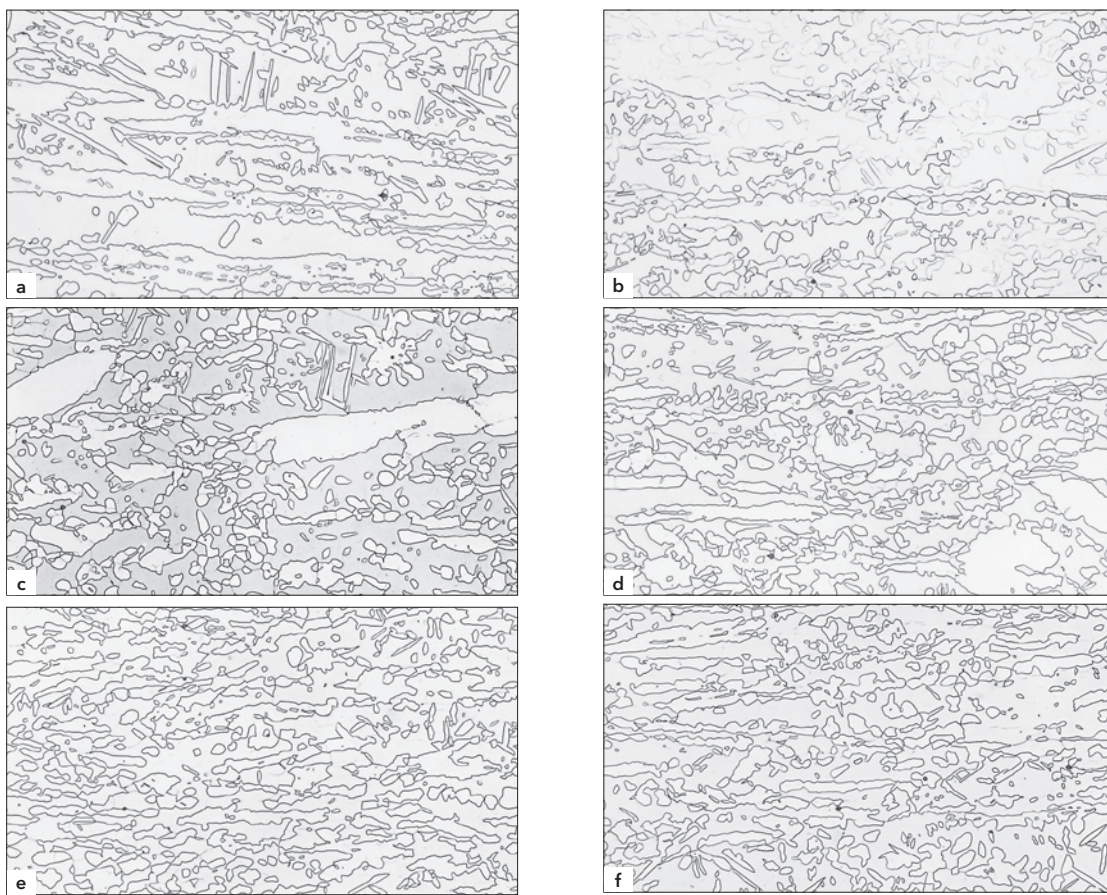
Figure 20.4. Microstructure of a duplex stainless steel revealed using a) alcoholic 15% HCl by immersion (30 min.); and, with b) glyceregia by swabbing (2 min.) c) aqueous 60% HNO$_3$ (1 V DC, 20 seconds); with d) aqueous 10% oxalic acid (6 V DC, 75 seconds); with e) aqueous 10% CrO$_3$ (6 V DC, 30 seconds); and, with (f) aqueous 2% H$_2$SO$_4$ (5 V DC, 30 seconds) at 200X.
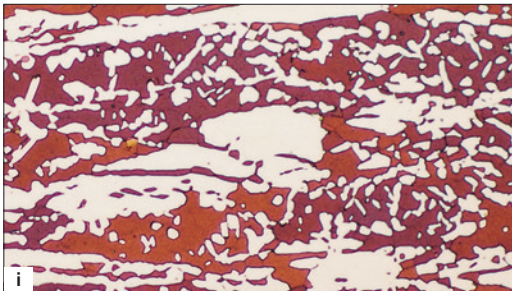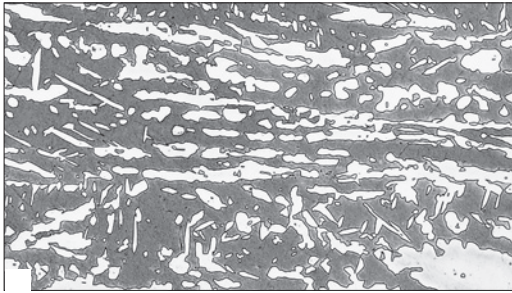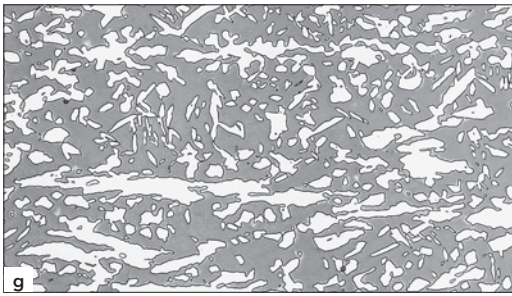
g

i

Figure 20.4. Selective coloring of ferrite in a duplex stainless steel by electrolytic etching with g) aqueous 20% NaOH (4 V DC, 10 seconds) and with h) aqueous 10 N KOH, (3 V DC, 4 seconds); and by immersion color etching i) with Beraha's reagent (100mℓ water, 10mℓ HCl and 1g $K_2S_2O_3$) at 200X.

tallic phases in aluminum alloys. This method was used for many years before the development of energy-dispersive spectroscopy. Today, it is still useful for image analysis work. Figure 20.6 shows selective coloration of theta phase, $CuAl_2$, in the Al-33% Cu eutectic alloy. As a final example, Figure 20.7 illustrates the structure of a simple WC-Co sintered carbide, cutting tool. In the as-polished condition, Figure 20.7a, the cobalt binder can be seen faintly against the more grayish tungsten carbide grains. A few particles of graphite are visible. In Figure 20.7b, light relief polishing has brought out the outlines of the cobalt binder phase, but this image is not particularly useful for image analysis. Etching in a solution of hydrochloric acid saturated with ferric chloride, Figure 20.7c, attacks the cobalt and provides good uniform contrast for measurement of the cobalt binder phase. Following this with Murakami's reagent at room temperature reveals the edges of the tungsten carbide grains, useful for evaluation of the WC grain size, Figure 20.7d.
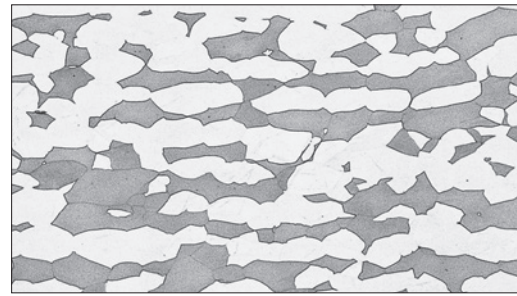
Figure 20.5. Beta phase colored in Naval Brass (Cu-39.7% Zn-0.8% Sn) by immersion color etching with Klemm's I reagent (200X).

Electrolytic Etching and Anodizing

The procedure for electrolytic etching is basically the same as for electropolishing except that voltage and current densities are considerably lower. The specimen is made the anode, and some relatively insoluble but conductive material such as stainless steel, graphite, or platinum is used for the cathode. Direct current electrolysis is used for most electrolytic etching. Electrolytic etching is commonly used with stainless steels, either to reveal grain boundaries without twin boundaries, or for coloring ferrite (as illustrated) in Figure 20.4i, delta ferrite, sigma or chi phases. Anodizing is a term applied to electrolytic etchants that develop grain coloration when viewed with crossed-polarized light, as in the case of aluminum, tantalum, titanium, tungsten, uranium, vanadium and zirconium [2]. Figure 20.8 shows the grain structure of 5754 aluminum revealed by anodizing with Barker's reagent, viewed with crossed-polarized light. Again, color image analysis makes this image useful now for grain size measurements.
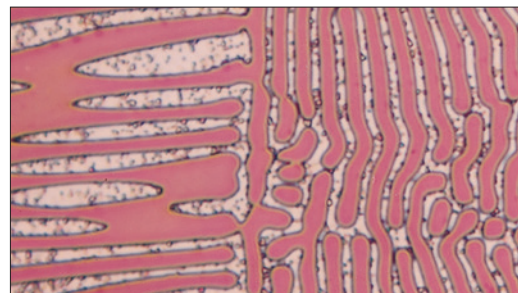
Figure 20.6. Theta phase, $CuAl_2$ colored in the α-Al/$CuAl_2$ eutectic in an as-cast Al-33% Cu specimen by immersion color etching with the Lienard and Pacque etch (200mℓ water, 1g ammonium molybdate, 6g ammonium chloride) at 1000X.

Heat Tinting

Although not commonly utilized, heat tinting [2] is an excellent method for obtaining color contrast between constituents or grains. An

ETCHING

a

b

c

d

Figure 20.7. Microstructure of WC-Co cutting tool: a) as-polished revealing graphite particles; b) relief polished revealing the cobalt binder phase; c) after immersion in Chaporova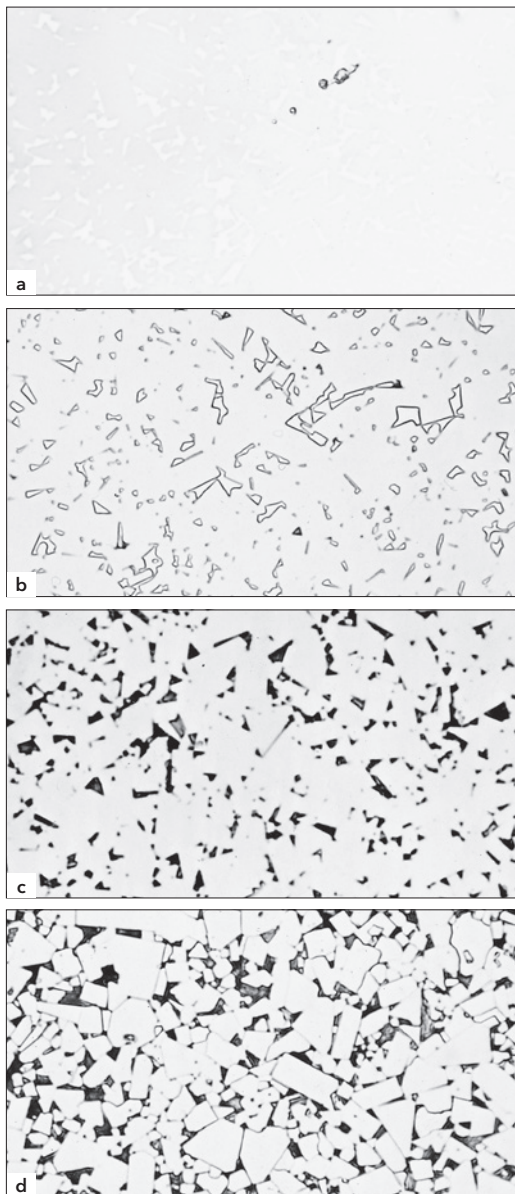's etch (HCl saturated with FeCl₃) to attack and "darken" the cobalt; and, d) after (c) plus Murakami's reagent to outline the WC grains (1000X).

unmounted polished specimen is placed face up in an air-fired furnace at a set temperature and held there as an oxide film grows on the surface. Interference effects, as in tint etching, create coloration for film thicknesses within a certain range, about 30-500nm. The observed color is a function of the function of the film thickness. Naturally, the thermal exposure cannot alter the microstructure. The correct temperature must be determined by the trial-and-error approach, but the procedure is reproducible and reliable. Figure 20.9 shows the grain structure of CP titanium revealed by heat tinting.
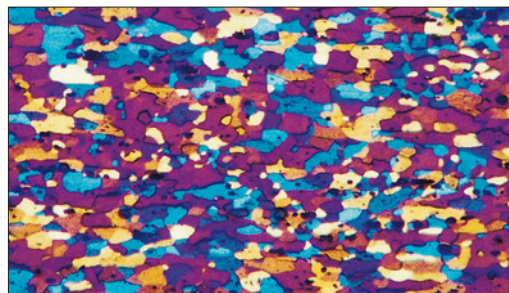
Figure 20.8. Grain structure of annealed 5754 aluminum sheet revealed by anodizing with Barker's reagent (30 V DC, 2 min.) and viewing with polarized light plus sensitive tint (100X).

Interference Layer Method

The interference layer method [2], introduced by Pepperhoff in 1960, is another procedure for obtaining a film over the microstructure that generates color by interference effects. In this method, a suitable material is deposited on the polished specimen face by vapor deposition to produce a low-absorption, dielectric film with a high refractive index at a thickness within the range for interference. Very small differences in the natural reflectivity between constituents and the matrix can be dramatically enhanced by this method. Suitable materials for the production of evaporation layers have been summarized in [22,23]. The technique is universally applicable, but does require a vacuum evaporator. Its main weakness is difficulty in obtaining a uniformly coated large surface area for image analysis measurements.

Figure 20.9. Grain structure of annealed CP titanium revealed by heat tinting on a hot plate (100X, polarized light plus sensitive tint).

See Tables 20.1 – 20.13 for a list of the most common etchants used for metals and alloys.

COMMON ETCHANTS FOR METALS & ALLOYS[3]

Table 20.1: LIGHT METALS - Aluminum Alloys

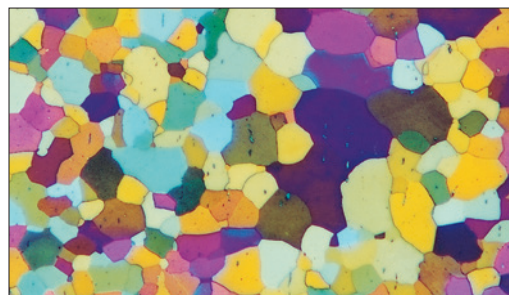| Composition	Comments
1. 95mℓ water 2.5mℓ HNO_3 1.5mℓ HCl 1.0mℓ HF	Keller's reagent, very popular general purpose reagent for Al and Al alloys, except high-Si alloys. Immerse sample 10-20 seconds, wash in warm water. Can follow with a dip in conc. HNO_3. Outlines all common constituents, reveals grain structure in certain alloys when used by immersion.
2. 90-100mℓ water 0.1-10mℓ HF	General purpose reagent. Attacks $FeAl_3$, other constituents outlined. The 0.5% concentrations of HF is very popular.
3. 84mℓ water 15.5mℓ HNO_3 0.5mℓ HF 3g CrO_3	Graff and Sargent's etchant, for grain sizes of 2XXX, 3XXX, 6XXX, and 7XXX wrought alloys. Immerse specimen 20-60 seconds with mild agitation.
4. 1.8% fluoboric acid in water	Barker's anodizing method for grain structure. Use 0.5-1.5 A/in², 30-45 V DC. For most alloys and tempers, 20 seconds at 1 A/in² and 30 V DC at 68° [20°C] is sufficient. Stirring not needed. Rinse in warm water and dry. Use polarized light; sensitive tint helpful.
5. 4g $KMnO_4$ 1g NaOH 100mℓ water	Weck's tint etchant for Al and Al alloys, a very popular etchant for general purpose, especially good for reveal the grain of wrought alloys. Immerse sample for 15-20 seconds and gently wave the sample until the surface is colored.

Table 20.2: Magnesium and Alloys

Composition	Comments
6. 25mℓ water 75mℓ 3-5 ethylene glycol 1mℓ HNO_3	Glycol etch, general purpose etch for pure MG and alloys. Swab specimen seconds for F and T6 temper alloys, 1-2 minutes for T4 and 0 temper alloys.
7. 19mℓ water 60mℓ ethylene glycol 20mℓ acetic acid 1mℓ HNO_3	Acetic glycol etchant for pure Mg and alloys. Swab specimen 1-3 seconds for F and T6 temper alloys, 10 seconds for T4 and 0 temper alloys. Reveals grain boundaries in solution treated castings and most wrought alloys.
8. 100mℓ ethanol 10mℓ water 5g picric acid	For MG and alloys. Use fresh. Immerse specimen for 15-30 seconds. Produces grain contrast.

ETCHING

HELPFUL HINTS FOR ETCHING

Many etchants can be used by swabbing or by immersion. Swabbing is preferred for those specimens that form a tight protective oxide on the surface in air, such as Al, Ni, Cr, stainless steels, Nb (Cb), Ti and Zr. However, if the etchant forms a film, as in tint etchants, then immersion must be used as swabbing will keep the film from forming. Keller's reagent reveals the grain size of certain aluminum alloys by forming a film. This will not occur if the etch is used by swabbing.

Many etchants, and their ingredients, do present potential health hazards to the user. ASTM E 2014, Standard Guide on Metallography Laboratory Safety, describes many of the common problems and how to avoid them.

[3] When water is specified, always use distilled water. It is best to use reagent grade chemicals. Etchants can be quite dangerous and it is advisable to consult with a chemist when dealing with new etchants that may be potentially dangerous. ASTM E 2014, Standard Guide on Metallographic Laboratory Safety, is a valuable reference.

COMMON ETCHANTS FOR METALS & ALLOYS[3]

Table 20.3: **LOW MELTING POINT METALS** - Sb, Bi, Cd, Pb, Sn and Zn	
Composition	**Comments**
9. 100mℓ water 30mℓ HCl 2g $FeCl_3$	For Sb, Bi and alloys. Immerse specimen up to a few minutes.
10. 100mℓ water 25mℓ HCl to a 8g $FeCl_3$	For Sb-Pb, Bi-Sn, Cd-Sn, Cd-Zn and Bi-Cd alloys. Immerse specimen up to a few minutes.
11. 95-99mℓ ethanol 1-5mℓ HNO_3	For Cd, Cd alloys, Sn, Zn alloys, Pb and alloys, Bi-Sn eutectic alloy and Bi-Cd alloys. Can add a few drops of zephiran chloride. Immerse sample. For Pb and alloys, if stain forms, wash in 10% alcoholic HCl.
12. 100mℓ water 10g ammonium molybdate 10g citric acid	Pollack's reagent for Pb and alloys. Immerse specimen 15-30 seconds. Other compositions used are: 100mℓ: 9g: 15g and 100mℓ: 10g: 25g.
13. 100mℓ water 2mℓ HCl 10g $FeCl_3$	For Sn-based babbitt metal. Immerse specimen up to 5 minutes.
14. 200mℓ water 40g CrO_3 3g Na_2SO_4	Palmerton reagent for pure Zn and alloys. Immerse specimen up to 3 minutes. Rinse in 20% aqueous CrO_3.
15. 200mℓ water 10g CrO_3 1g Na_2SO_4	Modified Palmerton reagent for Zn die-casting alloys. Immerse specimen for several seconds, rinse in 20% aqueous CrO_3.
16. 50mℓ Stock Solution* 1g $K_2S_2O_5$	Klemm's I Reagent. For Zn and lean alloys. Immerse the sample and gently shake it until the surface is colored, usually 30s.

* Stock solution: aqueous cold-saturated $Na_2S_2O_3$ solution

[3] When water is specified, always use distilled water. It is best to use reagent grade chemicals. Etchants can be quite dangerous and it is advisable to consul with a chemist when dealing with new etchants that may be potentially dangerous. ASTM E 2014, Standard Guide on Metallographic Laboratory Safety, is a valuable reference.

COMMON ETCHANTS FOR METALS & ALLOYS[3]

Table 20.4: Refractory Metals - Ti, Zr, Hf, Cr, Mo, Re, Nb, Ta, W and V	
Composition	**Comments**
17. 100mℓ water 1-3mℓ HF 2-6mℓ HNO_3	Kroll's reagent for Ti alloys. Swab specimen 3-10 seconds or immerse specimen 10-30 seconds.
18. 200mℓ water 1mℓ HF	For Ti, Zr and alloys. Swab or immerse specimen. Higher concentrations can be used but are prone to staining problems.
19. 30mℓ lactic acid 15mℓ HNO_3 30mℓ HF	For Ti alloys. Swab specimen up to 30 seconds. Decomposes, do not store. Good for alpha-beta alloys.
20. 30mℓ HCl 15mℓ HNO_3 30mℓ HF	For Zr, Hf and alloys. Swab specimen 3-10 seconds or immerse specimen up to 2 minutes.
21. 45mℓ H_2O (H_2O_2 or glycerol) 45mℓ HNO_3 8-10mℓ HF	Cain's chemical polish and etch for Hf, Zr and alloys. Can dilute aqueous solution with 3-5 parts water to stain the structure (swab specimen) after chemical polishing. Chemically polish and etch specimen by swabbing 5-20 seconds. Use polarized light.
22. 60mℓ HCl 20mℓ HNO_3	Aqua regia. For Cr and alloys. Immerse or swab specimen up to 1 minute. Use under a hood with care, do not store.
23. 30mℓ HCl 45mℓ glycerol 15mℓ HNO_3	Modified "Glyceregia." For Cr and alloys. Immerse specimen up to a few minutes.
24. 100mℓ water 10g KOH or NaOH 10g $K_3Fe(CN)_6$	Murakami's reagent. For Cr, Mo, Re, Ta-Mo, W, V and alloys. Use fresh, can immerse sample for up to 1 minute.
25. 70mℓ water 20mℓ H_2O_2 (30%) 10mℓ H_2SO_4	For Mo alloys. Immerse specimen 2 minutes. Wash with water and dry; immersion produces colors, swabbing produces grain-boundary etch.
26. 10-20mℓ glycerol 10mℓ HNO_3 10mℓ HF	For Mo and Mo-Ti alloys. Immerse specimen for up to 5 minutes.
27. 100mℓ water 5g $K_3Fe(CN)_6$ 2g KOH	For Mo-Re alloys. Use at 68°F [20°C] by immersion.
28. 50mℓ acetic acid 20mℓ HNO_3 5mℓ HF	For Nb, Ta and alloys. Swab specimen 10-30 seconds.
29. 50mℓ water 14mℓ H_2SO_4 5mℓ HNO_3	DuPont Nb reagent. For Nb-Hf and Nb alloys.
30. 50mℓ water 50mℓ HNO_3 1mℓ HF	Fo Nb-Zr and Nb-Zr-Re alloys. Swab specimen.
31. 30mℓ lactic acid 10mℓ HNO_3 5mℓ HF	For Re and W-Re alloys. Swab specimen.
32. 10mℓ HF 10mℓ HNO_3 10-30mℓ glycerol	For V and alloys; grain-boundary etch for Ta alloys. Swab specimen. Equal parts used for Ta and high Ta alloys.
33. 100mℓ water 50mℓ Ethanol 2g NH_4HF_2	Modified Weck's tint etchant. Good etchant for Ti and Ti alloys to reveal the grain structure. Use by immersion of sample until the sample surface is colored, usually around 15-25 seconds. The color thin film formation is more uniform if gently shake the sample during the etching process. The etch artifacts produced can be eliminated using only 25mℓ ethanol.

ETCHING

[3] When water is specified, always use distilled water. It is best to use reagent grade chemicals. Etchants can be quite dangerous and it is advisable to consul with a chemist when dealing with new etchants that may be potentially dangerous. ASTM E 2014, Standard Guide on Metallographic Laboratory Safety, is a valuable reference.

COMMON ETCHANTS FOR METALS & ALLOYS[3]

Table 20.5: Iron and Steel

Composition	Comments
34. 90-99mℓ methanol or ethanol 1-10mℓ HNO$_3$	Nital. Most common etchant for Fe, carbon and alloy steels, cast iron. Reveals alpha grain boundaries and constituents. Excellent for martensitic structures. The 2% solution is most common, 5-10% used for high alloy steels (do not store). Use by immersion or swabbing of sample for up to about 1 minute.
35. 100mℓ ethanol 4g picric acid	Picral. Recommended for structures consisting of ferrite and carbide. Does not reveal ferrite grain boundaries. Addition of 0.5-1% zephiran chloride improves etch rate and uniformity.
36. 100mℓ ethanol 5mℓ HCl 1g picric acid	Vilella's reagent. Good for ferrite-carbide structures. Produces grain contrast for estimating prior austenite grain size. Results best on matentsite tempered at 572-932°F [300-500°C]. Occasionally reveals prior-austenite grain boundaries in high alloys steels. Outlines constituents in stainless steels. Good for tool steels and martensitic stainless steels.
37. Saturated aqueous picric acid solution grain plus small amount of a wetting agent	Bechet and Beaujard's etch, most successful etchant for prior-austenite boundaries. Good for martensitic bainitic steels. Many wetting agents have been used, sodium tridecylbenzene sulfonate is one of the most successful (the dodecyl version is easier to obtain and works as well). Use at 68-212°F [20-100°C]. Swab or immerse sample for 2-60 minutes. Etch in ultrasonic cleaner (see ref. 2, pg 219-223). Additions of 0.5 CuCl$_2$ per 100mℓ solution or about 1% HCl have been used for higher alloy steels to produce etching. Room temperature etching most common. Lightly back polish to remove surface smut.
38. 150mℓ water 50mℓ HCl 25mℓ HNO$_3$ 1g CuCl$_2$	Modified Fry's reagent. Used for 18% Ni maraging steels, martensitic and PH stainless steels.
39. 100mℓ water 25g NaOH 2g picric acid	Alkaline sodium picrate. Best etch for McQuaid-Ehn carburized samples. Darkens cementite. Use boiling for 1-15 minutes or electrolytic at 6 V DC, 0.5 A/in^2, 30-120 seconds. May reveal prior-austenite grain boundaries in high carbon steels when no apparent grain boundary film is present.
40. 3 parts HCl 2 parts glycerol 1 part HNO$_3$	"Glyceregia." For austenitic stainless steels. Reveals grain structure, outlines sigma and carbides. Mix fresh, do not store. Use by swabbing.
41. 100mℓ ethanol 100mℓ HCl 5g CuCl$_2$	Kalling's no. 2 ("waterless" Kalling's) etch for austenitic and duplex stainless steels. Ferrite attacked readily, carbides unattacked, austenite slightly attacked. Use at 68°F [20°C] by immersion or swabbing. Can be stored.
42. 15mℓ HCl 10mℓ acetic acid 5mℓ HNO$_3$ 2 drops glycerol	Acetic glyceregia. Mix fresh, do not store. Use for high alloy stainless steels.
43. 100mℓ water 10g K$_2$Fe(CN)$_6$ 10g KOH or NaOH	Murakami's reagent. Usually works better on ferritic stainless grades than austenitic grades. Use at 68°F [20°C] for 7-60 seconds: reveals carbides sigma faintly attacked with etching up to 3 minutes. Use at 176°F [80°C] to boiling for 2-60 minutes: carbides dark, sigma blue (not always attacked), ferrite yellow to yellow-brown, austenite unattacked. Do not always get uniform etching.
44. 100mℓ water 10g oxalic acid	Use for stainless steels at 6 V DC. Carbides reveal by etching for 15-30 seconds, grain boundaries after 45-60 seconds, sigma outlined after 6 seconds. 1-3 V also used. Dissolves carbides, sigma strongly attacked, austenite moderately attacked, ferrite unattacked.
45. 100mℓ water 20g NaOH	Used to color ferrite in martensitic, PH or dual-phase stainless steels. Use at 3-5 V DC, 68°F [20°C], 5 seconds, stainless steel cathode. Ferrite outlines and colored tan.
46. 40mℓ water 60mℓ HNO$_3$	Electrolytic etch to reveal austenite boundaries but not twin boundaries in austenitic stainless steels (304, 316, etc.). Voltage is critical. Pt cathode preferred to stainless steel. Use at 1.4V DC, 2 minutes (see ref.2, pgs 235, 238 and 239).
47. 50mℓ Stock Solution* 1g K$_2$S$_2$O$_5$	Klemm's I Reagent. Immerse at room temperature until surface is colored. It is good to color ferrite in steel, reveals P segregation and overheating, etching time is 40-100 seconds. For coloring ferrite and martensite in cast iron low-alloy steels, usually up to 3 minutes.
48. 50mℓ Stock Solution* 5g K2S2O5	Klemm's II Reagent. For steel sample, immerse for 30-90 seconds at 20°C, reveals P segregation. For austenitic Mn Steels, gamma yellow to brown or light to dark blue, alpha-martensite dark brown. Good for austenitic manganese steels.
49. 100mℓ Ehtanol 2mℓ HCl 0.5mℓ H2SeO4	Beraha's Selenic Acid Etch 1. For cast iron, steels, and tool steels. For better result, Nital pre-etch is recommended. Use by immersion of sample until the sample surface is colored, usually up to 6 minutes. Cementite colored red-violet, ferrite colored yellow or brown, phosphide colored blue-green.
50. 100mℓ Ethanol 5-10mℓ HCl 1-3mℓ H2SeO4	Beraha's Selenic Acid Etch 2. In general, immerse sample at 20°C for 1-10 minutes. A yellow or light-brown surface color is formed to detect carbides and nitrides or an orange to red surface is formed to detect delta ferrite. Can use 20-30mℓ HCl for higher alloy grades, immersion time decrease with higher HCl.

Stock solution: aqueous cold-saturated Na$_2$S$_2$O$_3$ solution

[3] When water is specified, always use distilled water. It is best to use reagent grade chemicals. Etchants can be quite dangerous and it is advisable to consul with a chemist when dealing with new etchants that may be potentially dangerous. ASTM E 2014, Standard Guide on Metallographic Laboratory Safety, is a valuable reference.

COMMON ETCHANTS FOR METALS & ALLOYS[3]

Table 20.6: COPPER, NICKEL AND COBALT: COPPER AND ALLOYS

Composition	Comments
51. 25mℓ NH_4OH 25mℓ water (optional) 25-50mℓ H_2O_2 (3%)	General purpose grain contrast etch for Cu and alloys (produces a flat etch for some alloys). Use fresh, add peroxide last. Use under a hood. Swab specimen 5-45 seconds.
52. 100mℓ water 10g ammonium persulfate	General purpose etch for Cu and alloys. Immerse or swab for 3-60 seconds. Reveals grain boundaries but is sensitive to crystallographic orientation.
53. 100mℓ water 3g ammonium persulfate 1mℓ NH_4OH	General purpose etch for Cu and alloys, particulatly Cu-Be alloys.
54. 70mℓ water 5g $Fe(NO_3)_3$ 25mℓ HCl	Excellent general purpose etch, reveals grain boundaries well. Immerse specimen 10-30 seconds.
55. 300mℓ Ethanol 2mℓ HCl 0.5 -1mℓ Selenic Acid	Beraha's Selenic Acid Etchant for brass and Cu-Be alloys. Pre-etch the sample is useful for clear grain-boundary. Immerse sample until surface is violet-blue to blue. Use plastic tongs and storage in dark bottle. If the reaction is too fast and the color changes too quick, use 0.5mL selenic acid.
56. 50mℓ Stock Solution* 1g $K_2S_2O_5$	Klemm's I reagent. It is a good etchant for beta brass, alpha-beta brass, and bronzes. Immerse for 2 minutes or more and gently wave the sample until the surface is colored. For alpha brass, the reaction is very slow, usually up to 60 minutes.
57. 50mℓ Stock Solution* 1g $K_2S_2O_5$	Klemm's III reagent. Excellent etchant for copper alloys and best for the cartridge brass. Although the color range was limited, results were very good in bright field. Polarized light and sensitive tint improves color response. Immerse sample 3-5 minutes for bronze alloys.
58. 5mℓ Stock Solution* 45mℓ Water 20g $K_2S_2O_5$	Klemm's III reagent. Excellent etchant for copper alloys and best for the cartridge brass. Although the color range was limited, results were very good in bright field. Polarized light and sensitive tint improves color response. Immerse sample 3-5 minutes for bronze alloys.

Stock solution: aqueous cold-saturated $Na_2S_2O_3$ solution

Table 20.7: NICKEL AND ALLOYS

Composition	Comments
59. 5g $FeCl_3$ 2mℓ HCl 99mℓ ethanol	Carapella's etch for Ni and Ni-Cu (Monel) alloys. Use by immersion or swabbing.
60. 40-80mℓ ethanol 40mℓ HCl 2g $CuCl_2$	Kalling's no. 2 etch ("waterless" Kalling's) for Ni-Cu alloys and superalloys. Immerse or swab specimen up to a few minutes.
61. 50mℓ water 50mℓ HCl 10g $CuSO_4$	Marble's reagent for Ni, Ni-Cu and Ni-Fe alloys and superalloys. Immerse or swab sample 5-60 seconds. Reveals grain structure of superalloys.
62. 15mℓ HCl 10mℓ glycerol 5mℓ HNO_3	"Glyceregia," for superalloys and Ni-Cr alloys. Swab specimen for 5-60 seconds. Mix fresh. Do not store. Use under hood.
63. 60mℓ glycerol 50mℓ HCl 10mℓ HNO_3	Modified Glyceregia for superalloys. Reveals precipitates. Use under hood; do not store. Add HNO_3 last. Discard when dark yellow. Immerse or swab specime 10-60 seconds.

ETCHING

[3] When water is specified, always use distilled water. It is best to use reagent grade chemicals. Etchants can be quite dangerous and it is advisable to consul with a chemist when dealing with new etchants that may be potentially dangerous. ASTM E 2014, Standard Guide on Metallographic Laboratory Safety, is a valuable reference.

COMMON ETCHANTS FOR METALS & ALLOYS[3]

Table 20.8: Cobalt and Alloys

Composition	Comments
64. 60mℓ HCl 15mℓ water 15mℓ acetic acid 15mℓ HNO$_3$	For Co and alloys. Mix fresh and age 1 hour before use. Immerse specimen for up to 30 seconds. Do not store.
65. 200mℓ ethanol 7.5mℓ HF 2.5mℓ HNO$_3$	General etch for Co and alloys. Immerse specimen 2-4 minutes.
66. 50mℓ water 50mℓ HCl 10g CuSO$_4$	Marble's reagent for Co high temperature alloys. Immerse specimen for up to 1 minute.
67. 80mℓ lactic acid 10mℓ H$_2$O$_2$ (30%) 10mℓ HNO$_3$	For Co alloys. Use by swabbing.

Table 20.9: Precious Metals: Au, Ag, Ir, Os, Pd, Pt, Rh and Ru

Composition	Comments
68. 60mℓ HCl 40mℓ HNO$_3$	For gold, silver, palladium and high noble metal alloys. Use under hood. Immerse specimen up to 60 seconds. Equal parts of each acid also used.
69. 60mℓ HCl 20mℓ HNO$_3$	Aqua regia for pure gold, Pt and alloys, some Rh alloys. Use boiling for up to 30 minutes.
70. 1-5g CrO$_3$ 100mℓ HCl	For Au, Ag, Pd, and alloys. Swab or immerse specimen up to 60 seconds.
71. 30mℓ water 25mℓ HCl 5mℓ HNO$_3$	For pure Pt. Use hot, immerse specimen up to 5 minutes.
72. Conc. HCl	For Rh and alloys. Use at 5 V AC, 1-2 minutes, graphite cathose, Pt lead wires.
73. Solution A: 100mℓ water 40g NaCl Solution B: Conc. HCl	For Ru. Mix 4 parts Solution A to 1 part Solution B, use 5-20 V AC, 1-2 minutes, graphite cathode, Pt-lead wires.
74. 50mℓ NH$_4$OH 20mℓ H$_2$O$_2$ (3%)	For pure Ag, Ag solders and Ag-Pd alloys. Mix fresh. Swab specimen up to 60 seconds. Discard after etching. Use a 50:50 mix for sterling silver; for fine silver, use 30% conc. hydrogen peroxide.
75. Solution A: 10g NaCN 100mℓ water Solution B: H$_2$O$_2$ (30%)	For gold alloys up to 18 karat. Mix equal amounts of A and B directly on the specimen using eye droppers. Swab and replenish the etchants until desired etch level is obtained. If brown stain forms, swab with A to remove it.

Table 20.10: Sintered Carbides

Composition	Comments
76. 100mℓ water 10g KOH or NaOH 10g K$_3$Fe(CN)$_6$ Normally used at 20°C	Murakami's reagent, for WC-Co and complex sintered carbides. Immerse specimen seconds to minutes. Use 2-10 seconds to identify eta phase (colored). Longer times attack eta. Reveals phase and grain boundaries.
77. 97mℓ water 3mℓ H$_2$O$_2$ (30%)	For WC, MO$_2$C, TiC or Ni in sintered carbides. Use boiling for up to 60 seconds. For coarse carbides or high Co content, use short etch time.
78. 15mℓ water 30mℓ HCl 15mℓ HNO$_3$ 15mℓ acetic acid	For WC, WiC, TaC and Co in sintered carbides. Use at 68°F [20°C] for 5-30 seconds.
79. 100mℓ water 3g FeCl$_3$	To darken Co (or Ni) binder phase. Mix fresh. Swab 10 seconds.

[3] When water is specified, always use distilled water. It is best to use reagent grade chemicals. Etchants can be quite dangerous and it is advisable to consul with a chemist when dealing with new etchants that may be potentially dangerous. ASTM E 2014, Standard Guide on Metallographic Laboratory Safety, is a valuable reference.

COMMON ETCHANTS FOR METALS & ALLOYS[3]

Table 20.11: CERAMICS AND NITRIDES

Composition	Comments
80. Phosphoric acid	For alumina, Al_2O_3, and silicon nitride, Si_3N_4. Use by immersion at 482°F [250°C] for up to a few minutes for Al_2O_3 or up to 15 minutes for Si_3N_4.
81. 100mℓ water 15mℓ HNO_3	For magnesia, MgO. Use by immersion at 77-140°F [25-60°C] for several minutes.
82. 100mℓ water 5g NH_4FHF 4mℓ HCl	For titania, TiO_2. Immerse up to a few minutes.
83. 50mℓ water 50mℓ H_2SO_4	For zirconia, ZrO_2. Use by immersion in boiling solution for up to 5 minutes.
84. HCl	For calcia, CaO, or MgO. Immerse for up to 6 minutes.
85. HF	For Si_3N_4, BeO, BaO, MgO, ZrO_2 and Zr_2O_3. Immerse for up to 6 minutes.

Table 20.12: PLASTICS AND POLYMERS

Composition	Comments
86. 100mℓ water 60g CrO_3	For polypropylene (PP). Immerse for up to several hours at 158°F [70°C].
87. HNO_3	For polyethylene (PE). Immerse for up to a few minutes.
88. Xylol	Reveals spherolites in polyethylene (PE). Immerse for up to a few days at 158°F [70°C].
89. 70mℓ water 30mℓ HCl	For polyoxymethylene (POM). Immerse up to 20 seconds.
90. Xylene	For polyamid (PA) and polyethylene (PE). Use at 158°F [70°C] for 60 seconds. For Nylon 6, use at 149-158°F [65-70°C] for 2-3 minutes. For Nylon 6, use at 167°F [75°C] for 3-4 minutes.

Table 20.13: ELECTROLYTIC ETCHING

Composition	Comments
91. 700mℓ ethyl alcohol 120mℓ water 80mℓ perchloric acid 100mℓ butylglycol	Electrolyte for polishing and etching of Al and Al alloys. Polishing time is 30-60 seconds. at 30V DC, etching time is 20 seconds at 5-10V DC.
92. 625mℓ Ethanol 375mℓ Phosphoric Acid	Electrolyte for polishing of Mg and Mg alloys. Polishing time is up to several mins. at 30-40V DC.
93. 700mℓ ethyl alcohol 120mℓ water 80mℓ perchloric acid 100mℓ butylglycol	Electrolyte for polishing and etching of carbon and HS steels in different conditions. Polishing time is 60-90 seconds at 30-40V DC, etching time is 20 seconds at 5-10V DC.
94. 500mℓ water 250mℓ Ethanol 250mℓ Phosphoric Acid	Electrolyte for polishing and etching of Copper and alloys. Polishing time is 60 seconds at 20-30V DC and etching time is 20 seconds at 5-10V DC. Adding the phosphoric slowly at the end and wait till it's cooled down.

ETCHING

[3] When water is specified, always use distilled water. It is best to use reagent grade chemicals. Etchants can be quite dangerous and it is advisable to consul with a chemist when dealing with new etchants that may be potentially dangerous. ASTM E 2014, Standard Guide on Metallographic Laboratory Safety, is a valuable reference.

LIGHT OPTICAL MICROSCOPY

Metallurgical microscopes differ from biological microscopes primarily in the manner by which the specimen is illuminated due to the opacity of metals. Unlike biological microscopes, metallurgical microscopes must use reflected light, rather than transmitted light. Figure 21.1 is a simplified light ray diagram of a metallurgical microscope. The prepared specimen is placed on the stage with the surface perpendicular to the optical axis of the microscope and is illuminated through the objective lens by light from a lamp or arc source. This light is focused by the condenser lens into a beam that is made approximately parallel to the optical axis of the microscope by the half silvered mirror. The light then passes through the objective onto the specimen. It is then reflected from the surface of the specimen, back through the objective, the half silvered mirror, and then to the eyepiece to the observer's eye, or to a camera port or a film plane.

After the specimen has been properly sectioned and polished, it is ready for examination. However, in many cases, it is important to begin the examination with an as-polished specimen, particularly for quality control or failure analysis work. Certain microstructural constituents, such as nonmetallic inclusions, graphite, nitrides, cracks or porosity, are best evaluated in the as-polished condition as etching reveals other microstructural features that may obscure such details. In the as-polished condition, there is a minimum of extra information for the observer to deal with which makes examination of these features most efficient. Some materials do not require etching and, in a few cases, examination is less satisfactory after etching.

For most metals, etching must be used to fully reveal the microstructure. A wide range of processes are used, the most common being etching with either acidic or basic solutions. For most metals and alloys, there are a number of general-purpose etchants that should be used first to reveal the microstructure. Subsequent examination may indicate that other more specialized techniques for revealing the microstructure may be useful. For example, the examination may reveal certain constituents that the metallographer may wish to measure. Measurements, particularly those performed with automatic image analyzers, will be simpler to perform and more reliable if the desired constituent can be revealed selectively, and numerous procedures are available to produce selective phase contrasting.

The Light Microscope

When Henry Clifton Sorby made his first examination of the microstructure of iron on 28 July 1863, he was using a transmitted light petrographic microscope made by Smith, Beck and Beck of London that he had purchased in 1861. Although capable of magnifications up to 400X, most of his examinations were conducted at 30, 60 or 100X and
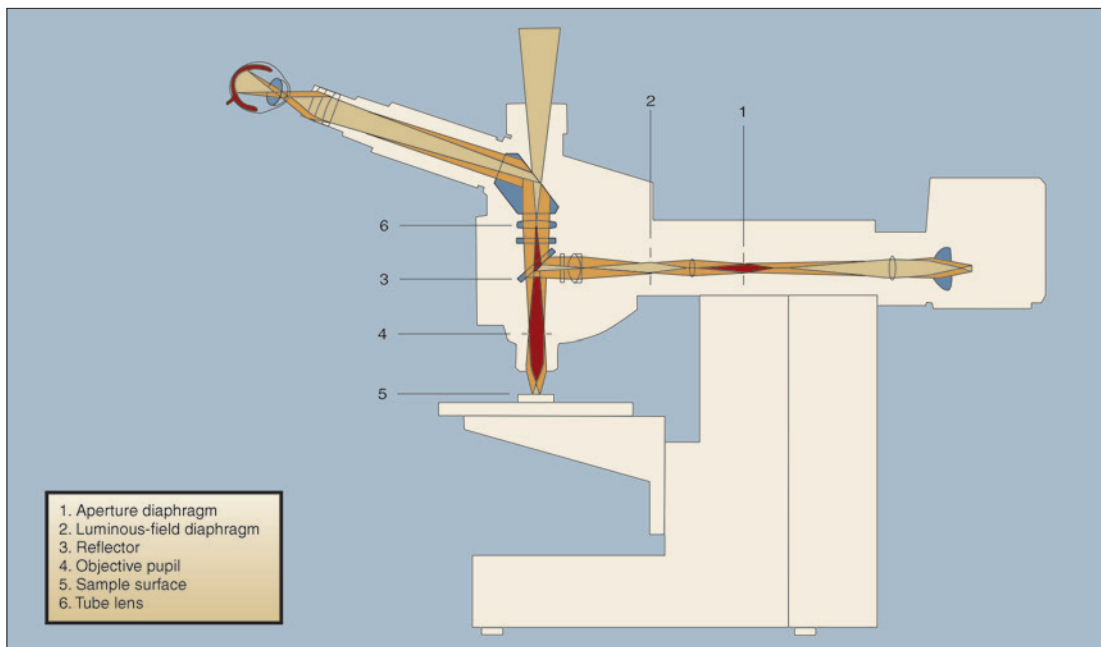

1. Aperture diaphragm
2. Luminous-field diaphragm
3. Reflector
4. Objective pupil
5. Sample surface
6. Tube lens

Figure 21.1. Schematic diagram showing the light path through an upright reflected light microscope operating with bright field illumination.

his first micrographs were produced at only 9X. The objective lenses of this microscope were equipped with Lieberkühns silvered concave reflectors for focusing light on opaque specimens.

Sorby quickly realized that reflected light produced with the Lieberkühns reflectors was inadequate and he developed two alternate methods for this purpose. Subsequently, others developed vertical illuminators using prisms or plane glass reflectors and Sorby's systems were not further developed. In 1886, Sorby reported on the use of "very high power" (650X) for the study of pearlite. This was accomplished using a 45° cover glass vertical illuminator made for him by Beck.

For many years, photomicroscopy was conducted using specially built reflected microscopes known as metallographs. These devices represented the "top-of-the-line" in metallurgical microscopes and were essential for quality work. In the late 1960's and early 1970's, manufacturers developed metallographs that were easier to use from the eyepiece viewing position. The temperamental carbon arc light sources were replaced by xenon arc lamps. The unwieldy bellows systems for altering magnification were replaced by zoom systems. Vertical illuminators, previously using single position objectives, were equipped with four to six position rotatable nosepiece turrets to minimize objective handling. The light path was deflected so that the film plane was conveniently near at hand. Universal type vertical illuminators and objective lenses were introduced so that the illumination mode could be readily switched from bright field to darkfield, polarized light or differential interference contrast. Such systems were very attractive in that separate vertical illuminators and objectives were no longer needed for each illumination mode and handling was eliminated. Exposure meters were also added at this time, chiefly as a result of the rising popularity of instant films where such devices are needed to minimize film wastage. But, by the late 1970s, these large metallographs had become very expensive, too expensive for most laboratories.

In 1979, microscope manufacturers began to introduce very high quality, reasonably priced, compact metallographs. These microscopes can be obtained with a wide variety of objective lens types and auxiliary accessories to meet the metallographer's needs. They are available with universal vertical illuminators that permit easy switching from one illumination mode to another using the same set of objective lenses. Furthermore, the manufacturers have introduced new glass compositions and lens formulations, generally by computer-aided design, for improved image contrast and brightness. Tungsten-halogen filament lamps have largely replaced xenon arc lamps as the preferred light source.

Microscope Components

Light Sources. The amount of light lost during passage from the source through a reflecting type microscope is appreciable because of the intricate path the light follows. For this reason, it is generally preferable that the intensity of the source be high, especially for photomicroscopy. Several types of light sources are used including tungsten-filament lamps, tungsten-halogen lamps, quartz-halogen lamps, and xenon arc bulbs. Tungsten-filament lamps generally operate at low voltage and high current. They are widely used for visual examination because of their low cost and ease of operation.

Tungsten-halogen lamps are the most popular light source today due to their high light intensity. They produce good color micrographs when tungsten-corrected films are employed. Light intensity can be varied easily to suit the viewing conditions by adjusting a rheostat, a distinct advantage over arc lamps.

Xenon arc lamps produce extremely high intensity light, and their uniform spectra and daylight color temperature makes them suitable for color photomicrography. The first xenon lamps produced ozone, but modern units have overcome this problem. Light output is constant and can only be reduced using neutral density filters.

Condenser. An adjustable lens free of spherical aberration and coma is placed in front of the light source to focus the light at the desired point in the optical path. A field diaphragm is placed in front of this lens to minimize internal glare and reflections within the microscope. The field diaphragm is stopped down to the edge of the field of view.

A second adjustable iris diaphragm, the aperture diaphragm, is placed in the light path before the vertical illuminator. Opening or closing this diaphragm alters the amount of light and the angle of the cone of light entering the objective lens. The optimum setting for the aperture varies with

each objective lens and is a compromise among image contrast, sharpness, and depth of field. As magnification increases, the aperture diaphragm is stopped down. Opening this aperture increases image sharpness, but reduces contrast; closing the aperture increases contrast, but impairs image sharpness. The aperture diaphragm should not be used for reducing light intensity. It should be adjusted only for contrast and sharpness.

Filters. Light filters are used to modify the light for ease of observation, for improved photomicroscopy, or to alter contrast. Neutral density filters are used to reduce the light intensity uniformly across the visible spectrum. Various neutral density filters, with a range of approximately 85 to 0.01% transmittance, are available. They are a necessity when using an arc-lamp but virtually unnecessary when using a filament lamp.

Selective filters are used to balance the color temperature of the light source to that of the film. They may be needed for faithful reproduction of color images, depending on the light source used and the film type. A green or yellow-green filter is widely used in black and white photography to reduce the effect of lens defects on image quality. Most objectives, particularly the lower cost achromats, require such filtering for best results.

Polarizing filters are used to produce plane polarized light (one filter) or crossed-polarized light (two filters rotated 90° to each other to produce extinction) for examinations of anisotropic noncubic (crystallographic) materials.

Objectives. The objective lens forms the primary image of the microstructure and is the most important component of the optical microscope. The objective lens collects as much light as possible from the specimen and combines this light to produce the image. The numerical aperture (NA) of the objective, a measure of the light-collecting ability of the lens, is defined as:

$$NA = n \sin \alpha$$

where n is the minimum refraction index of the material (air or oil) between the specimen and the lens, and α is the half-angle of the most oblique light rays that enter the front lens of the objective. Light-collecting ability increases with α. The setting of the aperture diaphragm will alter the NA of the condenser and therefore the NA of the system.

The most commonly used objective is the achromat, which is corrected spherically for one color (usually yellow green) and for longitudinal chromatic aberration for two colors (usually red and green). Therefore, achromats are not suitable for color photomicroscopy, particularly at higher magnifications. Use of a yellow-green filter yields optimum results. However, achromats do provide a relatively long working distance, that is, the distance from the front lens of the objective to the specimen surface when in focus. The working distance decreases as the objective magnification increases. Most manufacturers make long-working distance objectives for special applications, for example, in hot stage microscopy. Achromats are usually strain free, which is important for polarized light examination. Because they contain fewer lenses than other more highly corrected lenses, internal reflection losses are minimized.

Semi-apochromatic or fluorite objectives provide a higher degree of correction of spherical and chromatic aberration. Therefore, they produce higher quality color images than achromats. The apochromatic objectives have the highest degree of correction, produce the best results, and are more expensive. Plano objectives have extensive correction for flatness of field, which reduces eyestrain, and are usually found on modern microscopes.

With parfocal lens systems, each objective on the nosepiece turret will be nearly in focus when the turret is rotated, preventing the objective front lens from striking the specimen when lenses are switched. Many objectives also are spring loaded, which helps prevent damage to the lens. This is more of a problem with high magnification objectives, because the working distance can be very small.

Certain objectives are designed for use with oil between the specimen and the front lens of the objective. However, oil-immersion lenses are rarely used in metallography, because the specimen and lens must be cleaned after use. But, they do provide higher resolution than can be achieved when air is between the lens and specimen. In the latter case, the maximum possible NA is 0.95; oil-immersion lenses produce a 1.3 to 1.45 NA, depending on the lens and the oil-immersion used. Objective magnifications from about 25 to 200X are available,

depending upon the manufacturer. Use of oil also improves image contrast, which is valuable when examining low reflectivity specimens, such as coal or ceramics.

Eyepieces. The eyepiece, or ocular, magnifies the primary image produced by the objective; the eye can then use the full resolution capability of the objective. The microscope produces a virtual image of the specimen at the point of most distinct vision, generally 10in [250mm] from the eye. The eyepiece magnifies this image, permitting achievement of useful magnifications. The standard eyepiece has a 24mm diameter field of view; wide field eyepieces for plano-type objectives have a 30mm diameter field of view, which increases the usable area of the primary image. Today, the wide field plano-objective is standard on nearly all metallurgical microscopes.

The simplest eyepiece is the Huygenian, which is satisfactory for use with low and medium power achromat objectives. Compensating eyepieces are used with high NA achromats and the more highly corrected objectives. Because some lens corrections are performed using these eyepieces, the eyepiece must be matched with the type of objective used. The newer, infinity-corrected microscopes do not perform corrections in the eyepieces, but in the tube lens. Eyepieces, therefore, are simpler in infinity-corrected microscopes.

Eye clearance is the distance between the eye lens of the ocular and the eye. For most eyepieces, the eye clearance is 10mm or less – inadequate if the microscopist wears glasses. Simple vision problems, such as near sightedness, can be accommodated using the fine focus adjustment. The microscope cannot correct vision problems such as astigmatism, and glasses must be worn. High eyepoint eyepieces are available to provide an eye clearance of approximately 20mm, necessary for eyeglasses.

Eyepieces are commonly equipped with various reticles or graticules for locating, measuring, counting or comparing microstructures. The eyepiece enlarges the reticle or graticule image and the primary image. Both images must be in focus simultaneously. Special eyepieces are also produced to permit more accurate measurements than can be made with a graticule scale. Examples are the filar-micrometer ocular or screw-micrometer ocular. Such devices can be au-

tomated to produce a direct digital readout of the measurement, which is accurate to approximately 1μm.

A 10X magnification eyepiece is usually used; to obtain standard magnifications, some systems require other magnifications, such as 6.3X. Higher power eyepieces, such as 12, 15, 20, or 25X, are also useful in certain situations. The overall magnification is found by multiplying the objective magnification, M_o, by the eyepiece magnification, M_e. If a zoom system or bellows is also used, the magnification should be altered accordingly.

Stage. A mechanical stage is provided for focusing and moving the specimen, which is placed on the stage and secured using clips. The stage of an inverted microscope has replaceable center stage plates with different size holes. The polished surface is placed over the hole for viewing. However, the entire surface cannot be viewed, unless the specimen is smaller than the hole and it is mounted. At high magnifications it may not be possible to focus the objective near the edge of the hole due to the restricted working distance.

Using the upright microscope, the specimen is placed on a slide on the stage. Because the polished surface must be perpendicular to the light beam, clay is placed between the specimen bottom and the slide. A piece of lens tissue is placed over the polished surface, and the specimen is pressed into the clay using a leveling press. However, pieces of tissue may adhere to the specimen surface. An alternative, particularly useful with mounted specimens, is to use a ring instead of tissue to flatten the specimen. Aluminum or stainless steel ring forms of the same size as the mounts (flattened slightly in a vise) will seat on the mount rather than the specimen.

The upright microscope allows viewing of the entire surface with any objective, and the operator can see which section of the specimen is being viewed – a useful feature when examining specific areas on coated specimens, welds, and other specimens where specific areas are to be examined. For mounted specimens, an autoleveling stage holder can eliminate leveling specimens with clay.

The stage must be rigid to eliminate vibrations.

LIGHT OPTICAL MICROSCOPY

Stage movement, controlled by x and y micrometers, must be smooth and precise; rack and pinion gearing is normally used. Many stages have scales for measuring distances in the x and y directions. The focusing controls often contain rulings for estimating vertical movement. Some units have motorized stages and focus controls.

A circular, rotatable stage plate may facilitate polarized light examination. Such stages, common for mineralogical or petrographic studies, are graduated to permit measuring the angle of rotation. A rectilinear stage is generally placed on top of the circular stage.

Stand. Bench microscopes require a rigid stand, particularly if photomicroscopy is performed on the unit. The various pieces of the microscope are attached to the stand when assembled. In some cases, the bench microscope is placed on a separate stand that also holds the photographic system.

Resolution

To see microstructural detail, the optical system must produce adequate resolution, or resolving power, and adequate image contrast.

If resolution is acceptable but contrast is lacking, detail cannot be observed. In general, the ability to resolve two points or lines separated by distance d is a function of the wavelength, λ, of the incident light and the numerical aperture, NA, of that objective.

$$d = \frac{k\lambda}{NA}$$

where k is 0.5 or 0.61. Figure 21.2 illustrates this relationship for k = 0.61 and four light wavelengths. Other formulas have also been reported. This equation does not include other factors that influence resolution, such as the degree of correction of the objectives and the visual acuity of the microscopist. It was based on the work of Abbe under conditions not present in metallography, such as self-luminous points, perfect black-white contrast, transmitted light examination, an ideal point-light source, and absence of lens defects.

Using the above equation, the limit of resolution for an objective with an NA of 1.3 is approximately 0.2μm. To see lines or points spaced 0.2μm apart, the required magnification is determined by divid-
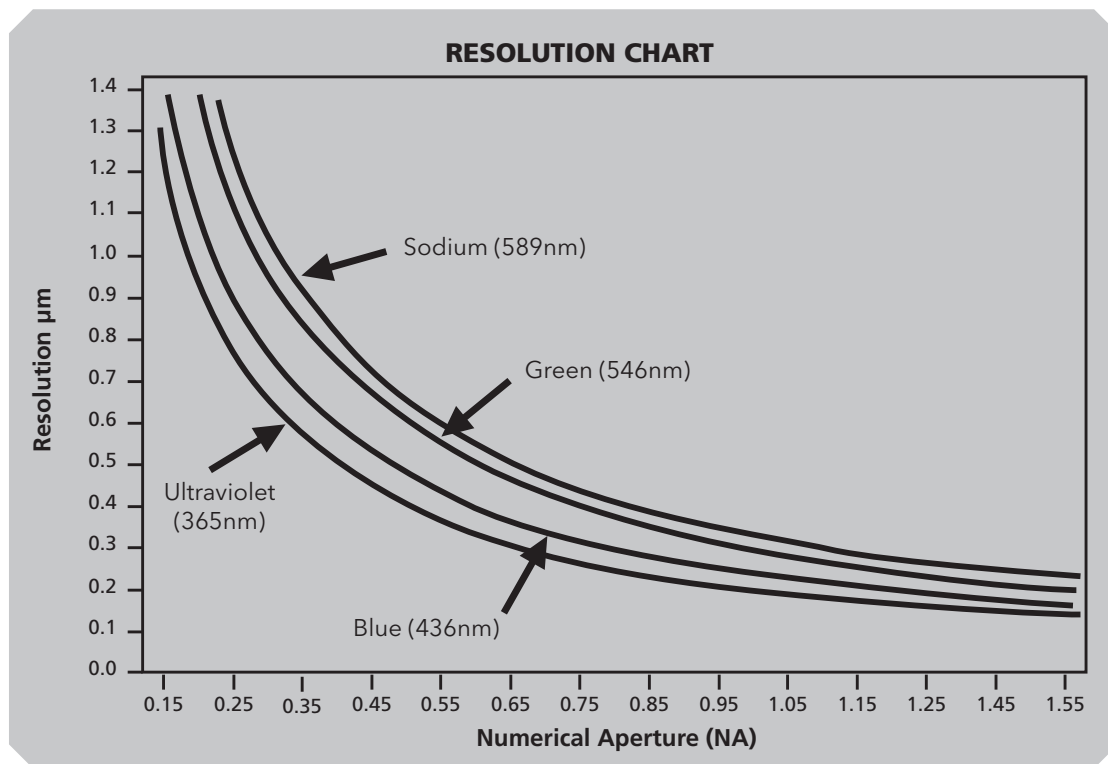

RESOLUTION CHART

Figure 21.2. Influence of objective numerical aperture and light wavelength on the resolution of the light microscope.

ing by the resolving power of the human eye, which is difficult to determine under observation conditions. Abbe used a value of 0.3mm at a distance of 10in [250mm] – the distance from the eye for optimum vision. This gives 1500x. For light with a mean wavelength of 0.55µm, the required magnification is 1100 times the NA of the objective. This is the origin of the 1000·NA rule for the maximum useful magnification. Any magnification above 1000·NA is termed "empty", or useless.

Strict adherence to the 1000·NA rule should be questioned, considering the conditions under which it was developed, certainly far different from those encountered in metallography. According to the Abbe analysis, for a microscopist with optimum 20/20 vision and for optimum contrast conditions and a mean light wavelength of 550nm, the lowest magnification that takes full advantage of the NA of the objective is 500 times the NA. This establishes a useful minimum magnification to use with a given objective. It has been suggested that the upper limit of useful magnification for the average microscopist is 2200·NA, not 1000·NA.

Depth of Field

Depth of field is the distance along the optical axis over which image details are observed with acceptable clarity. Those factors that influence resolution also affect depth of field, but in the opposite direction. Therefore, a compromise must be reached between these two parameters, which is more difficult as magnification increases. This is one reason light etching is preferred for high-magnification examination. The depth of field, d_f, can be estimated from:

$$d_f = \frac{\lambda \, (n^2 - NA^2)^{\frac{1}{2}}}{NA^2}$$

where n is the refractive index of the medium between the specimen and the objective (n ~ 1.0 for air), λ is the wavelength of light, and NA is the numerical aperture. This equation shows that depth of field increases as the NA decreases and when longer wavelength light is used, as shown in Figure 21.3.
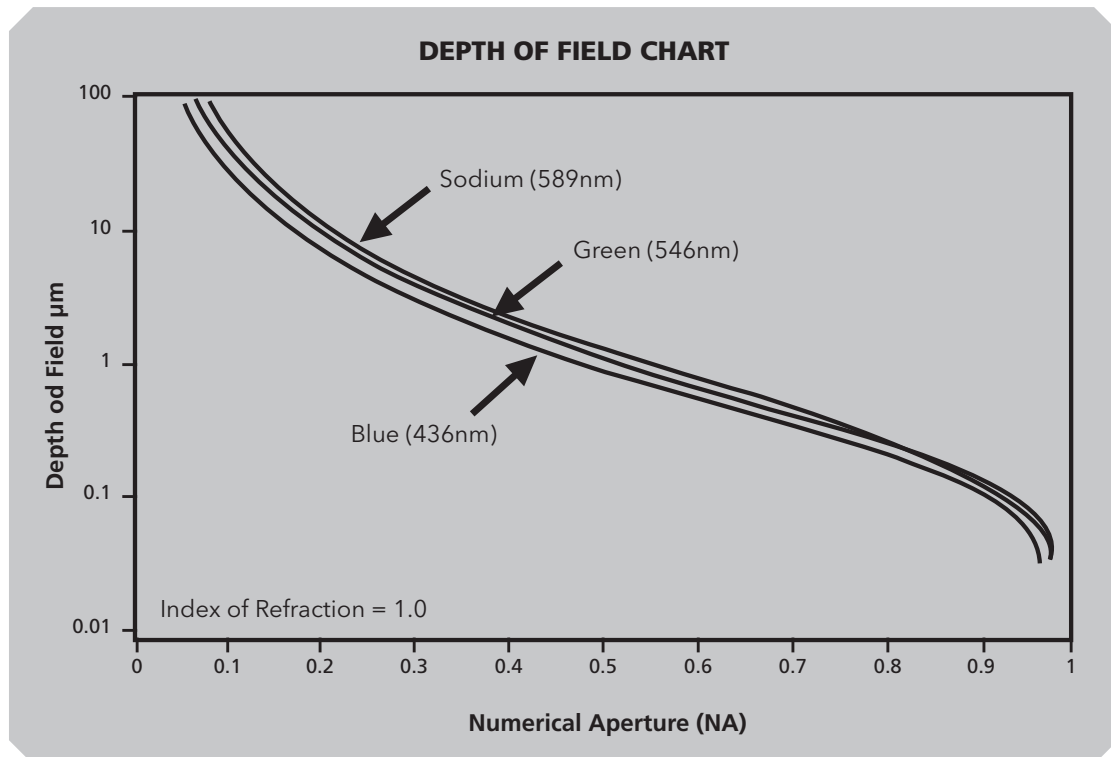
DEPTH OF FIELD CHART

Sodium (589nm)

Green (546nm)

Blue (436nm)

Index of Refraction = 1.0

Depth od Field µm

Numerical Aperture (NA)

Figure 21.3. Influence of the objective numerical aperture and light wavelength on the depth of field of the light microscope.

LIGHT OPTICAL MICROSCOPY

Imaging Modes

Most microscopical studies of metals are made using bright field illumination. In addition to this type of illumination, several special techniques (oblique illumination, dark field illumination, differential interference contrast microscopy and polarized light microscopy) have particular applications for metallographic studies.

Nearly all microscopes using reflected or transmitted light employ Köhler illumination because it provides the most intense, most even illumination possible with standard light sources. The reflected light microscope has two adjustable diaphragms: the aperture diaphragm and the field diaphragm, located between the lamp housing and the objective. Both are adjusted to improve illumination and the image. To obtain Köhler illumination, the image of the field diaphragm must be brought into focus on the specimen plane. This normally occurs automatically when the microstructural image is brought into focus. The filament image must also be focused on the aperture diaphragm plane. This produces uniform illumination of the specimen imaged at the intermediate image plane and magnified by the eyepiece.

Bright Field. In bright field illumination, the surface of the specimen is normal to the optical axis of the microscope, and white light is used. A ray diagram for bright-field illumination is illustrated in Figure 21.4. Light that passes through the objective and strikes a region of the specimen surface that is perpendicular to the beam will be reflected back up the objective through the eyepieces to the eyes where it will appear to be bright or white. Light that strikes grain boundaries, phase boundaries, and other features not perpendicular to the optical axis will be scattered at an angle and will not be collected by the objective. These regions will appear to be dark or black in the image. Bright field is the most common mode of illumination used by metallographers.

Oblique Illumination. The surface relief of a metallographic specimen can be revealed using oblique illumination. This involves offsetting the condenser lens system or, as is more usually done, moving the condenser aperture to a position slightly off the optical axis. Although it should be possible to continually increase the contrast achieved by oblique illumination by moving the condenser farther and farther from the optical axis, the numerical aperture of a lens is reduced when microstructural image is brought into focus. The filament image must also be focused on the aperture diaphragm plane. This produces uniform illumination of the specimen imaged at the intermediate image plane and magnified by the eyepiece.

Dark Field. Another method that can be used to distinguish features not in the plane of the polished and etched surface of a metallographic specimen is dark field (also called dark ground) illumination. This type of illumination (see Figure 21.5 for a ray diagram) gives contrast completely reversed from that obtained with bright-field illumination – the features that are light in bright field will be dark in dark field, and those
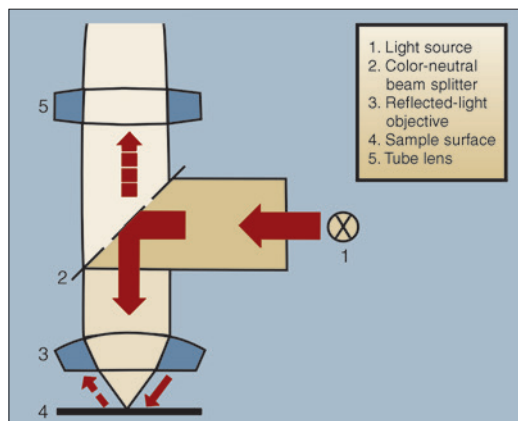

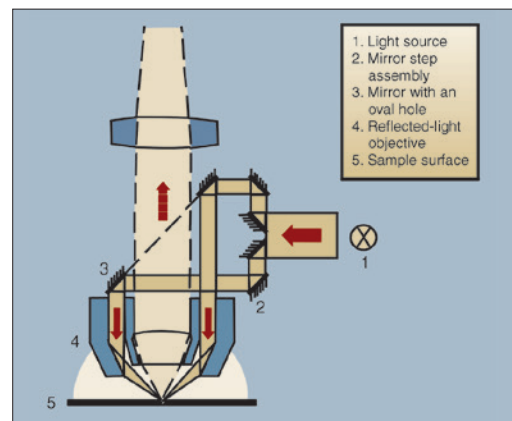*Figure 21.4. Schematic diagram showing the light path in bright field illumination.*

1. Light source
2. Color-neutral beam splitter
3. Reflected-light objective
4. Sample surface
5. Tube lens

Figure 21.5. Schematic diagram showing the light path in dark field illumination.

1. Light source
2. Mirror step assembly
3. Mirror with an oval hole
4. Reflected-light objective
5. Sample surface

that are dark in bright field will be light, appearing to be self luminous in dark field. This highlighting of angled surfaces (pits, cracks, or etched grain boundaries) allows more positive identification of their nature than can be derived from a black image with bright field illumination. Due to the high image contrast obtained and the brightness associated with features at an angle to the optical axis, it is often possible to see details not observed with bright field illumination.

Polarized Light. Because many metals and metallic and nonmetallic phases are optically anisotropic, polarized light is particularly useful in metallography. Polarized light is obtained by placing a polarizer (usually a Polaroid filter) in front of the condenser lens of the microscope and placing an analyzer (another Polaroid filter) before the eyepiece, as illustrated in Figure 21.6. The polarizer produces plane polarized light that strikes the surface and is reflected through the analyzer to the eyepieces. If an anisotropic metal is examined with the analyzer set 90° to the polarizer, the grain structure will be visible. However, viewing of an isotropic metal (cubic metals) under such conditions will produce a dark, "extinguished" condition (complete darkness is not possible using Polaroid filters). Polarized light is particularly useful in metallography for revealing grain structure and twinning in anisotropic metals and alloys (see the appendix for crystal structure information) and for identifying anisotropic phases and inclusions.
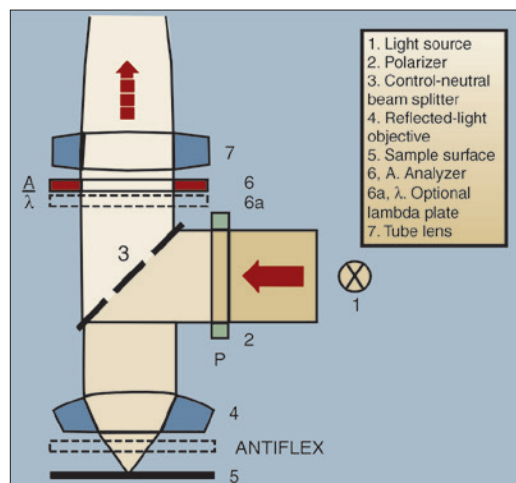
Differential Interference Contrast (DIC). When crossed polarized light is used along with a double quartz prism (Wollaston prism) placed between the objective and the vertical illuminator, Figure 21.7, two light beams are produced which exhibit coherent interference in the image plane. This leads to two slightly displaced (laterally) images differing in phase ($\lambda/2$) that produces height contrast. The image produced reveals topographic detail somewhat similar to that produced by oblique illumination but without the loss of resolution. Images can be viewed with natural colors similar to those observed in bright field, or artificial coloring can be introduced by adding a sensitive tint plate.

As an example of the use of these different imaging modes, Figure 21.8 shows the micro this happens because only a structure of an aluminum bronze specimen that was water quenched from the beta field forming martensite. The specimen was prepared but not etched. A minor amount of relief was introduced in final polishing. Figure 21.8a shows the surface in bright field illumination. Due to the relief present, a faint image of the structure is visible. Dark field, Figure 21.8c, and differential interference contrast, Figure 21.8d, make use of this relief to reveal much more detail than in bright field. However, as the martensite is noncubic in crystal structure, it responds well to crossed polarized light, Figure 21.8b. Of course, not every material can be examined using all four illumination modes, but there are many cases where two or more modes can be effectively utilized to reveal more information than given by bright field alone.
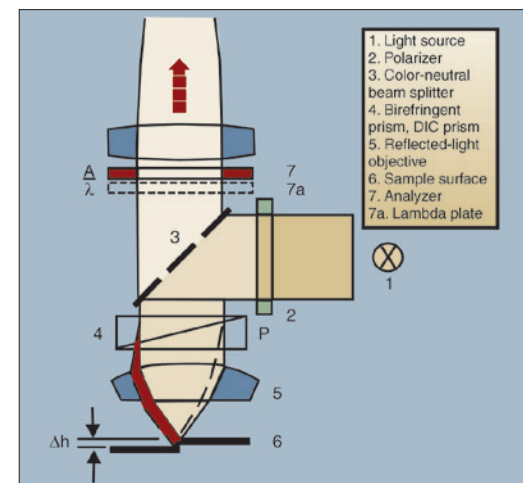
1. Light source
2. Polarizer
3. Control-neutral beam splitter
4. Reflected-light objective
5. Sample surface
6, A. Analyzer
6a, λ. Optional lambda plate
7. Tube lens

Figure 21.6. Schematic diagram showing the light path in polarized light with an optional lambda plate (sensitive tint plate).

1. Light source
2. Polarizer
3. Color-neutral beam splitter
4. Birefringent prism, DIC prism
5. Reflected-light objective
6. Sample surface
7. Analyzer
7a. Lambda plate

Figure 21.7. Schematic diagram showing the light path for differential interference contrast (DIC) illumination.
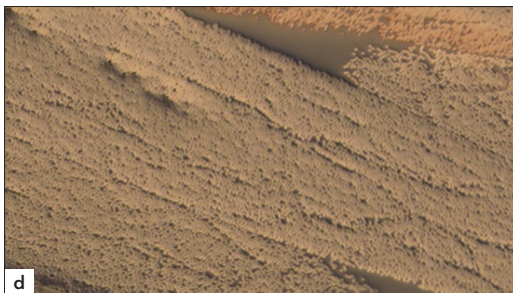
a

b

c

d

Figure 21.8.Carbon fiber reinforced composite images illustrating microscopy imaging modes. a) Brightfield is standard imaging mode for cursory analysis, b) Darkfield highlights cracks that might not be readily visible in bright field.
c) Polarised imaging helps with fiber orientation analysis, whereas d) differential interference contrast provides a topographic view of the imaged surface without loss of resolution.

HELPFUL HINTS FOR OPTICAL MICROSCOPY

It can be difficult to determine which objective you are using with an inverted microscope due to the limited view from above, as the magnification value may not be easily observed. However, each objective has a color-coded ring on it that is usually visible. The colors and corresponding magnifications are: red – 5X, yellow – 10X, green – 20X, blue – 50X, and white – 100X.

IMAGE CAPTURE & ANALYSIS

The progress in computer and video technology has created a movement toward electronic image acquisition. These images can be used in software applications such as word processing or desktop publishing programs allowing for fast report generation and electronic distribution. In addition, associated data can be stored with the images in a database allowing for easy retrieval of information through searches.

When adding imaging capabilities to the laboratory, it is important to consider your goals. Oftentimes, images are just another step in the documentation process. For example, in a failure investigation it is useful to capture the image of a complete component before the sectioning process. Or, an image of the microstructure might be attached to a report as an indication of a pass/fail condition. Additional functionality of an imaging system might include a scale marker overlay and point-to-point or other operator interactive measurements. Fully automated image analysis includes detection of the features of interest based on grey level or color differences as well as morphological characteristics such as size and shape. Automated imaging applications are based on a fundamental series of steps shown in Table 22.1. Depending on your goals, some or all of these steps may be utilized.

Aligned with these imaging goals, Buehler offers a series of upgradeable OmniMet imaging products (Figure 22.1). The OmniMet Capture Basic System provides digital or analog image capture, manual interactive measurements (length, parallel width, area, curve (string lenth), text annotation and scale bars), basic report generation in Microsoft® Word®, and databasing of saved images and measurements. The OmniMet Capture Advanced system expands upon the Capture Basic capabilities by including additional interactive measurements (radius, angle, and counting), adding a results window, and allowing the exporting of data to Microsoft Excel®. The OmniMet Express and OmniMet Enterprise products include automated measurement capabilities in addition to the manual measurements found in the Capture products. OmniMet Express can incorporate a variety of application specific metallographic analysis modules. OmniMet Enterprise is a full featured system including a routine builder which enables the user to generate custom analysis solutions to meet their specific needs.

Figure 22.1. OmniMet Digital Imaging System

Image Capture

Image capture is a term used to describe image acquisition by means of a camera and frame grabber or a digital camera. Because of the many choices of camera types, a video microscopy system must be flexible. Analog CCD cameras, black and white or color, or digital CCD or CMOS are most frequently used. Component video (Y/C or S-Video) and composite video signals and a number of color video standards such as NTSC, PAL and SECAM are generally supported. Images acquired in the materials laboratory are optimized in real time by adjusting brightness, contrast, and color saturation. The analog output camera signal is then digitized utilizing

Table 22.1: Primary Imaging Steps	
Imaging Steps	**Description**
Capture	Capture, load or import an image
Clarification	Develop the necessary contrast and clarity for detecting the features of interest
Thresholding	Detect the features of interest
Binary Operations	Clean-up any detection discrepancies, categorize features and overlay grids
Measurements	Conduct fiels or feature measurements
Data Analysis	Evaluate statictics and relevance of the measurements
Archive	Stor images, annotations and associated measurements in a database
Distribution	Printing or electronic distribution of images and results

an analog input frame grabber board.

Alternatively, digital cameras may be integrated with an IEE 1394 firewire or USB connection. Since the late 1990's digital video cameras have become the preferred choice for scientific imaging applications. This is largely accounted for by two factors: 1) higher pixel densities in the acquired images potentially provide higher spatial resolution and 2) the cost range of digital cameras has merged with the combined cost of an analog camera and specialized capture board. Capturing an image with a digital camera is often a two-stage process, first a preview image is viewed for focusing and selecting the field of interest and then a snapshot of the image is taken. The preview image window displays the image at a lower pixel density and the refresh rate of the camera will determine the amount of lag time between the operator adjusting the microscope and the new image being displayed. In order to be comparable to an analog camera, the refresh rate should approach 25 frames per second.

The resulting image files are based on two primary formats, bit-map and vector. The majority of scientific imaging programs are based on bit-mapped images. Bit-mapped graphics are essentially grids, consisting of rows and columns of pixels. When the image is zoomed to observe details, the individual pixels will become visible. Pixels, or picture elements, are the smallest part of an image that a computer, printer or monitor display can control. An image on a monitor consists of hundreds of thousands of pixels, arranged in such a manner that they appear to be connected. Figure 22.2 displays a cast iron image with a small segment zoomed such that the pixels are observed.

Each pixel in a bitmap is stored in one or more data bits. If an image is monochromatic (black and

white), then one bit is enough to store information for one pixel. If an image is colored or uses various shades of grey, then additional bits are required to store color and shading information. Table 22.2 lists some of the more common bit depths referred to in imaging software and camera literature.

Table 22.2: Bit Depth	
Bits	
1	2^1 or 2 tones, black and white
8	2^8 or 256 grey scale shades
24	2^{24} or 16.7 million colors
32	2^{32} or 4.29 billion colors

When working in grayscale, 256 values are used to represent each shade of grey, starting with black at 0 and transitioning to white at 255. For example, the information stored for the image in Figure 53 would be a combination of the x and y positions and a number from 0 to 255. For color images there are two common models used to numerically represent color, RGB and HLS. Each requires storing three values, in addition to the x and y position of the pixel, in order to recreate the image. In general, you will notice the file sizes for color images are larger than grey scale images of the same pixel density.

The RGB model is based on three primary colors – red, green and blue – that can be combined in various proportions to produce different colors. This scheme is considered additive because, when combined in equal portions, the result is white. Likewise, an absence of all three is black. The three primary colors are each measured as a value from 0-255. The colors produced by combining the three primaries are a result of the relative values of each primary. For example, pure red has a red value of 255, a green value of 0, and a blue value of 0. Yellow has a red value of 255, a green value of 255, and a blue value of 0. This system is modeled as a cube with three independent directions: red, green, and blue, spanning the available space. The body diagonal of the cube contains luminance, in other words, colorless information about how light or dark the signal is.

The HLS model uses the concept of Hue, Luminance, and Saturation. Hue is the color tone. For example, an etchant might highlight the different phases as unique shades of brown or blue. Saturation is complementary to the hue. It describes how brilliant or pure – as opposed to washed out or grayish a particular color is. Luminance is correlated with
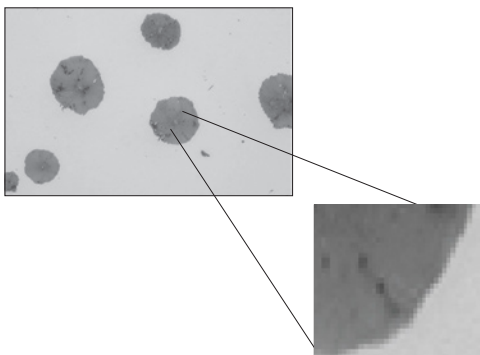

Figure 22.2. A small segment of an image zoomed to display the individual pixels.

the light intensity and viewed as comparable to the greyscale values where zero is black and 255 is white. The HLS color space can be viewed as a double cone, in which the z-axis of the cone is the grayscale progression from black to white, distance from the central axis is the saturation, and the direction or angle is the hue.

The illustration in Figure 22.3 represents pixels of nine different color values. These values are shown in Table 22.3 for the greyscale, RGB, and HLS models. When the pixels are not true grayscale tones, the greyscale number is not given.

Resolution is primarily the imaging system's ability to reproduce object detail by resolving closely spaced features. In digital microscopy, the resolution is examined in terms of both the microscope limitations and the pixel array of the camera. The clarity and definition of a digital image depends largely on the total number of pixels used to create it. Typical pixel array densities range from 640 x 480 to 5120 x 4096. Multiplying the two numbers gives you the total number of pixels used to create the image. Because a large number of pixels leads to sharper images, you might conclude that it is always desirable to capture images with the largest pixel array possible. That is not necessarily always true. The problem with large array images is that they consume a lot of storage space. As a result, these images are not recommended for posting on web sites or email use. It is important to take into account what you intend to do with your images when selecting the appropriate camera.

In order to perform measurements, the image or the image source must first be calibrated. Calibration is achieved by assigning a known distance to a pixel count. When using a light microscope this

is accomplished with a stage micrometer. Each of the objectives will have its own unique calibration factor. If the aspect ratio of the camera pixels is unknown, it can be determined by calibrating in both the x and y direction. If the aspect ratio is known, then calibration only needs to be completed in the x direction. This calibration technique is repeated for each objective or working magnification. It is also possible to calibrate images that have been imported from another source. The only requirement is to have a feature within the image that is of a known dimension. A scale marker is usually the best choice.
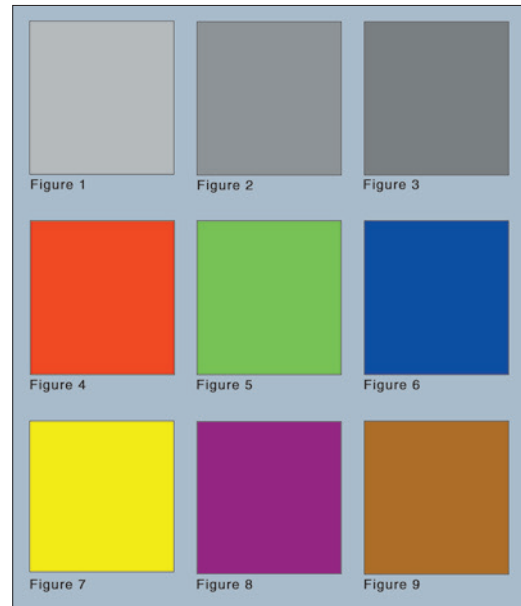

Figure 22.3. A representation of nine pixels with different color values.

Table 22.3: Values based on color models							
			HLS Method			RGB Method	
Pixels	Greyscale	Hue	Luminance	Saturation	Red	Green	Blue
1	192	0	192	0	192	192	192
2	150	0	150	0	150	150	150
3	128	0	128	0	128	128	128
4		0	127	255	255	0	0
5		85	127	255	0	255	0
6		170	127	255	0	0	255
7		60	98	100	255	255	0
8		310	36	79	148	31	129
9		25	100	176	169	113	31

Clarification

Image clarification is also referred to as image enhancement and is largely achieved through the use of greyscale filters. Filters perform one of several standard functions: edge detection, photographic enhancement, or grey level modification. Image clarifications, which modify the values of the pixels across an entire image, are conducted in one of two ways: adjusting limits such as contrast, brightness and gamma or comparing the values of neighboring pixels. The latter is referred to as a convolution or kernel operation. The neighborhood sizes are typically squares of pixels from 3x3, 5x5, 7x7, etc. A typical need is to increase the local contrast at the phase boundaries. Often times, the use of a neighborhood transformation filter results in a more narrow gray scale distribution of a given phase, making the subsequent thresholding or detection process easier.

Operator Interactive Measurements

Interactive measurements require the operator to use a mouse to select the start and end points for each measurement. The most common of these are linear measurements (point-to-point), parallel, radius or diameter, curvilinear, and angle (Figure 22.4). An operator will typically make several measurements over the area of interest. These are either electronically transferred to a file or transcribed by hand. This method is appropriate for a limited number of measurements or where an average is of interest rather than variation. Figure 22.5 represents the most direct way to take an average measurement of thickness using a parallel line tool. One line is aligned with the interface and then the opposite line is aligned with the median of the rough surface.

Figure 22.4. Operator interactive tools founds in imaging software.

In addition to measurements, other annotations are often placed on electronic images. Annotations can be as simple as a label that is referenced in the image caption or a complete text description of a feature within the image. A recommended annotation is a scale marker in the lower corner of the image. This is particularly important for distributing the image electronically because the final size of the printout is at the discretion of the recipient.

Automated Measurements

When more detailed measurements are required or a large quantity of measurements is important, it is useful to automate the process. For example, consider the coating that was measured in Figure 22.5. It is possible to duplicate that measurement as well as to evaluate any variation across the coating and determine the percentage porosity within the coating. A typical process for determining coating thickness is outlined in Figure 22.6 and is described below.
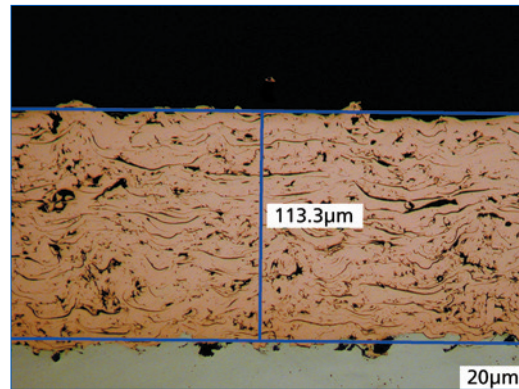

Figure 22.5. An example of an operator estimating coating thickness with the use of a parallel tool.

Thresholding

Thresholding is the method for representing ranges of pixel grey or color values with different color bitplane overlays. The bitplanes are a binary layer placed over the image plane superimposing the phase(s) of interest. Most of the measurements are accomplished on this layer not on the actual image.

The first step of the thresholding process is the development of a threshold histogram. Each pixel is assigned a value and then the frequency is graphed. When working in grayscale with an 8-color image, the same 256 values correspond to luminance. Figure 22.8 demonstrates a yellow bitplane being assigned to the coating (Figure 22.7) based on the HLS values of the pixels as discussed previously.

Binary Operations

After the thresholding process has been accomplished, the different phases and features in an image are represented by different bitplane color overlays. It is possible that more than one phase or feature of interest is detected by the same bitplane color because of a similar gray level range. A binary operation is the process that separates and classifies features within the same bitplane, based on mor-
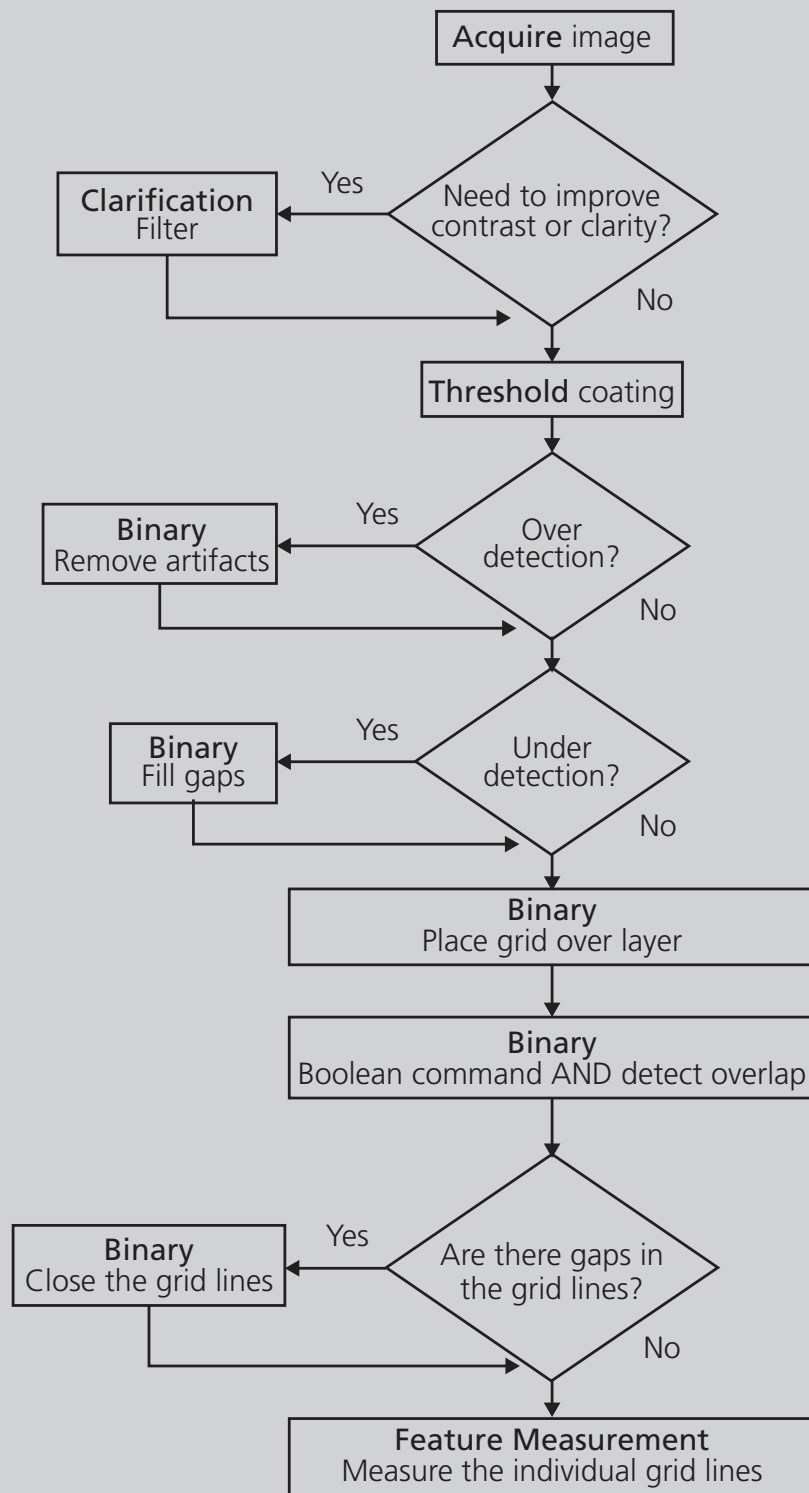
Figure 22.6. A flowchart of the logic used when creating an automated measurement routine for coating thickness.

phology or size. Additionally, not all images are detected as desired. There may be over-detected or under-detected regions that need to modified. In general, binary operations allow the operator to isolate the features that are of interest.

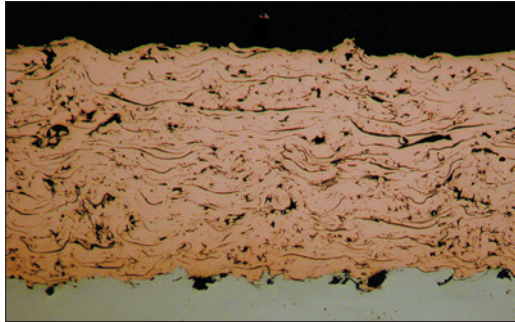Two common measurements on this type of coating

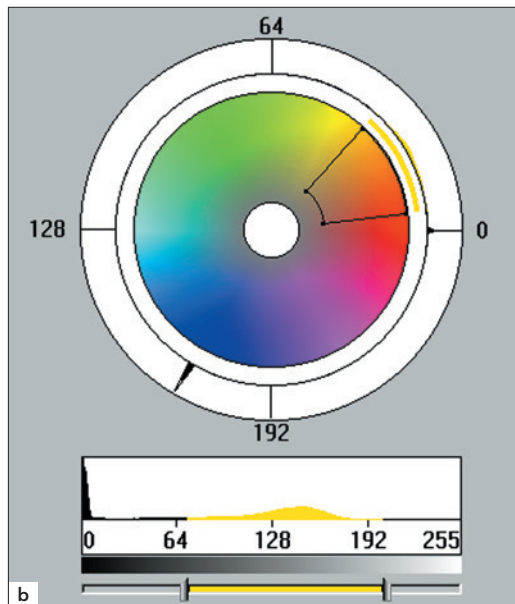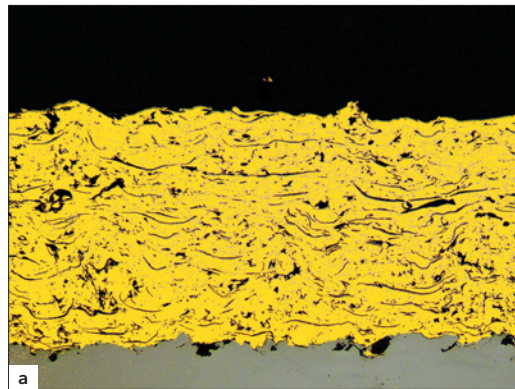*Figure 22.7. A TSC color image captured at 1200 x 1600 pixels.*

Figure 22.8. Thresholding the coating with a yellow bitplane overlay using the HLS color model.

would include the relative area fraction of porosity as well as the average thickness. Before either of these measurements can be made it is necessary to define the total area of the coating. A combination of the binary commands Fill and Close are used to eliminate the holes (undetected areas) that are totally enclosed by the detected bitplane. The thickness is determined by averaging a series of chord measurements across the coating, rather than a single measurement of the yellow bitplane. An additional binary layer is superimposed in the form of a grid using a Grid command. Figure 22.9 demonstrates the effects of these commands on the coating.

To define the individual chords, where the grid overlaps the coating, a Boolean command is employed. Boolean operations perform logical functions between bitplanes. The two most common are OR and AND. AND takes those portions of two bitplanes that overlap and integrates them into a single destination bitplane. In the coating, for example, AND considers only those segments of the red grid bitplane that overlap the yellow coating bitplane. Figure 22.10 shows the red chords which result from the AND command. The OR combines two source bitplanes together to form a single
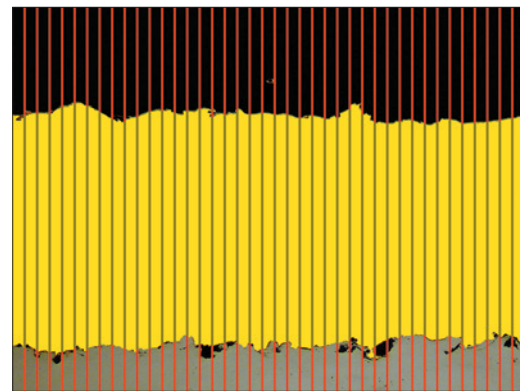

Figure 22.9. The effect of the Binary commands, Close, Fill, and Grid on the detected coating.

destination bitplane. For example, if two different inclusion types, oxides and sulfides, are detected initially, they can be combined to measure the total inclusion content.

One of the most powerful binary functions is the ability to categorize individual features based on shape or size. It can be a single specification (>5µm) or multiple specifications (>2 and <5) separated by an AND or an OR. If a maximum allowable

pore size is specified, all pores that exceed that limit can be flagged. The limit itself can be based on average diameter, maximum diameter, area, aspect ratio, etc. Additionally, the shape of the pore may have some significance. For example, in cast materials, a gas bubble tends to be circular while a shrinkage cavity will have a more complex shape. Common shape factors examine the relationship between the perimeter and area of the feature.
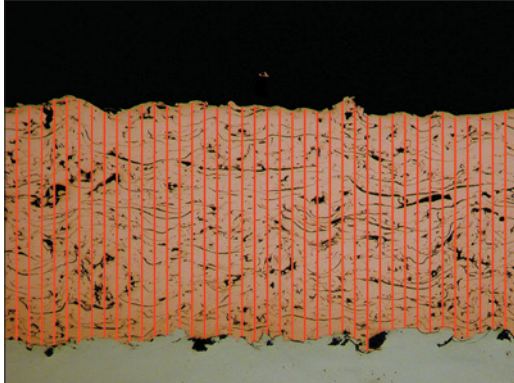

Figure 22.10. The chords that result from the overlapping coating and grid lines.

For this coating, the pores were separated from the elongated oxides to insure that the porosity level was not overestimated.

Returning to Figure 22.10, where grid lines are placed across the coating, the length of all the individual grid lines can be measured. The data can be shown as a histogram offering the minimum, maximum, mean and standard deviation of the measurements across the coating, see Figure 22.11.

Most measurements fall into one of two categories: field or feature-specific measurements. The grid lines across the coating are typical of feature measurements. Each individual feature is measured and then plotted based on frequency in the histogram and contributes to the overall statistics. In the case of grain or particle size measurements, features on the edge of the image are eliminated because the complete size of the feature is unknown.

Field measurements are performed on the entire field of view or a framed portion of the image, providing the sum of the individual measurements in the selected area. Statistical information for field measurements is only generated if multiple fields are analyzed. This can be employed to observe microstructural variations within different fields of
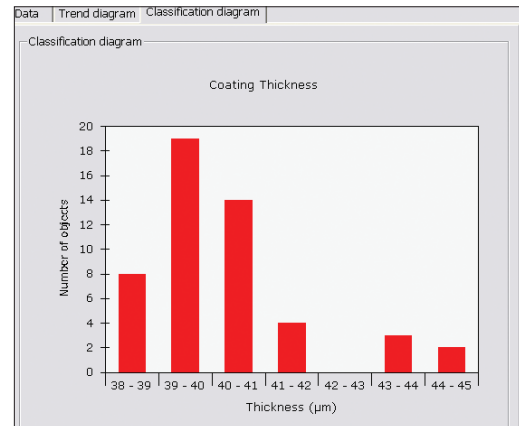

Figure 22.11. The histogram of results representing the chords shown in Figure 22.8.

a specimen. A fairly common field measurement is area percentage. An area percentage measurement results from dividing the number of pixels in the bitplane of interest by the total number of pixels in the image. In the case of the coating, there is a need to measure the area percent porosity relative to the coating area and not the entire image area. This is calculated by dividing the number of pixels in the bitplane of interest, i.e. the pores in the coating, by the total number of pixels representing the coating.

Common Applications

Throughout our involvement in image analysis, a number of applications stand out and may be of interest to most image analysis users. Below is a listing of these applications:

Grain Size. Image analysis provides a rapid and accurate means for determining grain size according to ASTM E112. Even if etching is unable to produce complete grain boundaries, or if there are twins that could skew the data, binary modifications can be employed to make corrections. If a specification cites maximum grain size limitations, the excessively large grains may be transferred to a different bitplane color to provide visual and numerical feedback.

Porosity. Porosity is detrimental to the physical properties of most engineering materials. Image analysis is able to characterize the pores according to the total number of pores, number per unit area, maximum size, average size and the size distribution in the form of a histogram.

Linear Measurements. Simple point-to-point measurements are widely used for making occasional

measurements; however, in cases where a high quantity of measurements and more statistics are required, automated image analysis is time saving. A coating or layer is detected based on the pixel values and then, after binary isolation of the coating, grid lines are superimposed. Using Boolean logic to evaluate the common pixels between the layer and grid lines, the result is many chords representing the thickness at given points in the coating.

Feature Shape and Size. The shape of the graphite constituent in ductile irons is critical. Ductile iron was developed such that the graphite would occur in the form of spherical nodules with the result of dramatically improved mechanical properties. However, variations in chemistry and other factors can cause the nodules to be irregular, leading to some degradation of the properties. The ability to monitor the graphite shape or determine "nodularity" is another ability of image analysis. These same techniques are applicable to any constituent that can be detected.

Phase Percentage. The area percent of various phases in a microstructure influences the properties. The tensile strength of grey iron, for example, is directly related to the percentage of pearlite in its microstructure. In addition, in a single image, multiple phases can be detected, measured and presented in one graph. For example, when evaluating the inclusion content of steels, it would be useful to examine the overall inclusion content as well as isolating particular inclusion types, such as oxides and sulfides.

**HELPFUL HINTS FOR
IMAGE CAPTURE & ANALYSIS**

Determine the required pixel density of a camera system based on the size of the features of interest and acceptable file size

When distributing an image, always add a scale bar

When saving images for analysis at a later time, verify that the image is stored as captured, i.e., without any filters applied.

HARDNESS TESTING

A BRIEF HISTORY OF HARDNESS TESTING

Hardness, as applied to most materials, and in particular metals, is a valuable, revealing and commonly employed mechanical test that has been in use in various forms for more than 250 years. Although hardness is not considered a fundamental physical property, its value and importance cannot be understated. The information from a hardness test can complement, and often be used in conjunction with, other material verification techniques such as tensile or compression testing to provide critical performance information.

Over the years, various methods for determining the hardness of materials have been developed and employed. From early forms of scratch testing to sophisticated automated imaging, hardness testing has evolved into an efficient, accurate and valued material test method.

While testing techniques and hardware have significantly improved in step with rapidly advancing electronics, computer, hardware and programming capabilities, earlier basic forms of hardness testing, such as the simple scratch test, sufficed for the needs of the relevant era.

Some of the earliest forms of bar scratch testing date back to about 1722. These tests were based on a bar that increased in hardness from end to end. The point at which the material being tested could form a scratch on the bar was the determining factor in the specimen's hardness. In 1822, hardness testing techniques were introduced that included scratching material surfaces with a diamond and measuring the width of the resultant line. This approach eventually developed into the Mohs scale, a hardness reference that is still widely used in geological circles.

The Mohs scale [34] consists of 10 minerals, ordered from hardest at 10 (diamond) to softest at 1 (talc). Each mineral can scratch those that fall below it in the scale hierarchy. The Mohs scale is not linear; the difference in hardness between 9 and 10 is significantly more than that between 1 and 2. To put the Mohs scale into perspective, a tangible example is that of hardened tool steel which falls at approximately 7 or 8 on the scale.

During the next 75 years, other more refined ver-sions of the scratch test were introduced including integrated microscope, stage and diamond apparatus that applied increasing loads up to 3 grams. The material to be tested was scratched under load variants and then compared to a standard set of scratches of known value.

Later, indentation type hardness was introduced. One early form developed around 1859, was based on the load required to produce a 3.5 millimetre indent in the material. The first widely accepted and standardized indentation-hardness test was proposed by J. A. Brinell in 1900 [35]. The Brinell hardness test, still widely used today, involves indenting the metal surface with a 1 to 10mm diameter steel ball (now Tungsten Carbide) at loads of up to 3,000 kilograms. The resultant impression, the diameter of the indentation, is measured and a mathematically calculated hardness value derived.

The hardened steel ball of the Brinell test limited the test to materials with a hardness of less than ~450 HBS (~48HRC). The harder tungsten carbide ball extends this range to ~650HBW, but this was not available in 1925.

Figure 23.1. A legacy Wilson "J" Series Rockwell tester, circa 1960s.

Although conceived as an idea in 1908 by a Viennese professor, Paul Ludwik [36], the Rockwell indentation test did not become of commercial importance until around 1914 when brothers Stanley and Hugh Rockwell used a conical diamond indention test based on displacement and applied for a patent for a Rockwell tester design. The principal criterion for this tester was to provide a quick method for determining the effects of heat treatment on steel bearing races.

Patent applications were made and approved between 1919 and 1924 refining the test method, and Stanley Rockwell simultaneously started commercial production of Rockwell testers in collaboration with instrument manufacturer Charles H. Wilson in Hartford, CT, a later Rockwell model is shown in figure 23.1. The company grew into the Wilson

HARDNESS TESTING

Mechanical Instrument Co. and became known as the premium producer of Rockwell testers. After some ownership changes through the latter 1900s, Wilson became an integral part of Illinois Tool Works after it was acquired by Instron in 1993 and subsequently Buehler in 2011. Acquisitions of Wolpert Hardness and Reicherter Hardness have resulted in the engineering and production of cutting edge hardness testing systems.

The Rockwell test remains one of the most efficient and widely used hardness test types in use. However, a major disadvantage of Rockwell type tests are the different scales (15 standard and 15 superficial) that are required in order to evaluate the full material hardness range.

Originally conceived as an alternative to the Brinell method, the Vickers hardness [37] test was developed around 1922 by Smith and Sandland, at Vickers Ltd., a British engineering conglomerate. In designing the new indenter, they chose a geometry that would produce hardness numbers nearly identical to Brinell numbers in the range where both tests could be used. This was a very wise decision as it made the Vickers test very easy to adopt. The ideal d/D ratio (d = impression diameter, D = ball diameter) for a spherical indenter is 0.375. If tangents are drawn to the ball at the impression edges for d/D = 0.375, they meet below the center of the impression at an angle of 136°, the angle chosen for the Vickers indenter. This resulted in a more consistent and versatile hardness test that could evaluate any material (except diamond) and had the very important advantage of placing the hardness of all materials on one continuous scale.

The original Vickers testers were developed for test loads of 1 to 120kgf – which produces rather large indents. Recognizing the need for lower test loads, the National Physical Laboratory (UK) reported on use of lower test loads in 1932. Lips and Sack developed the first low-load Vickers tester in 1936.

In 1939, an alternative to the Vickers test was introduced by Fredrick Knoop at the U.S. National Bureau of Standards. The Knoop hardness [38] test utilized a shallower, elongated form of the diamond pyramid and was designed for use under lower test forces than the Vickers hardness test, allowing for more accurate testing of brittle or thin materials. Both the Vickers and Knoop tests continue as popular hardness analysis methods today.

Hardness testing technology remained fairly consistent throughout the mid to late 1900s, with most systems typically utilizing the dead weight method of applying the test forces. While the deadweight technique is fairly simple, reliable and was widely accepted, the method is not without some ambiguities. Labour-intensive production, coupled with the complexities involved in a highly mechanical systems depending on levers, pivots and guides led to the need for further development and it became evident that other newly developed forms of force regulation used in measurement instrumentation could be applied to hardness testing as well.

With increasing demands for productivity, accuracy, user features and repeatability, closed-loop, load cell technology became a factor in hardness testing. During the 1950s, Instron pioneered the use of closed loop systems on tensile testing instruments. Closed loop systems differ from open loop (deadweight) systems in that they have a means to electronically measure the force being applied during every test and feed (or loop) the information back to the control system. With the teaming up of Instron and Wilson, the ability to adapt the closed loop control effectively to hardness testers led to the development of systems with repeatability never before realized. During the 1990s this technology was introduced first to Rockwell testers, and later to Knoop/Vickers, as well as Brinell systems. Today the technology is a popular and widely used format. Together with advances in computer control, fully automated systems are now available that can achieve higher levels of speed and accuracy than ever before.

A SUMMARY OF HARDNESS TEST METHODS

Brinell Testing

Brinell test methods are defined in the following standards: ASTM E10 and ISO 6506. It is recommended that operators have a current copy of the relevant standards, which are updated regularly.

The Brinell test consists of applying a constant load or force, usually between 187.5 and 3000Kgf, for a specified time (from 10 - 30 seconds) typically using a 2.5 or 10mm diameter tungsten carbide ball, see schematic in Figure 23.3. The time under load (dwell) period is required to ensure that plastic flow of the metal
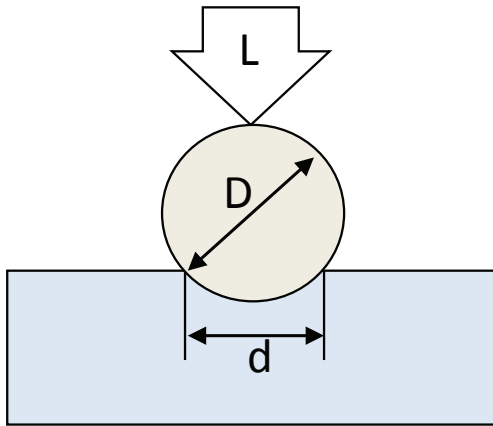
Figure 23.3. Schematic of the Brinell ball indenter making an impression in a test surface.

has ceased. Lower forces and smaller diameter balls are also used in specific applications. Similar to Knoop and Vickers testing, the Brinell test applies only a single test force. After removal of the load, the resultant recovered round impression is measured across the indent at right angles using a low-power microscope or an automatic measuring device and the average value used to calculate hardness.

The actual Brinell hardness (HB) is calculated by factoring the indent size and the test force, such that:

$$HB = \frac{2L}{\frac{\pi D}{2}(D - \sqrt{D^2 - d^2})}$$

Where L = load, D = diameter of the ball indenter and d=diameter of the impression. It is not necessary to make the actual calculation for each test - calculation tables have been published for various combinations of diameters of impressions, load and ball size. In addition various forms of automatic Brinell reading devices are available to perform these tasks.

Brinell testing is typically used in testing aluminium and copper alloys (at lower forces) and steels and cast irons at the higher force ranges. As the Brinell test uses relatively high loads, and therefore relatively large indent, it is frequently used to determine the hardness in circumstances where the overall material properties are being ascertained and local variations in hardness or surface conditions make other methods unsuitable, such as forgings or castings of large parts. Highly hardened steel or other materials

are usually not tested by the Brinell method. As such, Brinell testers are often manufactured to accommodate large parts such as engine castings and large diameter piping. A minimum material thickness of at least 8x the testing depth is recommended (ISO 6506).

Due to the wide number of ball sizes and loads available, it is possible to test a very wide range of hardness values using the Brinell method. This is constrained by the indenter ball itself, which can become deformed by testing harder materials.

It should be noted that there is a relationship between load and ball diameter (L/D^2), whereby tests with load/indenter combinations having the same ratio give the equivalent HB values. Tests with different ratios are not comparable. Errors in Brinell measurement are usually attributable to poor surface condition or operator errors in optical measurement, but due to the large size of indent these errors tend to be limited.
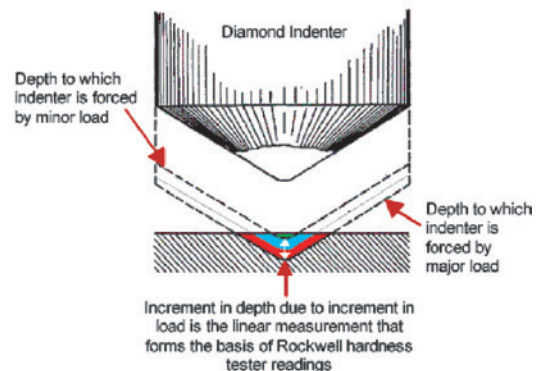
Rockwell Testing

Figure 23.4. Schematic of the Rockwell indentation process, using a diamond brale indenter.

Rockwell test methods are defined in the following standards: ASTM E18 Metals, ISO 6508 Metals, and ASTM D785 Plastics. It is recommended that operators have a current copy of the relevant standards, which are updated regularly.

The Rockwell test is based on the measurement of the depth to which an indenter is forced by a heavy (major) load beyond the depth resulting from a previously applied preliminary (minor) load. The test follows the sequence, see Figure 23.4:

• Application of minor load. A 'zero' position is

Table 23.1: Some common scales in Rockwell and Superficial Rockwell testing			
Scale	Minor Load (kg)	Major Load (kg)	Penetrator
HRA	10	60	Diamond
HRB	10	100	1/16in ball
HRC	10	150	Diamond
HR15N	3	15	Diamond
HR30N	3	30	Diamond
HR45N	3	45	Diamond

recorded.

- Gradual application of load until the major load is reached. The maximum penetration position is recorded.

- Removal of load until the minor load is reached

The resulting Rockwell number represents the difference in depth from the zero datum position as a result of the application of the major load. The entire procedure requires as little as a few seconds (up to 15 for plastics), so the major advantage of the Rockwell test is that results are quickly and directly obtained without the need for a secondary, dimensional measurement requirement.

There are two types of Rockwell test (Table 23.1):

1. Rockwell: the minor load is 10 kgf, the major load is 60, 100, or 150 kgf.

2. Superficial Rockwell: the minor load is 3 kgf and major loads are 15, 30, or 45 kgf.

In both tests, the indenter may be either a diamond cone or tungston carbide ball, depending upon the characteristics of the material being tested. Rockwell hardness values are expressed as a combination of a hardness number and a scale symbol representing the indenter and the minor and major loads. The hardness number is expressed by the symbol HR and the scale designation.

The most common indenter type is a diamond
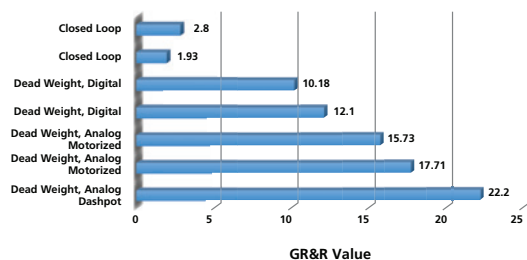
Figure 23.5. GR&R results of various Rockwell testers.

cone ground at 120 degrees for testing hardened steels and carbides. Softer materials are typically tested using tungsten carbide balls ranging in diameters from 1/16 in up to 1/2in. The combination of indenter and test force make up the Rockwell scale. These combinations make up 30 different scales and are expressed as the actual hardness number followed by the letters HR and then the respective scale. A recorded hardness number of 63HRC signifies a hardness of 63 on the Rockwell C scale. Higher values indicate harder materials such as hardened steel or tungsten carbide. These can have HRC values in excess of 70HRC. Rockwell test forces can be applied by either closed-loop load cell or traditional deadweight systems.

Factors Affecting Accuracy, Precision and Bias for Rockwell Testing

One of the primary factors affecting the precision and reliability in the Rockwell test is the test machine itself. Variability in test results is the sum of the variability from the equipment and variability from the operator and environmental conditions. Tests of Gauge Repeatability and Reproducibility (GR&R) can show the degree to which machine variability takes up the available process tolerance. High variability (or a narrow process tolerance) results in high GR&R. A GR&R test on different equipment used for the same process allows the user to directly compare the in-built variability of the machine values (the lower the GR&R value, the better the performance of the equipment), see Figure 23.5. Research has shown [39] that closed-loop loading systems, as well as other design factors and build quality, can greatly improve the performance of the equipment.

Rockwell testing is usually perceived to be one of the simpler testing methods, and indeed in principle it can be very quick and accurate. However, it is important to ensure that the condition of the machine, specimen and set-up are correct to ensure

consistent and accurate results. There are, in fact, many factors that can adversely affect the Rockwell result. For the most part, they can be avoided by ensuring that correct practices are carried out, and by proper calibration and maintenance.

One of the more common sources of error is simply indenter damage. Diamond indenters are very hard, but also relatively brittle, and so it is possible for them to be damaged by a heavy impact. Such damage (or excessive wear of the tip) will change the resistance to penetration, and typically result in a high hardness reading. A deformed Ball indenter can similarly induce high readings.

Another common cause of error is deflection in the loading train. This can come from many sources, all of which will typically cause low readings for Rockwell hardness. If the anvil is damaged, or per-haps has had an indent put into it, then any raised material at the surface will absorb some deflection under loading. Similarly, if there is dirt or grease in the contact area between the anvil and the elevat-ing screw or the elevating screw mechanism itself this will cause excess deflection and change the hardness reading.

The specimen itself is clearly of importance in getting good results. While Rockwell testing is insensitive to surface preparation relative to optical test methods, the better the surface condition the more accurate and reproducible will be the results. The under-side of the specimen must also contact the anvil securely and without interference from debris or other loose material. Specimens should always be cleaned before testing. The specimen must also be held in a stable manner during the test, as any movement of the specimen can significantly affect the result. A movement of 2μm is equivalent to 1 Rockwell point. Excess vibration from the local environment or unsuitable table can cause similar problems.

It should be apparent from the above that if the operator is adequately trained on proper fixturing and testing techniques, the environment is suitable for testing, and that proper cleanliness and main-tenance regimes are carried out then most of the problems described above can be avoided.

Vickers Testing

Vickers and Knoop test methods are defined in the following standards: ASTM E384 and ISO 6507. It is
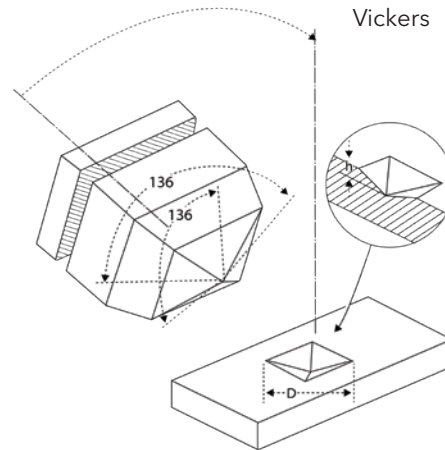

Figure 23.6. Schematic of the Vickers indenter and the shape of an impression.

recommended that operators have a current copy of the relevant standards, which are updated regularly.

In the Vickers test, a load is applied smoothly, with-out impact, forcing a diamond indenter into the test piece, as illustrated in Figure 23.6. The Vickers diamond produces a square based pyramidal shape with a depth of indentation of about 1/7th of the diagonal length. The indenter is held in place for 10 to 15 seconds and then fully unloaded. The physical quality of the indenter and the accuracy of the applied load (which are defined in E 384) must be controlled in order to get the correct results.

The two impression diagonals are measured, usually to the nearest 0.1μm, and averaged. The Vickers hardness (HV) is calculated using:

$$HV = \frac{1854.4L}{d^2}$$

Where the load L is in gf and the average diagonal d is in μm (this produces hardness number units of $gf/\mu m^2$ although the equivalent units kgf/mm^2 are preferred; in practice the numbers are reported without indication of the units). Tables or automatic electronic or imaging measurements are a more common and convenient way to generate Vickers hardness numbers.

HARDNESS TESTING

Since the 1960s, the standard symbol for Vickers hardness per ASTM E 2 and E384, has been HV. This should be used in preference to the older, obsolete symbols DPN or VPN. Rigorous application of the SI system results in hardness units expressed not in the standard, understandable kgf/mm² values but in GPa units that are meaningless to most engineers and technicians. ASTM recommends a 'soft' metric approach in this.

In the Vickers test, it is assumed that elastic recovery does not occur once the load is removed. However, elastic recovery does occur, and sometimes its influence is quite pronounced. Generally, the impression (Figure 23.7) appears to be square, and
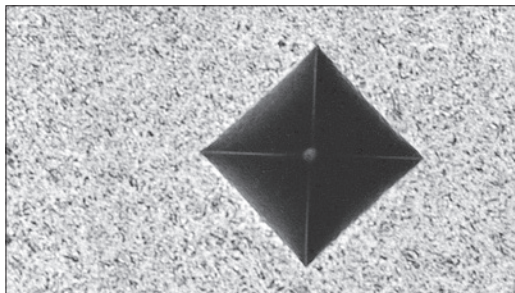
Figure 23.7: Example of a well-formed Vickers indentation (400X).
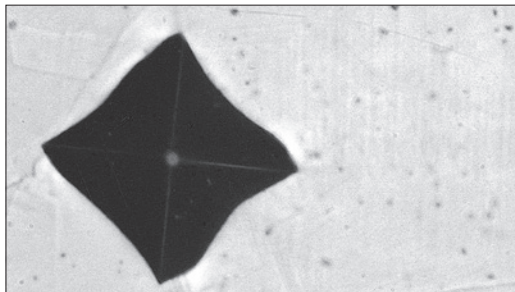
Figure 23.8: Example of a distorted Vickers indentation (400X).

the two diagonals have similar lengths. As with the Brinell test, the Vickers hardness number is calculated based on the surface area of the indent rather than the projected area. If the impression shape is distorted due to elastic recovery (very common in anisotropic materials), Figure 23.8, should the hardness be based on the average of the two diagonals? It is possible to calculate the Vickers hardness based on the projected area of the impression, which can be measured by image analysis. While rigorous studies of this problem are scant in the literature, the diagonal measurement is the preferred approach even for distorted indents, at this time.

The Vickers test has two distinct force ranges,

"Microindentation Vickers" (10 - 1000g) and "Macroindentation Vickers" (1 - 100kg), to cover all testing requirements. The indenter is the same for both ranges; therefore Vickers hardness values are continuous over the total range of hardness for metals (typically HV100 - HV1000). Because the shape of the Vickers indentation is geometrically similar at all test loads, the HV value is constant, within statistical precision, over a very wide test load range as long as the test specimen is reasonably homogeneous.

Microindentation Hardness Testing

Microindentation hardness testing, more commonly (but incorrectly) called microhardness testing, is widely used to study fine scale changes in hardness. Although the term "microhardness" is generally understood by its users, the word implies that the hardness is extremely low, which is not the case. The applied load and the resulting indent size are small relative to bulk tests, but the same hardness number is obtained. Consequently, ASTM Committee E-4 on Metallography recommends use of the term "microindentation hardness testing" which could be given the acronym MHT.

The only difference between standard Vickers testing and micro-indentation testing is the lower
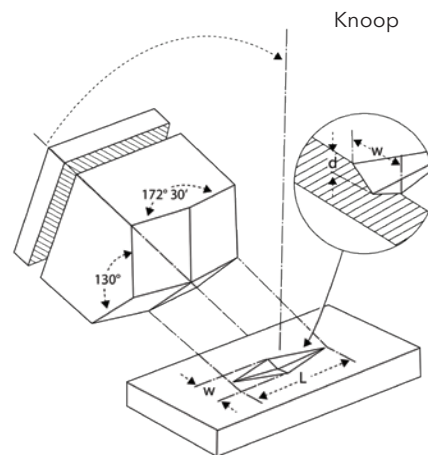

Figure 23.9. Schematic of the Knoop indenter and the shape of an impression

loads (<1kg) used. The indents made in the material are therefore smaller, and this means that more localised areas of the material can be assessed. For example, heat treaters have utilized the technique for many years to evaluate the success of surface hardening treatments or to detect and assess decarburization, by measuring hardness at finely spaced intervals from the specimen surface.

Metallographers and failure analysts use the method for a host of purposes including evaluation of homogeneity, characterization of weldments, as an aid to phase identification, or simply to determine the hardness of specimens too small for traditional bulk indentation tests.

Knoop Testing

The Knoop test uses a rhombohedral-shaped diamond indenter, Figure 23.9. The long diagonal is seven times (7.114 actually) as long as the short diagonal. With this indenter shape, elastic recovery can be held to a minimum. The Knoop test is conducted in the same manner, and with the same tester, as the Vickers test. However, only the long diagonal is measured. This, of course, saves a little time.

The Knoop hardness is calculated from:

$$HV = \frac{14229L}{d^2}$$

where the load L is in gf and the long diagonal d is in μm. The Knoop hardness is expressed in the same manner as the Vickers hardness; i.e., 375 HK0.3 means that a 300 gf load produced a Knoop hardness of 375. (The kgf/mm2 unit information is no longer reported). Again, the symbol HK was adopted in the early 1960's while other terms; e.g., HKN or KHN, are obsolete and should not be used.

Knoop tests, like Microindentation Vickers, are typically performed at test forces from 10 - 1000g. The measured area is used in a formula that includes applied force to determine a hardness value. Tables or automatic electronic or imaging measurements are a more common and convenient way to generate Knoop hardness numbers.

Aside from minor savings of time, one chief merit of the Knoop test is the ability to test thin layers more easily. Around any indent in a material there will be an area of plastic deformation. If this deformed area coincides with an interface (such as the edge of the material) or a similar deformed area from another indent, then the resultant hardness reading will be incorrect. This deformed area is linked to the indent size, and to ensure correct tests it is usually recommended that the indents should be at least 2.5x the diagonal of the indent apart. The elongated form of the Knoop indent means that they can be placed much closer together than a Vickers test using the same load. Furthermore, if the hardness varies strongly with the depth, the Vickers indent can be distorted by this change; that is, the diagonal parallel to the hardness change will be affected by the hardness gradient. Care should also be taken that the thickness of the material is sufficient for testing. For Vickers tests, the thickness should also be at least 2.5x the diagonal measurement. However, because the Knoop indent is shallower than the Vickers at the same load, significantly thinner specimens can be tested.

The down side of the Knoop indent is that the three dimensional indent will change with test load and, consequently, HK varies with load. At higher loads this variation is not substantial. Conversion of HK values to other test scales can only be done reliably for HK values performed at the standard load, generally 500gf, used to develop the correlations. All hardness scale conversions are based on empirical data. Conversions are not precise but are estimates.

Table 23.3: Factors Affecting Precision and Bias in Microindentation Hardness Testing		
Instrument Factors	**Measurement Factors**	**Material Factors**
Accuracy of the applied load	Calibration of the measurement system	Heterogenity of the specimen
Inertia effects, speed of loading	Numberical aperture of the objective	Strength of crystallographic texture, if present
Lateral movement of the indenter or specimen	Magnification	Quality of specimen preparation
Indention time	Inadequate image quality	Low reflectivity or transparency
Indenter shape deviations	Uniformity of illumination	Creep during indentation
Damage to the indenter	Distortion in optics	Fracture during indentation
Inadequate spacing between indents or form edges	Operator's visual acuity	Oil, grease or dirt on indenter or specimen
Angle of indentation	Focusing of the image	

HARDNESS TESTING

Factors Affecting Accuracy, Precision and Bias for Vickers and Knoop

Numerous studies of microindentation hardness test results conducted over a wide range of test loads have shown that results are not constant at very low loads. This problem, called the "indentation size effect", or ISE, has been attributed to fundamental characteristics of the material. However, the same effect is observed at the low load test range (1-10kgf) of bulk Vickers testers [41] and an ASTM interlaboratory "round robin" of indents made by one laboratory but measured by twelve different people, reported all three possible ISE responses [42, 43] , for the same indents!

Many factors (see Table 23.3) can influence the quality of microindentation test results [42]. In the early days of low-load (< 100gf) hardness testing, it was quickly recognized that improper specimen preparation may influence hardness test results. There are cases where improper preparation can lead to higher test results and other cases that can lead to lower results. For many years, it was considered necessary to electrolytically polish specimens so that the preparation-induced damage could be removed, thus permitting bias-free low-load testing. However, the science behind mechanical specimen preparation, chiefly due to the work of Len Samuels [44] , has led to the development of excellent mechanical specimen preparation procedures, and electropolishing is no longer required. Specimen preparation quality becomes more important as the load decreases, and it must be at an acceptable level.

There are several operational factors that must be controlled in order to obtain optimum test results. First, it is a good practice to inspect the indenter periodically for damage, for example, cracking or chipping of the diamond. If you have metrology equipment, you can check the face angles and the sharpness of the tip. Specifications for Vickers and Knoop indenter geometries are given in E384 or ISO 6507. Place the indent in the center of the measuring field, if it is not already there, as lens image quality is best in the center. The light source should provide adequate, even illumination to ensure maximum contrast and resolution.

One source of error in the tests is the alignment of the specimen surface relative to the indenter. The indenter itself must be properly aligned perpen-

dicular (±1°) to the stage plate. Next, the specimen surface must be perpendicular to the indenter. Most testers provide holders that align the polished face perpendicular to the indenter (parallel to the stage). If a specimen is simply placed on the stage surface, its back surface must be parallel to its polished surface. Tilting the surface of the specimen by more than 1° from perpendicular results in non-symmetrical impressions and can produce lateral movement between specimen and indenter.

In most cases, the major source of error is not the mechanical indentation process - although this can occur [45], and it is important to check the performance of your tester regularly using a certified test block. It is best to use a test block manufactured for microindentation testing and certified for both the test type (Vickers or Knoop) and the load that you intend to use. However, because of the light loads used, vibration can contribute to loading inaccuracy. Micro-indentation hardness testers should always be placed on a level, sturdy (preferably dedicated anti-vibration) surface. The surface should not be in contact with an adjoining table or wall, as vibration can be transferred by such prosaic events as a slamming door, or by someone working on an adjoining surface. Similarly, the floor on which the test table is placed should be solid.

Figure 23.10. Wilson® VH3100 Automatic Vickers/Knoops Hardness Tester.

The greatest source of error is in the process of actually measuring the indent, as has been documented in an ASTM inter-laboratory tests [41, 42]. Because of the method of defining HV and HK, where we divide by d2, measurement errors become more critical as d gets smaller.

For the Knoop indent, the main problem in resolving

the diagonal accurately is the image contrast at the indent tips, which can be very narrow. This tends to result in undersized indents that, along with the variable indent shape, both result in increasing HK with decreasing test load. For the Vickers test, although both under-sizing and over-sizing can occur, experience suggests that oversizing is much more commonly encountered for low loads and small indents.

Automation

Manual microindentation hardness testing can be tedious for an operator, so anything that can be done to simplify testing is valuable, especially for laboratories that do substantial testing. Many adjuncts to indent measurement have been tried and a variety of such systems are available. There has been considerable interest in applying image analyzers to the indent measurement task. Further, with stage automation, it is possible to automate the indenting process itself, and with the same equipment. Figure 23.10 shows a Wilson automated hardness testing system. Such systems can be used in fully automatic, semiautomatic or manual modes, depending upon the nature of the testing. An automated system can be programmed to make multiple indents in either a defined pattern or statistical groups.

The system then makes the indents at the requested loads and locations, measures each indent, calculates the hardness (and desired conversions to other scales), and prints/plots the results. Statistical analysis can also be done.

In general, Vickers indents are easier to measure by image analysis than Knoop due to the lower contrast at the tip of the Knoop indents (leads to undersizing and higher HK values). Metal flow (plastic deformation) or surface debris around the indent edges can also interfere with measurement precision. Vickers indents, like the ones shown in Figure 23.6, exhibit excellent contrast and shape and are easily measured. If the magnification is too high (and this may be influenced by the numerical aperture of the objective) for a given indent size, image contrast suffers and correct detection of the indent will be very difficult. On the other hand, if the indent is very small on the screen, it will be hard for the system to detect it automatically. As such, properly selected magnifications are very important to achieving accurate results over a range of conditions. Some systems are available with "zoom" objectives, that effectively ensure the optimum magnification is always selectable.

Microindentation hardness testing is a very valuable tool for the materials engineer but it must be used with care and a full understanding of the potential problems that can occur.

- Always try to use the highest possible load depending upon the indent spacing or closeness to edges.

- Try to keep indents greater than 20μm in diameter, as recommended in ASTM standard E384 and ISO 6507.

If you have not read the standards mentioned above, or it has been some time since you read it (standards must be reviewed and revised, if necessary, regularly), get the latest copy and go over it. Sections such as 'Precision and Bias' and appendix X1 of E384 contain a wealth of practical advice.

HELPFUL HINTS FOR MICROINDENTION HARDNESS TESTING

If you suspect that the test results obtained on a specimen are questionable, verify the tester using a certified test block. When testing thin coatings, use the Knoop indenter with the long axis parallel to the surface. If you have a specimen where the hardness changes rapidly, Vickers indents may be distorted badly in the direction of the hardness gradient. Switch to a Knoop indenter and place the long axis perpendicular to the hardness gradient.

LABORATORY SAFETY

The metallographic laboratory is a relatively safe working environment; however, there are dangers inherent to the job. Included in these dangers is exposure to heat, acids, bases, oxidizers and solvents. Specimen preparation devices, such as drill presses, shears and cutoff saws, also present hazards. In general, these dangers can be minimized if the metallographer consults documents such as ASTM E 2014 (Standard Guide on Metallographic Laboratory Safety) and relevant Safety Data Sheets (SDS) before working with unfamiliar chemicals. Common sense, caution, training in basic laboratory skills, a laboratory safety program, access to safety reference books – these are some of the ingredients of a recipe for laboratory safety. Table 24.1 lists the main requirements for a comprehensive safety program.

Safe working habits begin with good housekeeping. A neat, orderly laboratory promotes safe working habits, while a sloppy, messy work area invites disaster. Good working habits include such obvious, commonsense items as washing the hands after handling chemicals or before eating. Simple carelessness can cause accidents (see Figure 24.1). For example, failure to clean glassware after use can cause an accident for the next user. Another common problem is burns due to failure to properly clean acid spills or splatter.

Most reagents, chemical or electrolytic polishing electrolytes, and solvents should be used under a ventilation hood designed for use with chemicals.

Figure 24.1. Carelessness in the laboratory can result in dangerous chemical spills and a clean-up nightmare.

Many of these chemicals used in metallography can cause serious damage on contact. It is best to assume that all chemicals are toxic and all vapors or fumes will be toxic if inhaled or will be damaging to the eyes. A hood will prevent the working area from being contaminated with these fumes. However, you will not be protected when your head is inside the hood. In most cases, a protective plastic or shatter-proof glass shield can be drawn across the front of the hood for further protection from splattering or any unexpected reactions.

All laboratories should be equipped with a shower and eyewash for emergency use. This equipment should be near the work area so that the injured can reach it quickly and easily. Fire alarms and fire extinguishers (CO_2 type) should be available and tested periodically. A good first aid kit and a chemical spill treatment kit should be readily available.

Table 24.1. Elements of a Comprehensive Metallography Laboratory Safety Plan

1. Emergency Response
   a. First Responders
   b. Emergency Phone Numbers
   c. First Aid
   d. Spills
2. Chemical Procurement, Distribution, and Storage
   a. SDS Management
   b. Labeling/Storage
3. Laboratory SOPs
   a. Handling Chemicals
   b. Mixing Chemicals
   c. Rules
   d. Hygiene
4. Employee Health and Exposure
   a. Personal Protective Equipment
   b. Monitoring of exposure levels
   c. Medical Evaluations
   d. Additional protections for employees working with particularly hazardous substances (carcinogens, toxins)
5. Disposal of Hazardous Materials
   a. Etchants and Chemicals
   b. Recirculating Tanks
   c. Lubricants and Abrasives

6. Equipment Use (JSAs)
   a. Sectioning
   b. Mounting
   c. Grinding and Polishing
   d. Electropolishing
   e. Etching
7. Facility
   a. Facility and Equipment Maintenance
   b. Housecleaning
   c. Fume Hood Air Flow
   d. Fire Extinguishers and Fire Protection
   e. Spill Response
   f. Showers and Eye Washes
   g. Air Lines and Filters
   h. Plumbing
   i. Electricals
8. Employee Training
   a. Safety
   b. Indications and Symptoms of Exposure
   c. Job Functions
9. Scheduled and Documented Safety Inspections and Meetings

Laboratory Equipment

Specimen preparation devices used in metallographic laboratories are generally quite safe to use. Information supplied by the manufacturer usually describes safe operating procedures. It is good laboratory practice to prepare a job safety analysis (JSA) detailing potential hazards and describing the safe operating procedure for each piece of equipment. This information should be provided to all users and it must be revised and reviewed periodically.

Band saws or abrasive cutters are commonly used by metallographers. The cutting area of band saws is exposed and potentially dangerous. Your hands should never be used to guide the work piece during cutting. A guiding device or block of wood should always be used between the work piece and your hands. After cutting is completed, the saw should be turned off before pieces near the blade are removed. Samples should be handled carefully, because considerable heat can be generated. In addition, sharp burrs are often present, which should be carefully removed by filing or grinding. Abrasive cutoff saws are safer to use because the cutting area is closed off during use. The chief danger is from flying pieces from a broken wheel. Fortunately, the closed cover contains these pieces within the cutting chamber. Wheel breakage usually occurs when the part is not firmly clamped in place or if excessive pressure is applied, a bad practice from the standpoint of specimen damage as well.

Dust produced during grinding of metals is always dangerous. For certain metals, like beryllium, magnesium, lead, manganese, and silver, the dusts are extremely toxic. Wet grinding is preferred both for dust control and for preventing thermal damage to the specimen. Bench grinders must be firmly mounted to prevent sudden movement. Care must be exercised to avoid grinding one's fingers or striking the edge of a grinding belt, which will cause painful lacerations. With nearly all materials, wet grinding is preferred and produces best results. For routine handling of dangerous metals, grinding should be done wet under a ventilation hood. Waste must be handled carefully and disposed of properly. Radioactive materials require special remote- handling facilities and elaborate safety precautions.

A drill press is frequently used in the laboratory. Drilling holes in thin sections requires secure clamping; otherwise the sample can be grabbed by the drill and spun around, inflicting serious lacerations. Hair, ties, and shirt cuffs can become tangled in a drill, inflicting serious injuries. Safety glasses should always be worn when using drill presses or when cutting or grinding. Mounting presses or laboratory heat-treatment furnaces present potential burn hazards. It is a good practice to place a "hot" sign in front of a laboratory furnace when it is in use. Gloves should be worn when working with these devices. Modern mounting presses that cool the cured resin back to near room temperature dramatically reduce the potential for burns.

It is occasionally necessary to heat solutions during their preparation or use. Although Bunsen burners are commonly employed for this purpose, it is much safer to use a hot plate or water bath and thus avoid the use of an open flame. If a Bunsen burner is used, the flame should never be applied directly to a flask, beaker, or dish. Plain- or asbestos-centered wire gauze should always be placed between the flame and the container.

Personal Protective Equipment (PPE)

Metallographers must take certain precautions to insure their personal safety. A laboratory coat is useful for protecting the operator's clothing, and should be changed regularly and cleaned professionally. When handling caustics, a rubberized or plastic coated apron provides better protection. Gloves should be worn when handling bulk samples, working with hot material, or using hazardous solutions. Lightweight surgeons' gloves are very popular, because the operator retains the ability to "feel." Many metallographers wear these to protect their skin when mounting with epoxies and polishing with oxide suspensions. When using these gloves with chemicals, always inspect for holes, as they are easily punctured. Thick rubber gloves are often used for handling specimens during macroetching, chemical polishing, pickling, etc. The gloves should always be checked first for small holes or cracks, because gloves can impart a false sense of security. The operator's hands generally perspire when using rubber gloves and it is sometimes difficult to tell if the moisture is due solely to perspiration or to leakage. Safety glasses should be worn during processes that generate particulate matter. Goggles are appropriate for use with chemicals, and a chemical face shield is recommended when handling large quantities of hazardous liquids. The appropriate PPE will be specified on the SDS for most laboratory chemicals and products.

LABORATORY SAFETY

Chemicals, Storage and Handling

Many of the chemicals used in metallography are toxic, corrosive, flammable, or potentially explosive. Whenever possible, purchase small quantities that are likely to be used within a reasonably short time. Flammable solvents should be stored in fireproof steel cabinets. Acids and bases should be stored separately, again in fireproof steel cabinets. Strong oxidants must not be stored along with acids, bases or flammable solvents.

Reagent-grade chemicals or solvents of highest purity are recommended. Although more expensive, the amounts used are small and the gain in safety and reliability compensates for the cost difference. Chemicals may deteriorate during storage. Exposure to light can accelerate deterioration of some chemicals. Hence, they should be stored in a closed metal cabinet.

Etchants

Most laboratories mix commonly used reagents in quantities of 250 to 1000mℓ and then store them as stock reagents. Many reagents can be safely handled in this manner. It is best to store only those reagents that are used regularly. Glass-stoppered bottles are commonly used as stock reagent bottles. If these bottles are opened regularly, the stopper will not become "frozen". However, if they are used infrequently, a frozen stopper often results. Holding the neck of the bottle under a stream of hot water will usually loosen them. If thermal expansion does not free the stopper, the stopper can be gently tapped with a piece of wood. Glass bottles with plastic screw-on tops can be used so long as the solution does not attack the plastic. These bottles are useful for holding solutions, such as nital, that can build up gas pressure within a tightly stoppered bottle. A small hole can be drilled through the cap top to serve as a pressure relief vent. Tightly stoppered bottles of nital and some other solutions have exploded as the result of pressure buildup. Be certain that the reagent is safe to store and store only small quantities. All bottles should be clearly labeled. Polyethylene bottles are required for etchants containing hydrofluoric acid, which attacks glass.

Most recipes for etchants or electrolytes list the ingredients by weight if they are solids and by volume if they are liquids. In a few cases, all amounts are given in weight percentages. In most cases, reagent compositions are not extremely critical. An ordinary laboratory balance provides adequate weighing accuracy, while graduated cylinders provide acceptable accuracy for volumetric measurements. These devices should be cleaned after use to prevent accidents to the next user. For weight measurements, a clean piece of filter paper, or a cup, should be placed on the balance pan to hold the chemical, to protect the pan surface, and to facilitate transfer to the mixing beaker. A large graduated beaker is usually employed for mixing solutions.

Figure 24.2. When mixing etchants, use a magnetic stirring plate with a magnetic bar for stirring. Slowly add the liquid ingredients to the solvent by dripping them down a glass stirring rod. If the solution is more dangerous than this one, wear protective gloves and use a face shield. If mixing generates substantial heat, it is a good practice to place a cooling jacket around the beaker.

With many etchants, the mixing order is important, especially when dangerous chemicals are used. When water is specified, distilled water should always be used, because most tap water contains minerals or may be chlorinated or fluorinated. Tap water can produce poor results or unexpected problems. Cold water should always be used, never warm or hot water, which can cause a reaction to become violent. In mixing, one should start with the solvents, such as water and alcohol; then dissolve the specified salts. A magnetic stirring device is of great value, as shown in Figure 24.2. Then, the dangerous chemicals, such as acids, should be added carefully and slowly while the solution is being stirred. Whenever sulfuric acid (H_2SO_4) is specified, it should be added last. It should be added slowly, while stirring, and it should be cooled, if necessary, to minimize heating. Never just pour one liquid into another, as shown in Figure 24.3. If sulfuric acid is added to water without stirring, it can collect at the bottom of the beaker and enough local heating can occur to throw the contents out of the beaker.

Figure 24.3. Illustration of a bad mixing practice. The acid ingredient was poured into an empty acid bottle and the solvents were added without stirring or cooling. The solution may erupt in the metallo-grapher's face at any moment. It is recommended to keep the hands clear of the area and remove rings.

The literature contains references to a great many formulas for etchants, chemical polishes, and electrolytes that are potentially dangerous or extremely dangerous. Few of these references contain comments regarding safe handling procedures or potential hazards. Fortunately, metallographic applications involve small quantities of these solutions, and accidents do not usually produce catastrophic results. However, even with small solution volumes considerable damage can be, and has been, done. Table 24.2 lists examples from the literature [27-29] of chemical polishing solutions, electrolytic polishing solutions and etchants that have been involved in accidents. Table 24.3 lists a number of commonly used chemicals and incompatible chemicals.

Solvents

Numerous organic solvents are used for cleaning or are ingredients in chemical or electrolytic polishing solutions or etchants, where they are used to control ionization or the speed and mode of attack. Commonly employed solvents include water, acetone, ethyl ether, ethylene glycol, glycerol (glycerin), kerosene, petroleum ether, trichloroethylene, butyl cellosolve, and alcohols, such as amyl alcohol, ethanol, methanol, and isopropyl alcohol. Most are flammable and their vapors can form explosive mixtures with air. They should be kept closed when not in use and should be stored in a cool place away from heat and open flames.

Acetone (CH_3COCH_3) is a colorless liquid with a fragrant mint-like odor. It is volatile and highly flammable. It is an irritant to the eyes and the mucous membranes. Its vapor is denser than air and can travel along the ground and can be ignited at a distance. Acetone can form explosive peroxides on contact with strong oxidizers such as acetic acid, nitric acid and hydrogen peroxide. It is an irritant to the eyes and respiratory tract, and will cause the skin to dry and crack.

Butyl cellosolve ($HOCH_2CH_2OC_4H_9$), or ethylene glycol monobutyl ether, is a colorless liquid with a rancid odor that is used in electropolishing solutions. It is combustible and may form explosive peroxides. It is toxic in contact with the skin, can be absorbed through the skin, can cause serious damage to the eyes, irritation to the skin and respiratory tract.

Carbitol ($C_2H_5OCH_2CH_2OCH_2CH_2OH$), or diethylene glycol monoethyl ether, is a colorless, viscous solvent that is compatible with water and is used in electropolishing solutions. It irritates the skin, eyes, mucous membranes, and upper respiratory tract, and is harmful if inhaled or swallowed.

Ethylene glycol ($HOCH_2CH_2OH$) is a colorless, hygroscopic liquid with a sweet taste (but do not swallow as it is poisonous) that reacts with strong oxidants and strong bases. It is flammable. The substance irritates the eyes, skin, and the respiratory tract.

Glycerol (glycerin) ($CH_2OHCHOHCH_2OH$) is a colorless or pale yellow, odorless, hygroscopic, syrupy liquid with a sweet, warm taste. It is relatively nontoxic and nonvolatile but can cause iritis (inflammation of the iris). It is combustible and is a moderate fire hazard. Glycerol should never be used in anhydrous solutions containing nitric and sulfuric acids, because nitroglycerin can form. Glycerol should not be used with strong oxidizing agents, such as chromium trioxide and potassium permanganate, as an explosion may occur. Glycerol is often added to aqua regia (glyceregia). This mixture decomposes readily and should be discarded immediately after use. This etchant should not be allowed to stand for more than about 15 minutes after mixing.

Kerosene is occasionally employed in grinding samples and with diamond paste as a lubricant. Only the deodorized form should be used. It is flammable, but the vapors do not readily explode. Contact defattens the skin and can cause dermatitis, irritation or infections.

Trichloroethylene ($CHCl:CCl_2$) is a stable,

LABORATORY SAFETY

Table 24.2: Chemical and Electrolytic Polishing Solutions and Etchants Known to be Dangerous

Solution	Use	Problems
5 parts Lactic Acid 5 parts HNO_3 2 parts water 1 part HF	Chemical polishing solution for Zr	Lactic and nitric acids react automatically. Explosion can occur if stored
50 parts Lactic Acid 30 parts HNO_3 2 parts HF	Chemical polishing solution for Ta, Nb, and alloys	Lactic and nitric acids react automatically. Explosion can occur if stored
3 parts Perchloric Acid 1 part Acetic Anhydride	Electropolishing solution for Al	Mixture is unstable and can, and has, exploded with heating or in the presence of organic compounds adding to the potential hazard.
60-90 parts Perchloric Acid 10-40 parts Butyl Cellosolve	Electropolishing solution	Solution will explode at room temperature. Solutions with ≤30% $HClO_4$ will be safe if T is <20°C
100g CrO_3 200mℓ water 700mℓ Acetic Anhydride	Electropolishing solution	CrO_3 was dissolved in water, cooled to about 20°C; the acetic anhydride was added very slowly with stirring. The solution became warm to the touch. About 20 seconds later it erupted from the beaker.
1 part HNO_3 2 parts Methanol	Electropolishing solution for Muntz (Cu-40% Zn) metal	Mixture is unstable and cannot be stored.
20mℓ HF 10mℓ HNO_3 30mℓ Glycerol	Etchant fo Nb, Ta, Ti, V, Zr	This etchant is unstable. At 20°C, it reacted after 18 hours. at 30-35°C, it reacted after 8 hours with violence. At 100°C, it will react after 1 minute.
20-30mℓ HCl 10mℓ HNO_3 30mℓ Glycerol	Etchant for Ni and stainless steels	Two incidents occurred when a violent reaction resulted producing NO_2 and a spray of acid after the etch was left standing for 2-3 hours.
40mℓ Acetic Acid 40mℓ Acetone 40mℓ HNO3	Etchant for Ni	A closed bottle exploded about 4 hours after it was mixed. Authors state that solutions without the acetic acid also cannot be stored.
10mℓ HNO3 10mℓ Acetic Acid 20mℓ Acetone	Etchant	The solution reacted spontaneously about 2 minutes after mixing with evolution of heat and fumes (nitrous and nitric oxides). The mixed acids were poured into the acetone. The beaker was externally cooled.
50mℓ Nitric Acid 90mℓ Isopropyl Alcohol	Etchant	Mixtures have exploded violently after mixing or about 20 minutes after mixing.

Table 24.3: Some Incompatible Chemicals

Chemical	Use in Metallography	Do Not Mix with the Following:
Acetid Acid	Chemical polishing, Electrolytic polishing	Chromic acid, glycol, hydroxol compounds, nitric acid, peroxides, permanganates
Acetone	Degreasing, cleaning, etchants	Concentrated solutions of nitric and sulfuric acid
Chromic Acid	Electropolishing	Acteic acid, flammable liquid, glycerol
Hydrogen Peroxide	Chemical polishing, etchants	Flammable liquid, organic materials
Nitric Acid (conc)	Chemical polishing, etchants	Acetic acid, chromic acid, flammable liquids, isopropyl alcohol
Perchloric Acid	Electropolishing	Acetic anhydride, alcohol, some organics, oil, grease
Sulfuric Acid	Etchants	Methyl alcohol, potassium chlorate, potassium perchorate and potassium permanganate

colorless liquid with a chloroform-like odor. Effective laboratory ventilation is necessary. At ambient temperatures it is nonflammable and nonexplosive, but becomes hazardous at higher temperatures. In the presence of strong alkalies, with which it can react, it can form explosive mixtures. In the presence of moisture, the substance can be decomposed by light to corrosive hydrochloric acid. It is carcinogenic to humans, and toxic when inhaled or ingested, which may cause acute poisoning.

Amyl alcohol ($CH_3(CH_2)_4OH$), or 1-Pentanol, is a colorless liquid with noxious odor. It is flammable and the vapors may form explosive mixtures at elevated temperatures. The substance reacts violently with strong oxidants and attacks alkaline metals. The fumes are irritating to the eyes, upper respiratory tract, and skin. The substance is toxic through ingestion, inhalation, or absorption through the skin.

Ethyl alcohol (CH_3CH_2OH), or ethanol, is a colorless, inoffensive solvent commonly used in metallography. Ethanol is miscible with water and rapidly absorbs up to 5% water from the air. The denatured version is less expensive and contains 5% absolute methanol and is suitable for any recipe requiring ethyl alcohol. It is a dangerous fire hazard, and its vapors are irritating to the eyes and upper respiratory tract. High concentrations of its vapor can produce intoxication. Because ethanol is completely burned in the body, it is not a cumulative poison like methanol.

Methyl alcohol (CH_3OH) is an excellent, non-hygroscopic solvent, but it is a cumulative poison. Ingestion, inhalation or absorption through the skin in toxic levels can damage the central nervous system, kidneys, liver, heart, and other organs. Blindness has resulted from severe poisoning. It is particularly dangerous because repeated low-level exposures can also cause acute poisoning as a result of accumulation. Ethanol should be used whenever possible. When using methanol, always work under a ventilation hood. Mixtures of methanol and sulfuric acid can form dimethyl sulfate, which is extremely toxic. Solutions of methanol and nitric acid are more stable than mixtures of nitric acid and higher alcohols.

Isopropyl alcohol [$CH_3CH(OH)CH_3$], also known as 2-propanol, is a clear, colorless liquid that, like ethanol, does not accumulate in the body, although it does have a strong narcotic effect. It is a flammable liquid and is a dangerous fire hazard. Metallographers have used it as a substitute for ethanol but isopropyl alcohol has quite different characteristics and should not be used. Fatal injuries and explosions have been reported due to its use.

Acids

Inorganic and organic acids are common constituents in chemical and electrolytic polishing solutions and in etchants. The inorganic, or mineral acids, including the very familiar acids such as hydrochloric, nitric, perchloric, phosphoric, and sulfuric, are highly corrosive and poisonous. They should be stored in a cool, well-ventilated location away from potential fire hazards and, of course, away from open flames. They should not be stored in a location that receives direct sunlight. When the pure acids contact metals, most liberate hydrogen gas – a fire and explosion hazard. The organic acids are naturally occurring substances in sour milk, fruits, and plants and include the following acids: acetic, lactic, citric, oxalic, and tartaric.

Hydrochloric acid (HCl), commonly used in metallography, is a colorless gas or fuming liquid with a sharp, choking odor. It is very dangerous to the eyes and irritating to the nose and throat. It attacks the skin strongly causing severe burns.

Nitric acid (HNO_3), also commonly used in metallography, is a colorless or yellowish fuming liquid, highly toxic and dangerous to the eyes. If it contacts organic material or other easily oxidizable materials, it can cause fires and possibly explosions. When it reacts with other materials, toxic oxides of nitrogen are produced. The oxides, which vary with the conditions, include nitrous acid, nitrogen dioxide, nitric oxide, nitrous oxide, and hydroxylamine. A commonly encountered problem involves pouring nitric acid into a graduated cylinder that contains some methanol or ethanol from prior use. The brown fumes given off are quite harmful. Mixtures of nitric acid and alcohols higher than ethanol should not be stored. Mixtures of concentrated nitric and sulfuric acids are extremely dangerous, while strong mixtures of nitric acid and glycerin or glycols can be explosive. Aqua regia, a mixture of one part nitric acid and two to four parts hydrochloric acid, forms several products including nitrosyl chloride, an exceptionally toxic gas. Aqua regia is a popular etchant but must be used with care under a hood.

Ethanol with additions of up to 3% nitric acid (nital)

can be safely mixed and stored in small quantities. Higher concentrations result in pressure buildup in tightly stoppered bottles. Explosions of 5% nitric acid in ethanol have occurred as a result of failure to relieve the pressure. If higher concentrations are desired, they can be mixed daily, placed in an open dish, and used safely. Discard the etchant at the end of the day. Mixtures of methanol with up to 5% nitric acid are safe to use and store in small quantities. Mixtures of methanol with more than 5% nitric acid if heated are subject to violent decomposition. Mixtures of 33% nitric acid in methanol have decomposed suddenly and violently.

Never add nitric acid to isopropyl alcohol. Anderson [27] reported that a liter bottle of 5% nitric acid in isopropyl alcohol was mixed and placed in a cabinet. Although this had been done many times in the past without problems, twenty minutes later the bottle exploded destroying the cabinet, other stored bottles, and throwing debris up to 20 feet away. Anderson [27] also reported that a metallographer was pouring a freshly mixed liter of 5% nitric acid in isopropyl alcohol into another bottle when it exploded. The person died within three hours without being able to tell anyone what happened. As with the other explosion, this same procedure had been performed many times previously without mishap. Anderson recommends avoiding use of isopropyl alcohol completely.

Most metallographers consider nital to be very safe to use, and indeed it is. However, even with such an apparently safe solution, one can have accidents. One such accident occurred when an employee, not a skilled metallographer, was replenishing a stock of 5% nitric acid in ethanol using a procedure that he had claimed to have performed many times previously (he was not taught the safe way to mix nital as it was a union chemist's job to mix nital i.e., not his job). The worker began by adding the desired volume of concentrated nitric acid into the container that contained a small residual amount of stale 5% nital. To his surprise, the contents began "boiling" and spewing out of the container along with dense, brown fumes. The acid splashed the worker, resulting in burns on his forehead, face and eyes. The small amount of aged nitric acid solution (the concentration may have been increased due to evaporation of the alcohol), present in the container when the fresh acid was added, created a dangerous chemical reaction. An experiment also showed that a similar reaction can occur when nitric acid is poured into a graduated cylinder containing only remnants of ethanol or methanol.

Sulfuric acid (H_2SO_4) is a colorless, oily liquid that is highly corrosive, a strong oxidizing agent, and dangerously reactive. It reacts violently with bases and is corrosive to most metals forming flammable/explosive hydrogen gas. It reacts violently with water and organic materials with evolution of heat. Upon heating, toxic sulfur oxides are formed. slowly to water with constant stirring. If added without stirring, it will produce a pocket of steam in the bottom of the vessel, throwing the contents out of the vessel. Concentrated sulfuric acid can cause severe, deep burns on contact with the skin, and permanent vision loss on contact with the eyes. Tissue is destroyed by the acid's dehydrating action. Lungs may be affected by long-term or chronic exposure to aerosol. Skin lesions, tooth erosion, and conjunctivitis are other long-term effects.

Hydrofluoric acid (HF) is a clear, colorless, fuming liquid or gas with a sharp, penetrating odor. It is very dangerous to the eyes, skin, and upper respiratory tract. The substance can be absorbed into the body by inhalation, through the skin and by ingestion. A harmful concentration of the gas in air can be reached quickly, making it very dangerous to handle. Exposure by ingestion, inhalation or contact, can be fatal. Undissociated HF poses a unique threat in that it can destroy soft tissues and result in decalcification of the bone. Moreover, the effects may be delayed. Laboratories where HF is used should stock an antidote kit to be used in case of exposure. Although it is a relatively weak mineral acid, HF will attack glass or silicon compounds, and should be measured, mixed, and stored in polyethylene vessels. HF reacts with many compounds, including metals, and will liberate explosive hydrogen gas.

Orthophosphoric acid (H_3PO_4), a colorless thick liquid or hygroscopic crystal, is a medium strong acid. It is corrosive to the skin, eyes and respiratory tract. Phosphoric acid decomposes on contact with alcohols, aldehydes, cyanides, sulfides, ketones, and can react with halogenated organic compounds forming organophosphorus nerve-gas type compounds that are extremely toxic. It reacts violently with bases, and will generate hydrogen gas when it reacts with metals.

Perchloric acid ($HClO_4$) is a colorless, fuming, hygroscopic liquid. It is extremely unstable in
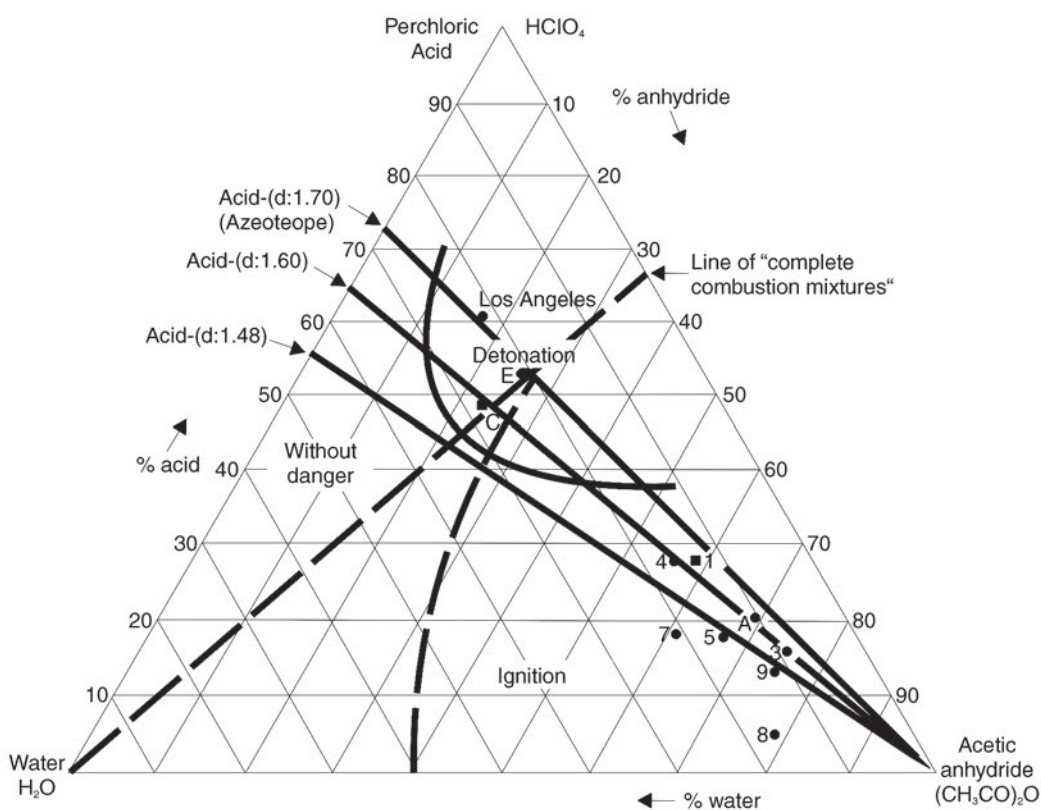
Figure 24.4. Diagram developed by Médard, Jacquet and Sartorius showing safe (A and 1 to 9) nonexplosive electropolishing solutions and explosive (C, E and Los Angeles) compositions of perchloric acid, acetic anhydride and water (brought in by the perchloric acid, but not corrected for the effect of acetic anhydride).

concentrated form and may explode by shock or concussion when dry or drying, so commercially available perchloric acids come in concentrations of 65 to 72%. In this form, contact with perchloric acid will cause irritation and burns, while its fumes are highly irritating to the mucous membranes. Contact with organic, or other easily oxidized material, can form highly unstable perchlorates, which can ignite and cause explosions. Regular use of perchloric acid requires that the ventilation system must be specifically designed and maintained for perchloric acid. Special fume hoods with a waterfall-type fume washer will remove the perchlorate fumes before they can enter the exhaust system.

Perchloric acid is very useful in electropolishing solutions. Never electropolish samples mounted in phenolic (Bakelite) or other plastics in perchloric acid solutions as explosions can result. The mixture of perchloric acid and acetic anhydride, which was developed by Jacquet, is difficult to prepare and highly explosive. Jacquet has reviewed the accidents involving perchloric acid and has described safety procedures [30]. The worst accident occurred

on February 20, 1947, in an electroplating factory in Los Angeles. In this accident 17 people were killed and 150 were injured [29]. Medard, Jacquet, and Sartorius have prepared a ternary diagram showing safe compositions of perchloric acid, acetic anhydride, and water, Figure 24.4. Anderson, however, states that accidents have still occurred with solutions in the "safe" region of this diagram [27]. Electropolishing solutions composed of perchloric acid and acetic anhydride are not recommended. Many companies forbid the use of such mixtures, and some cities have banned their use. Electropolishing solutions of perchloric acid and alcohol, with or without organic additions, and mixtures of perchloric acid and glacial acetic acid are safe to use. Nevertheless, in using these "safe" mixtures, one should follow the formula instructions carefully, mix only small quantities, keep the temperature under control, and avoid evaporation. These solutions should not be stored.

Mixtures of acetic acid and 5-10% perchloric acid have been commonly used to electropolish iron-based alloys and are reasonably safe. Do

not use these solutions to electropolish bismuth, arsenic or tin, as explosions have occurred. Anderson suggests that arsenic, antimony, and tin may also be incompatible with perchloric electrolytes [27]. Do not store these electrolytes . Discard them when they become colored by dissolved metallic ions (from electropolishing). Always keep these solutions cool; increasing the temperature increases the oxidizing power of perchloric acid.

Comas et al. have studied the hazards associated with mixtures consisting of butyl cellosolve and from 10 to 95% of 70% perchloric acid [31]. Mixtures with 60 to 90% acid were explosive at room temperature. Acid concentrations of 30% or less were inflammable but were judged to be safe to use as long as the operating temperature does not exceed 20°C.

Acetic acid (CH_3COOH) is a clear, colorless liquid with a pungent odor. It is a weak acid that reacts with strong oxidizers, bases and metals. It is flammable and is not easily ignited, although when heated, it releases vapors that can be ignited and can travel some distance to an ignition source. Contact with the skin results in serious burns. Inhalation of the fumes irritates the mucous membranes. Anderson states that acetic acid is a good solvent for nitric acid and that a 50% solution can be prepared, but not stored, without danger [27]. Sax, however, states that mixtures of nitric and acetic acids are dangerous [32].

Acetic anhydride (($CH_3CO)_2O$), or acetic oxide, is a colorless liquid with a very strong acetic odor. It can cause irritation and severe burns to the skin and eyes. Acetic anhydride decomposes on heating producing toxic fumes. It reacts violently with boiling water, steam, strong oxidants (specifically sulfuric acid), alcohols, amines, strong bases, and others. It attacks metals, and is very corrosive, especially in presence of water or moisture. It is extremely flammable and should be avoided. The electrolytic polishing mixtures of acetic anhydride and perchloric acid (4-to-1 to 2-to-1 mixtures) developed by Jacquet, as mentioned above, are exceptionally dangerous and should never be used. Dawkins [33] reported an accident involving a mixture of chromium trioxide and acetic anhydride that had been used for electropolishing (Table 24.2).

Citric acid ($C_3H_4(OH)(COOH)_3 \cdot H_2O$) comes as colorless, odorless crystals that are water-soluble. It

is an irritant to the skin, eyes and respiratory tract; no unusual problems are encountered except for occasional allergic reactions.

Lactic acid ($CH_3CHOHCOOH$) is a yellow or colorless thick liquid. It is damaging to the eyes.

Oxalic acid ($COOHCOOH \cdot 2H_2O$) comes as transparent, colorless crystals. It is poisonous if ingested and irritating to the upper respiratory tract and digestive system if inhaled. Skin contact produces caustic action and will discolor and embrittle the fingernails. It is not compatible with nitric acid, as it reacts violently with strong oxidants. It can also form explosive compounds due to reacts with silver.

Picric acid (($NO_2)_3C_6H_2OH$), or 2,4,6-trinitrophenol, comes as yellow crystals that are wet with 10 to 35% water. When picric acid is dry, it is a dangerous explosive. It is toxic and stains the skin and is incompatible with all oxidizable substances. Picrates, which are metal salts of picric acid, are explosive. When picrates are dry, they can detonate readily, possibly spontaneously. Purchase in small quantities, keep it moist, and store it in a safe, cool place. If it starts to dry out, add a small amount of water to keep it moist. The maximum solubilities of picric acid in water and in ethanol are about 1.3 and 8g per 100mℓ, respectively. Picral can be stored safely. During use, the solution should not be allowed to dry out. The etching residue should be discarded at the end of the day to avoid potential explosions.

Bases

Bases, such as ammonium hydroxide (NH_4OH), potassium hydroxide (KOH), and sodium hydroxide (NaOH), are commonly used in metallography, chiefly in etchants.

Ammonium hydroxide is a colorless liquid with a strong, obnoxious odor. Solutions are extremely corrosive and irritating to the skin, eyes, and mucous membranes. It reacts exothermically with sulfuric acid and other strong mineral acids, producing boiling solutions.

Sodium and potassium hydroxides are strong bases, available as white deliquescent pellets that are soluble in water. They can rapidly absorb carbon dioxide and water from the air. Solutions stored in flasks with ground glass stoppers may leak air and freeze the stoppers, making re-opening difficult. Dissolving NaOH or KOH in water will generate

considerable heat. Do not dissolve either in hot water. Never pour water onto these hydroxides; always add the pellets slowly to the water. Alkali metal hydroxides react violently with acid, and are corrosive in moist air to metals like zinc, aluminum, tin and lead forming flammable/explosive hydrogen gas. They are very corrosive to skin, eyes and respiratory tract. Long-term exposure may lead to dermatitis. Potassium hydroxide is somewhat more corrosive than sodium hydroxide.

Other Chemicals

Hydrogen peroxide (H_2O_2) is available as a liquid in concentrations of either 3 or 30%. The 3% solution is reasonably safe to use, while the 30% solution is a very powerful oxidant whose effect on the skin is about as harmful as that produced by contact with sulfuric acid. Hydrogen peroxide by itself is not combustible, but if brought in contact with combustible materials, it can produce violent combustion. Hydrogen peroxide is very damaging to the eyes. Because release of oxygen can cause high pressures to develop within the container, the container caps are vented.

Bromine (Br_2), a fuming reddish brown liquid with a pungent, suffocating odor, is commonly used in deep-etching solutions. It is very corrosive, reacting violently with easily oxidized substances, including some metals. Bromine is a dangerous liquid that should only be handled by well-qualified personnel. Its vapors are extremely irritating to the eyes, skin, and mucous membranes. Skin contact produces deep, penetrating burns that are slow to heal. Contact with organic matter can cause fires.

Chromic acid (H_2CrO_4) is formed when chromium trioxide (CrO_3) is dissolved in water. CrO_3 is used in electropolishing solutions (see previous comment and Table 49 about explosive nature of mixtures with acetic anhydride). Dilute aqueous solutions are widely used for attack polishing. It is a powerful oxidant; always wear gloves when using it for attack polishing or use automatic devices and avoid contact potential. Chronic or long-term inhalation exposure may produce asthma-like reactions.

Potassium permanganate ($KMnO_4$), a black crystalline powder, is a powerful oxidant used in etchants. It is a dangerous fire and explosion hazard, especially when in contact with organic materials. Ingestion produces serious damage. $KMnO_4$ and sulfuric acid should never be mixed together because a violent explosion can result.

Potassium dichromate ($K_2Cr_2O_7$), a bright orange crystalline powder, is another powerful oxidant that is used in etchants. Contact can cause ulceration of the hands, severe damage to nasal tissue, or asthma and allergies with long-term exposure.

Cyanide compounds are occasionally used in metallographic applications. Potassium cyanide (KCN) and sodium cyanide (NaCN) are extremely dangerous and highly toxic. Exposure by eye or skin contact, or by ingestion, is fatal. NaCN and KCN vapors are intensely poisonous. They are particularly hazardous when brought in contact with acids or acid fumes because of liberation of hydrogen cyanide, which is extremely toxic and highly flammable. Potassium ferricyanide ($K_3Fe(CN)_6$), a ruby-red crystalline powder and an ingredient in Murakami-type reagents, is poisonous but stable and reasonably safe to use.

A number of nitrates, such as ferric nitrate [$Fe(NO_3)_3\cdot6H_2O$], lead nitrate [$Pb(NO_3)_6$], and silver nitrate ($AgNO_3$), are employed by metallographers. Since they are powerful oxidizers, they pose a dangerous fire hazard, especially when in contact with organic materials. They may evolve toxic fumes, such as oxides of nitrogen and lead, and are poisonous and corrosive to the eyes, skin and respiratory tract.

Summary

In general, the metallographic laboratory is a reasonably safe environment. However, depending on the materials being prepared, dangerous situations can arise. Some hazards, such as the preparation of radioactive or reactive metals, are quite obvious, while others are not. In the preceding discussion, some of the potential hazards that can be encountered are summarized; others undoubtedly exist that are not covered.

Most accidents can be prevented by simple common sense rules. It is best to assume that all metal dust and all chemicals are hazardous. Inhalation of dust and fumes, ingestion, or bodily contact should be avoided. Personal protective equipment is very useful, but it should not be used as a substitute for good laboratory procedures. The use of such equipment does not guarantee freedom from injury.

LABORATORY SAFETY

The metallographic literature contains many references to the use of dangerous materials, often without any mention of the dangers involved or safe handling procedures. This is unfortunate because the unwary may be injured. Many of us are tempted to experiment when a recommended procedure does not work as claimed. The development of electrolytes, chemical polishing agents, or etchants should be left to those who are fully versed in the potential dangers. Metallographic laboratories should have some of the referenced safety publications (see bibliography) readily available in the laboratory and these safety publications should be consulted when working with new or infrequently used materials.

HELPFUL HINTS FOR LABORATORY SAFETY

Purchase relatively small quantities of chemicals that are used routinely, especially those that have a short shelf life, or are dangerous.

After pouring acids, carefully replace the cap, wash the exterior of the bottle under running water, and dry off the surface with a paper towel before replacing the acid bottle in its fireproof cabinet.

Schedule regular time periods for laboratory clean-ups. These should be weekly for high volume laboratories. A clean lab is an essential ingredient in all laboratory safety programs.

SUMMARY

Preparation of metallographic specimens is based upon scientific principles that are easily understood. Sectioning creates damage that must be removed by the grinding and polishing steps if the true structure is to be examined. Each sectioning process exhibits a certain amount of damage, thermal and/or mechanical. Consequently, choose a procedure that produces the least possible damage and use the correct wheel or blade. Grinding also causes damage, with the depth of damage decreasing as the abrasive size decreases. Materials respond differently to the same size abrasive, so one cannot generalize on removal depths. Removal rates also decrease as the abrasive size decreases. Through an understanding of these factors, good, reproducible preparation procedures, such as presented in this guide, can be established for the materials being prepared. Automation in specimen preparation offers much more than reduced labor. Specimens properly prepared using automated devices consistently exhibit much better flatness, edge retention, relief control and freedom from artifacts such as scratches, pull out, smearing and comet tailing, than specimens prepared manually.

GLOSSARY OF TERMS

Abrasive Cutting – the process of removing a sample from a part or component using an abrasive wheel and cutter, typically performed wet; also referred to as abrasive sectioning

Acrylic – a compression mounting compound that produces a clear mount; a two part castable mounting system with fast cure times

Agglomeration – a mass or cluster of particles, particularly in a powder or suspension; when used in polishing, an agglomerated material will act more aggressive then a deagglomerated material of the same micron size

Analysis – the process of examining, interpreting, documenting and measuring a material for structural or processing details

Anvil – a test fixture that helps ensure accurate results by supporting the test piece during the application of the major load during a hardness test

Artifact – a substance or structure not naturally present

Aspect Ratio – ratio of the width to height in a camera

Automated Imaging – detection of the features of interest based on grey level or color differences or morphological characteristics

Automated Preparation - the use of a grinder-polisher with both a base and a power head to prepare a specimen or group of specimens for analysis

Blade – a metal or resin bonded cutting tool containing abrasive, particularly related to a precision saw or precision sectioning

Bright Field – the surface of the specimen is normal to the optical axis of the microscope and a white light is used; grain boundaries, phase boundaries and other features not perpendicular will appear dark in the image

Calibration – as related to microscopy or imaging, the assignment of a known distance to a pixel count; calibration should be applied to each objective in the x & y direction

Castable Mounting – the process of mounting samples that are sensitive to high pressures and/or temperatures, contain high levels or porosity, or have high levels of corrosion; typically a two part liquid or liquid and powder system

Central Force – an approach for applying downward pressure to an entire specimen holder with the specimens evenly against the preparation surface

Chop Cut – cut style where wheel is pulled downward through the work piece; can be pulsed to assist in revealing fresh abrasive

Clamp Mounting – the clamping of several specimens in sandwich form typically used with thin sheets

Clarification – image enhancement largely achieved through the use of grey scale filters

Cloth – a fabric surface to which a polishing suspension or paste is added when used in materials preparation, typically with a backings of pressure sensitive adhesive, magnetic or high friction materials for easily application

Comet Tail – dull, broad depressed lines emanating from a hard particle or from a hole in a specimen in a pattern that resembles the tail of a comet

Compression Mounting – the process of mounting samples using heat and pressure to encapsulate the specimen

Condenser – an adjustable lens free of spherical aberration and coma placed in front of the light source to focus light at the desired point in the optical path

Deagglomerated – process in which loosely adhered clumps of particles are broken apart, particularly in a powder or suspension containing abrasive; when used in polishing, a deagglomerated material will act less aggressive than an agglomerated material of the same micron size

Dark Field – provides contrast completely reversed from that obtained with bright-field illumination, highlighting angled surfaces allowing more positive identification of details such as pits, cracks or etched grain boundaries

Deformation – microstructure that has been altered due to cutting or grinding-polishing

Depth of Field – the distance along the optical axis over which image details are observed with acceptable clarity

Diallyl Phthalate – a thermoset resin compression mounting compound that provides good edge retention

Differential Interference Contrast (DIC) – crossed polarized light used along with a double quartz prism placed between the objective and the vertical illuminator

Disc – in reference to sample preparation, a grinding surface like silicon carbide paper or diamond grinding discs used on a platen

Dwell Time – the amount of time a load is applied to a work piece during a hardness test

Edge Rounding – when the edge or edges of a specimen are abraded at a faster rate than the interior, leading to a change in flatness at the edge such that the edge cannot be brought into focus along with the interior

Electrolytic Anodizing – the electrolytic process of growing an oxide layer on the surface of the specimen; differs from electrolytic polishing and etching in that the specimen is set as the anode within the electrical circuit

Electrolytic Etching – achieved through the completion of an electrical circuit through an electrolyte; also applies to electrolytic polishing

Embedding – hard abrasive particles that become fixed in the surface of a softer specimen

Epoxy – two part liquid systems that physically adhere to specimens; typically low viscosity, low shrinkage and can

Etching – a process used to reveal particular structural characteristics of metal that are not evident in the as polished condition

Eyepiece – magnifies the primary image produced by the objective, otherwise known as ocular

Filter – used to modify the light for ease of observation, for improved photo-microscopy, or to alter contrast

Grinding – the process of removing deformation induced by sectioning from a specimen; part of the third step in material preparation

Grinding & Polishing – the third primary segment of materials preparation and analysis during which the true microstructure or characteristics of a material are revealed

Grinding Media – the abrasive systems used in grinding; typically these are silicon carbide paper or discs, diamond grinding discs, or other medias like alumina or zirconia bonded to discs, strips, belts or cloths; can also include rigid grinding discs with added abrasives

Hardness testing – the fifth primary segment of materials preparation and analysis used to determine a material's resistance to permanent indentation; an empirical test, not a material property, determined by the particular test method and parameters used

Image Analysis – the fourth primary segment of materials preparation and analysis used to detect the features of interested based on gray level or color differences as well as morphological characteristics such as size and shape

Image Capture – the acquisition of an image by means of a camera and frame grabber or digital camera

Immersion – as it applies to materials preparation and analysis, placing a specimen completely into an etch

Indentation – the deformation mark left by a hardness test

Indenter – the tool that contacts the specimen during a hardness test; indenter sizes and shapes vary depending on the scale and test type used

Individual Force – an approach for applying downward pressure to an individual specimen against the preparation surface; also known as single force

Interactive Measurements – measurements in image analysis that require the operator to select the start and end points for the measurement

Knoop Test – a hardness test that uses a rhombohedral-shaped diamond indenter with the 7.114 ratio of long diagonal to short diagonal, keeping elastic recovery to a minimum; only the long diameter is measured in this test

Lapping – an abrasive action in which the particles roll freely on the grinding or polishing surface; in standard materials preparation, this action exists briefly until the abrasive embeds in the disc

Linear Measurement – simple point to point measuremnts used for making the occasional measurment

MACC – minimal area of contact cutting, a technique that keeps the contact area between an abrasive wheel and a sample constant and low, allowing for lower wheel wear, less induced deformation and the sectioning of larger parts; also reffered to as MAC (minimal area of contact)

GLOSSARY OF TERMS

Macroindentation – a test performed using 1-100kg force

Manual Preparation – in materials preparation the process of preparing a specimen without an automated power head; also referred to as hand polishing or hand preparation

Materials Analysis – the examination, interpreation, documentation and measurment of a specimen to gather detailed information on the material's characteristics such as case depth, coating thickness, grain size, hardness, phase percentage porosity

Materials Preparation – a multi step process including sectioning, mounting, grinding-polishing, image anaysis and hardness testing to reveal the true mictrostructure or characteristics of a material; also referred to as met prep, metallographic preparation or metallography

Metallographer – a person involved in the study of metals or materials

Metallographic Preparation – see materials preparation

Metallography – the study of metals and alloys; can also be referred to as material science which broadens the study to include metals and alloys, ceramics, polymers, composites, biomaterials, electronics, etc.

Method – typically the parameters of grinding-polishing, including grinding surface, abrasive suspensions or pastes, polishing cloths, platen speed, power head direction, force, time, etc

Microindentation – a test performed using 10-1000g force

Microstructure – the structure of a material as viewed in a microscope

Mounting – the second primary segment in materials preparation and analysis during which the sample is encapsulated in a compression or castable mounting compound or system

Napless – a polishing cloth, woven or chemotextile, with no nap

Napped – a polishing cloth, typically flocked, with nap of various length

Nonwoven – a polishing cloth consisting of chemotextile material

Numerical Aperature (NA) – a meure of the light-collecting ability of the lens

Objective – the objective lens forms the primary image of the microstructure

Orbital Cutting – combines the best features of chop, oscillation and traverse-and-increment cutters, while minimizing cutting time

Paste – a type of polishing compound made from finely ground or powdered diamond particles, usually water based

Phenolic – a thermoset resin compression mounting compound that provides good edge retention and moderate shrinkage

Planar Grinding – the intial grinding step that established a common plane for all specimens in the holder; applies primarily to central force holders

Platen – the work surface to which grinding discs and polishing cloths are applied; occassionally referred to as a wheel

Precision Sectioning – the process of removing a sample from a part or component using an blade and saw, typically performed wet; also referred to as precision cutting

Preparation Procedure – the multi step process followed during grinding-polishing

Polarized Light – light obtained by placing a polarizing filter in front of the condenser lens of the microscope and placing an anaylzer before the eyepice; when used with an anisotropic metal with the analyzer set to 90degrees to the polaizer, the grain structure will be visible

Polishing – the process of removing the final layers of deformation induced by grinding from a specimen; part of the third step in material preparation

Polishing Abrasives – the fine abrasives, typically diamond, alumina or colloidal silica, used in suspensions for the polishing portion of materials preparation

Porosity – the ratio of the volume of the pores or voids of a material, to the total volume of the mass

PSA – Pressure Sensitive Adhesive used as a backing on abrasive discs and polishing cloths

Pull Out – removal of second phase particles during preparation; pull out may be enhanced if the interface between the particle and matrix is weak or if the particle is particularly brittle

Pulse Cutting – a chop cut style where wheel is pulsed as it is pulled downward through the work piece assisting in revealing fresh abrasive

Relief – an excessive height difference between second-phase particles and the matrix due to differences in grinding-polishing rates

Resolution – the imaging system's ability to reproduce object detail by resolving closely space features; applies to both the microscope and the pixel array of the camera

Sample – a material or section of a material used for testing; typically referred to as a sample before and during testing, but as a specimen from the mounting step on

Saw Cut – cutting action where the sample is fed into the wheel, the wheel contact arc is precisely controlled via depth increment and cutting takes part along both the forward stroke

Scratches – a linear cut along the surface of a specimen due to contact with a correctly oriented abrasive particle; a groove caused by the abrasion process

Sectioning – the process of removing a sample from a part or component using an abrasive wheel and cutter, typically performed wet; also referred to as abrasive cutting/sectioning or precision sectioning

Single Force – an approach for applying downward pressure to an individual specimen against the preparation surface; also known as individual force

SmartCool – a built in feedback loop in an automatic compression mounting system that intelligently ends the cooling step and completes the mounting process when a safe handling temperature has been reached

SmartCut – a built in feedback loop in an automatic cutters and saws that prevents overheating specimens, equipment damage and improves cut quality by monitoring load at the motor and adjusting feedrate to safe zones

Smear – matrix flow over voids, cracks or second phase particles that makes detection of these features and measurement of their extent difficult or impossible

Specimen – a mounted sample; typically referred to as a specimen from the mounting step on

Specimen Preparation – a multi step process including sectioning, mounting, grinding-polishing, image analysis and hardness testing to reveal the true microstructure or characteristics of a material; also referred to as met prep, metallographic preparation or metallography

Stain – a residue left on the surface of a specimen, that may form around second-phase particles due to interactions between the specimen and the abrasives and/or lubricants,or may form on the matrix due to inadequate cleaning or drying or may from due to interactions between the specimens and solvents after preparation or etching

Swabbing – as it applies to materials preparation and analysis, applying an etch to a specimen using a cotton ball soaked in etch solution

Thresholding – the method for representing ranges of pixel grey or color values with different color bitplane overlays

Traverse and Increment – cutting action where the sample is fed into the wheel, the wheel contact arc is precisely controlled via depth increment and cutting takes part along both the forwards and backwards stroke

Vibratory Polishing – a polishing process in which the polisher generates a high frequency, variable amplitude vibrational motion producing a stress-free polish

Wheel – an abrasive wheel used for cutting; can also be used in reference to a platen

Woven – a fabric formed by interlaced threads or fibers

REFERENCES

1. Metallography and Microstructures, Vol. 9, Metals Handbook, 9th ed., American Society for Metals, Metals Park, OH, 1985.

2. G. F. Vander Voort, Metallography: Principles and Practice, ASM International, Materials Park, OH, 1999.

3. L. E. Samuels, Metallographic Polishing by Mechanical Methods, 3rd Ed., American Society for Metals, Metals Park, OH, 1982.

4. J. A. Nelson, "Heating of Metallographic Specimens Mounted in 'Cold Setting' Resins", Practical Metallography, Vol. 7, 1970, pp. 188-191.

5. G. F. Vander Voort, "Trends in Specimen Preparation," Advanced Materials & Processes, Vol. 157, February 2000, pp. 45-49.

6. G. F. Vander Voort, "Metallography for Edge Retention in Surface Treated Sintered Carbides and Steels," Industrial Heating, Vol. 67, March 2000, pp. 85-90.

7. S. L. Hoyt, Men of Metals, ASM, Metals Park, OH, 1979, pp. 99-100.

8. J. Benedict, R. Anderson and S. J. Klepeis, "Recent Developments in the Use of the Tripod Polisher for TEM Specimen Preparation," Specimen Preparation for Transmission Electron Microscopy of Materials – III, Materials Research Society, Pittsburgh, PA, 1992, Vol. 254, pp. 121-140.

9. G. Petzow and V. Carle, Metallographic Etching, 2nd ed., ASM International, Materials Park, OH, 1999.

10. G. F. Vander Voort, "Grain Size Measurement," Practical Applications of Quantitative Metallography, STP 839, American Society for Testing and Materials, Philadelphia, 1984, pp. 85-131.

11. G. F. Vander Voort, "Wetting Agents in Metallography," Materials Characterization, Vol. 35, No. 2, September 1995, pp. 135-137.

12. A. Skidmore and L. Dillinger, "Etching Techniques for Quantimet Evaluation," Microstructures, Vol. 2, Aug./Sept. 1971, pp. 23-24.

13. G. F. Vander Voort, "Etching Techniques for Image Analysis," Microstructural Science, Vol. 9, Elsevier North-Holland, NY, 1981, pp. 135-154.

14. G. F. Vander Voort, "Phase Identification by Selective Etching," Applied Metallography, Van Nostrand Reinhold Co., NY, 1986, pp. 1-19.

15. E. E. Stansbury, "Potentiostatic Etching," Applied Metallography, Van Nostrand Reinhold Co., NY, 1986, pp. 21-39.

16. E. Beraha and B. Shpigler, Color Metallography, American Society for Metals, Metals Park, OH, 1977.

17. G. F. Vander Voort, "Tint Etching," Metal Progress, Vol. 127, March 1985, pp. 31-33, 36-38, 41.

18. E. Weck and E. Leistner, Metallographic Instructions for Colour Etching by Immersion, Part I: Klemm Colour Etching, Vol. 77, Deutscher Verlag für Schweisstechnik GmbH, Düsseldorf, 1982.

19. E. Weck and E. Leistner, Metallographic Instructions for Colour Etching by Immersion, Part II: Beraha Colour Etchants and Their Different Variants, Vol. 77/II, Deutscher Verlag für Schweisstechnik GmbH, Düsseldorf, 1983.

20. E. Weck and E. Leistner, Metallographic Instructions for Colour Etching by Immersion, Part III: Nonferrous Metals, Cemented Carbides and Ferrous Metals, Nickel Base and Cobalt Base Alloys, Vol. 77/III, Deutscher Verlag für Schweisstechnik GmbH, Düsseldorf, 1986.

21. P. Skocovsky, Colour Contrast in Metallographic Microscopy, Slovmetal, Žilina, 1993.

22. H. E. Bühler and H. P. Hougardy, Atlas of Interference Layer Metallography, Deutsche Gesellschaft für Metallkunde, Oberursel, Germany, 1980.

23. H. E. Bühler and I. Aydin, "Applications of the Interference Layer Method," Applied Metallography, Van Nostrand Reinhold Co., NY, 1986, pp. 41-51.

24. G. F. Vander Voort, "Results of an ASTM E-4 Round-Robin on the Precision and Bias of Measurements of Microindentation Hardness Impressions," Factors that Affect the Precision of Mechanical Tests, ASTM STP 1025, ASTM, Philadelphia, 1989, pp. 3-39.

25. G. F. Vander Voort, "Operator Errors in the Measurement of Microindentation Hardness," Accreditation Practices for Inspections, Tests, and Laboratories, ASTM STP 1057, ASTM, Philadelphia, 1989, pp. 47-77.

26. G. F. Vander Voort and Gabriel M. Lucas, "Microindentation Hardness Testing," Advanced Materials & Processes, Vol. 154, No. 3, September 1998, pp. 21-25.

27. R. L. Anderson, "Safety in Metallographic Laboratory," Westinghouse Research Laboratory Science Paper, No. 65 – 1P30 – METLL – P2, March 29, 1965.

28. G. F. Vander Voort, Metallography: Principles and Practice, ASM International, Materials Park, OH., 1999, pp. 148-159.

29. R. C. Nester and G. F. Vander Voort, "Safety in the Metallographic Laboratory," Standardization News, Vol. 20, May 1992, pp. 34-39.

30. P. A. Jacquet, "The Safe Use of Perchloric-Acetic Electropolishing Baths," Met. Finish, Vol. 47, 1949, pp. 62-69.

31. S. M. Comas, R. Gonzalez Palacin and D. Vassallo, "Hazards Associated with Perchloric Acid-Butylcellosolve Polishing Solutions," Metallography, Vol. 7, 1974, pp. 47-57.

32. N. I. Sax, Dangerous Properties of Industrial Materials, 5th ed., Van Nostrand Reinhold Co., Inc., N.Y., 1979.

33. A.E. Dawkins, "Chromic Acid-Acetic Anhydride 'Explosion,'" J. Iron and Steel Institute, Vol. 182, 1956, p. 388.

34. F. Mohs, "Mohs Hardness" [1822] Grundriß der Mineralogie (two volumes, 1822 and 1824): Dresden, Arnoldschen Buchhandlung (translated to English by Wilhelm Ritter von Haidinger as Treatise on Mineralogy in 1825 and published by Constable & Co. Ltd. of Edinburgh).

35. Wahlberg, A., "Brinells Method of Determining Hardness and Other Properties of Iron and Steel" [1901] J. Iron Steel Inst., Vol 59 No. I pp243-298 and Vol 60 No. II pp234-271.

36. Kehl, G.L. [1949], "The Principles of Metallographic Laboratory Practice", 3rd Ed., McGraw-Hill Book Co., 1949, p 229

37. Grundriß der Mineralogie (two volumes, 1822 and 1824): Dresden, Arnoldschen Buchhandlung (translated to English by Wilhelm Ritter von Haidinger as Treatise on Mineralogy in 1825 and published by Constable & Co. Ltd. of Edinburgh).

38. Wahlberg, A., "Brinells Method of Determining Hardness and Other Properties of Iron and Steel" [1901] J. Iron Steel Inst., Vol 59 No. I pp243-298 and Vol 60 No. II pp234-271

39. Kehl, G.L. [1949], "The Principles of Metallographic Laboratory Practice", 3rd Ed., McGraw-Hill Book Co., 1949, p 229

40. O'Neil, B [2010] "Gage Repeatability and Reproducibility in Rockwell Hardness Testing", Advanced Materials and Processes, Nov-Dec pp33-35

41. International System of Units (Le Système International d'Unités)

42. Vander Voort, G. F. [1999] "Metallography: Principles and Practice", ASM International, Materials Park, OH

43. Vander Voort, G.F. [1989] "Results of an ASTM E-4 Round-Robin on the Precision and Bias of Measurements of Microindentation Hardness Impressions," Factors that Affect the Precision of Mechanical Tests, ASTM STP 1025, ASTM, Philadelphia pp. 3-39

44. Vander Voort, G.F. [1989] "Operator Errors in the Measurement of Microindentation Hardness," Accreditation Practices for Inspections, Tests, and Laboratories, ASTM STP 1057, ASTM, Philadelphia pp. 47-77

45. Samuels, L.E. [1982] "Metallographic Polishing by Mechanical Methods", 3rd Ed., American Society for Metals, Metals Park, OH

46. Vander Voort, G.F. and Lucas, G.M. [1998] "Microindentation Hardness Testing," Advanced Materials & Processes, Vol. 154, No. 3, pp. 21-25

47. R.W. Johnson, Wear, Vol 16, 1970 p.351-358

Selected Bibliography of Books on Laboratory Safety

ASTM E-2014, "Standard Guide on Metallographic Laboratory Safety".

N. Van Houten, Laboratory Safety Standards for Industry, Research, Academe, Sci. Tech. Pubs., Lake Isabella, CA. 1990.

E. Gershey, A. Wilkerson and E. Party, Laboratory Safety in Practice, Van Nostrand Reinhold, N.Y., 1991.

A. A. Fuscaldo et al. (eds), Laboratory Safety: Theory & Practice, Academic Press, San Diego, CA. 1980.

S. B. Pal (ed.), Handbook of Laboratory Health & Safety Measures, Kluwer Academic, Norwell, MA., 1985.

Norman V. Steere (ed.), Handbook of Laboratory Safety, 2nd ed., CRC Press, Boca Raton, FL., 1971.

A. Keith Furr (ed.), Handbook of Laboratory Safety, 3rd ed., CRC Press, Boca Raton, FL., 1989.

L. J. Diberardinis et al., Guidelines for Laboratory Design: Health & Safety Considerations, J. Wiley & Sons, N.Y., 1987.

S. R. Rayburn, The Foundations of Laboratory Safety, Brock-Springer Series in Contemporary Bioscience, Springer-Verlag, N.Y., 1989.

C. A. Kelsey and A. F. Gardner (eds.), Radiation Safety for Laboratory Technicians, Warren H. Green, Inc., St. Louis, MO., 1983.

N. I. Sax, Dangerous Properties of Industrial Materials, 5th ed., Van Nostrand Reinhold Co., N.Y., 1979.

Prudent Practices for Handling Hazardous Chemicals in Laboratories, National Academy Press, Washington, D.C., 1981.

Prudent Practices for Disposal of Chemicals from Laboratories, National Academy Press, Washington, D.C., 1983.

N. Proctor and J. Hughes, Chemical Hazards in the Workplace, J. B. Lippincott Co., Philadelphia, 1978.

F. A. Patty (ed.), Industrial Hygiene and Toxicology, Volume II – Toxicology, 3rd ed., Wiley-Interscience, N.Y., 1980.

L. Bretherick, Handbook of Reactive Chemical Hazards, 2nd ed., Butterworths, London, 1979.

R. J. Lewis, Sr. (ed.), Hawley's Condensed Chemical Dictionary, 12th ed., Van Nostrand Reinhold, New York, 1993.

APPENDIX

Table A1: Abrasive Cutting Troubleshooting Guide

Issue	Possible Cause	Suggested Remedy
Burning (bluish discoloration)	Overheated specimen	Increase coolant flow rate Reduce cutting pressure Select a wheel with softer bonding (faster breakdown)
Rapid wheel wear	Wheel bonding breaks down too rapidly	Select a wheel with harder bonding Reduce cutting pressure
Frequent wheel breakage	Uneven coolant distribution	Adjust coolant flow to be even on both sides of the wheel
	Loose specimen fixturing	Clamp the specimen more securely
	Abrupt contact with specimen	Start cut contact carefully
	Wheel was previously cracked at start up	Handle carefully
Resistance to cutting	Slow wheel bond breakdown	• Select a wheel with softer boding • Use a "pulse" cutting mode • Use cutter with oscillating motion or with Minimal Area of Contact Cutting* (MACC) ability
Stalled wheels	Inadequate cutter capacity	• Use cutter with greater horsepower • Reduce pressure or feed rate • Use with cutter oscillating motion or with MACC ability
	Pinched blade due to movement of specimen	Tighten the vise on one side less than on the other side

Figure A1. Abrasive Cutting Troubleshooting Guide

Pointed: Wheel bond too hard

Chisel: Nonuniform coolant flow

Glazed Surface: Wrong wheel

Normal: For solid materials

Normal: For structural or medium wall tubing

Normal: For light tubing or other thin wall section
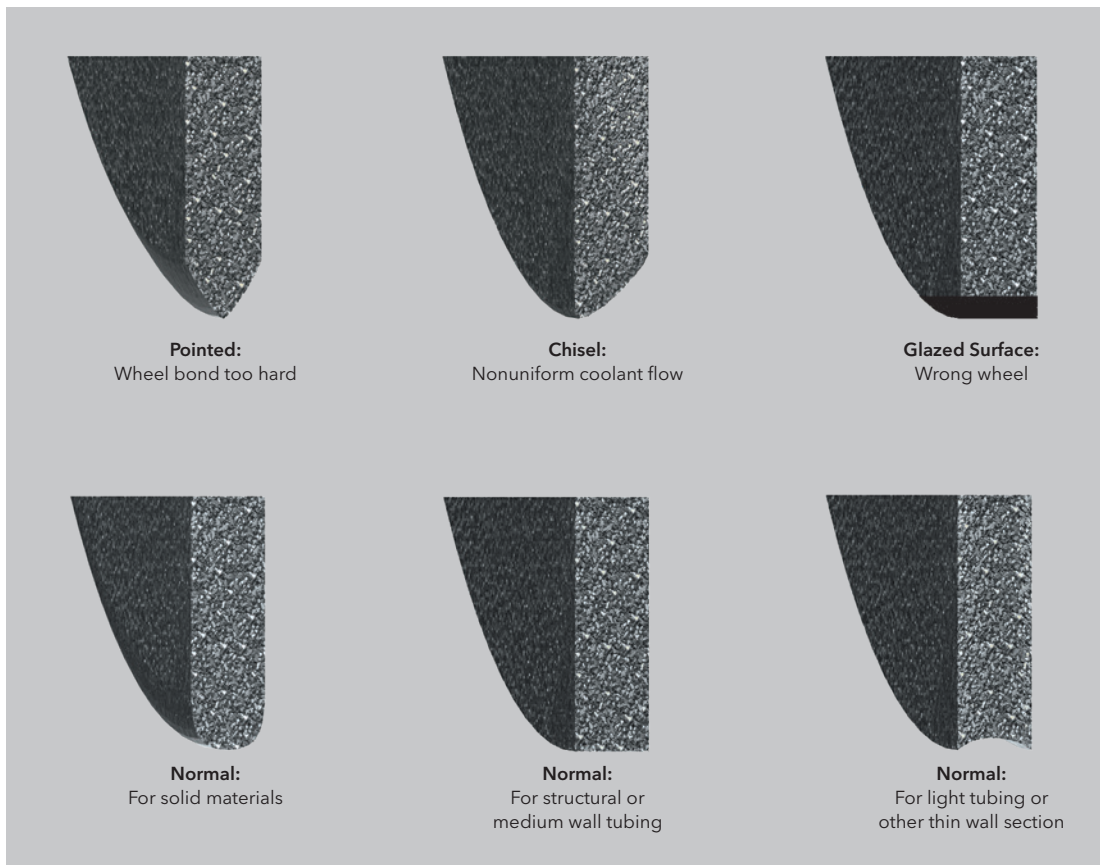
Table A1: Abrasive Cutting Troubleshooting Guide

Thermosetting Resins (Epoxies, Diallyl Phthalates and Phenolics)

	Defect	Probable Cause	Suggested Remedy
	Radial splits or cracks	Specimen cross sectional area too large; specimen with sharp corners	Use a larger mold size; decrease the specimen size; bevel sharp corners, if possible
	Shrinkage gaps	Specimen surfaces dirty; specimen cooled quickly after polymerization; wrong resin used	Clean and dry specimens carefully; after polymerization, cool under pressure to near ambient; use EpoMet G or EpoMet F
	Circumferential cracks	Resin contained moisture	Keeps resins dry during storage; keep containers closed when not using; dry resin by baking at 100-120°F [38-49°C]
	Bulging or soft mount	Inadequate curing (polymerization) time	Increase polymerization time and pressure
	Mount appears grainy and unfused	Time at temperature too short; temperature for polymerization too low; molding pressure too low	Increase polymerization time, temperature and pressure

Thermosetting Resins (Acrylics)

	Defect	Probable Cause	Suggested Remedy
	Cottonball	Incomplete polymerization of resin; not enough time at temperature	Use less resin; use longer heating and cooling periods; use controlled linear cooling
	Crazing	Relief of internal stresses upon ejection of mount	Cool mount to a lower temperature before ejection; use controlled linear cooling

Figure A2. Comparison of Six Edge Retention Compression Mounting Compounds

PhenoCure phenolic resin

EpoMet thermosetting epoxy resin

ProbeMet Cu-filled conductive resin

KonductoMet C-filled conductive resin

EpoxiCure cast epoxy resin with Conductive Filler particles

SamplKwick cast acrylic resin with Conductive Filler particles
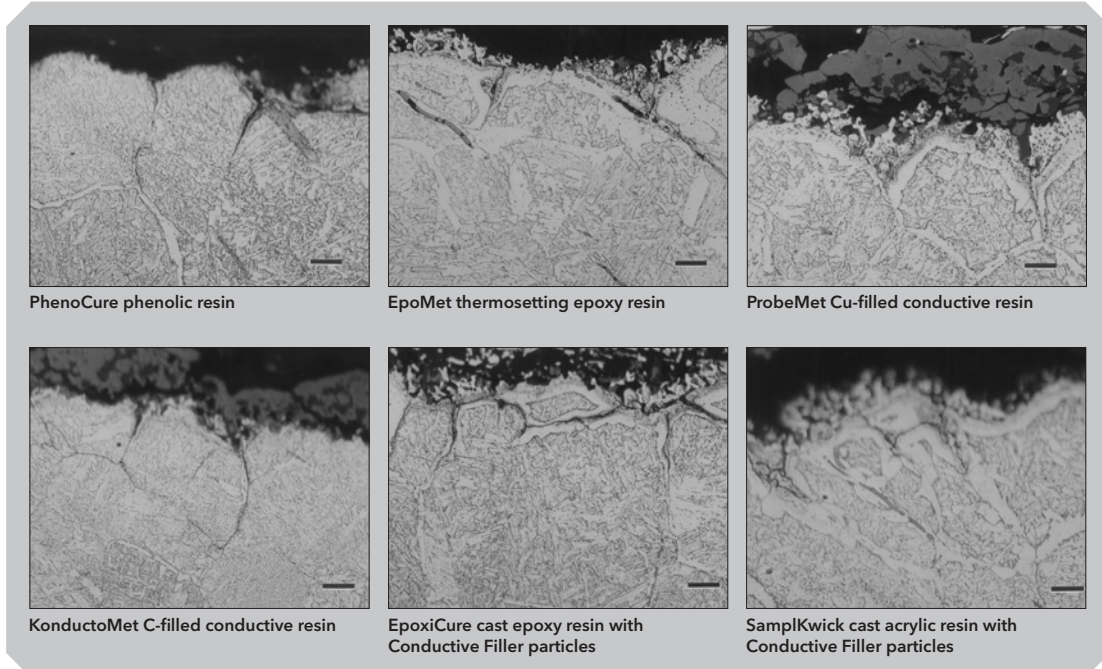
Micrographs showing the as-forged surface of a hardened modified 5130 alloy steel part mounted using a variety of resins showing different degrees of edge retention. The specimens were polished simultaneously in the same holder and were etched with 2% nital. The magnification bars are 20 µm long. Best results were obtained with EpoMet, ProbeMet and EpoxiCure resin with the Conductive Filler particles.

APPENDIX

Table A3: Castable Mounting Selection Guide

Acyrlic	Description
SamplKwick	• Translucent, general purpose acrylic system • 5-8 minute cure time at room temperature • ~179°F [79°C] Peak Temperature • ~85 Shore D Hardness
VariKleer	• Clear, general purpose acrylic system - Requires pressure vessel for clear mounts • 5-15 minute cure time at room temperature • ~212°F [100°C] Peak Temperature • ~84 Shore D Hardness
VariDur 10	• Semi transparent, low odor system • Low shrinkage, high viscosity • 8 minute cure time • ~212°F Peak Temperature • ~80 Shore D Hardness
VariDur 200	• Dark blue, low odor system • Low shrinkage, high viscosity • 8 minute cure time • ~212°F Peak Temperature • ~85 Shore D Hardness
VariDur 3003	• Blue, mineral filled acrylic system • Hardest castable mounting system • 15-30 minute cure time at room temperature • ~252°F [122°C] Peak Temperature • ~90 Shore D Hardness
Epoxy	**Description**
EpoxiCure 2	• Clear, general purpose epoxy system • 6hr cure time at room temperature • <104°F [40°C]Peak Temperature • ~80 Shore D Hardness
EpoHeat CLR	• Transparent clear epoxy system • Long pot-life allows product to be mixed in large batches • 1hr cure time in oven at 149°F [65°C] • <324°F [65°C] Peak Temperature • ~82 Shore D Hardness
EpoThin 2	• Clear, low viscosity epoxy system • 9hr cure time at room temperature • <149°F [65°C] Peak Temperature • ~78 Shore D Hardness
EpoKwick FC	• Clear, fast curing epoxy system with low shrinkage • 2hr cure time at room temperature • <250°F [121°C] Peak Temperature • ~82 Shore D Hardness

Curing time is not a precise quantity as it can be influenced by the nature of the mold (how well, or how poorly, the exothermic heat of polymerization is extracted), by the volume of the cast resin used, by the volume and nature of the specimen being encapsulated, by the temperature of the mix, by room temperature and air circulation and by the age of the resin and hardener (i.e., curing time is longer for products beyond their normal shelf life). The values listed are typical for metallic specimens cast in 1.25-inch (30-mm) diameter Sampl-Kup molds at 21 °C (70 °F) using fresh resin and hardener.

Table A4: Final Polishing Abrasives Selection Guide

Name	Characteristics	Applications
MicroPolish Alumina Powder and Suspensions	1, 0.3 and 0.05µm alumina powder and suspensions	Inexpensive general purpose abrasive for polishing most minerals and metals
MicroPolish II Deagglomerated Alumina Powder and Suspensions	1 and 0.3 deagglomerated alumina powders suspensions	Higher quality alumina suitable for polishing most minerals and metals; produces a better surface finish than alumina abrasives of the same size
MasterPrep Deagglomerated Seeded Gel Alumina Suspension	0.05µm sol-gel alumina suspension	Unique seeded gel alumina produces better finishes than calcined alumina abrasives; excellent with minerals, metals, carbides, PCBs. Good for precious metals
MasterMet Colloidal Silica Suspension	0.06µm amorphous silicia colloidal suspension with a pH of ~10 producing a chemo-mechanical polishing	Chemo-mechanical action produces excellent surfaces for metals, minerals, polymers, PCBs, electronic devices, etc., especially soft metals but not for precious metals
MasterMet 2 Non-Crystallizing Colloidal Silica Suspension	0.02µm non-crystallizing amorphous colloidal silica suspension with a pH of ~10.5 producing chemo-mechanical polishing action	Non-crystallizing version of colloidal silica yields similar results as MasterMet but without the danger of crystallized silica particle contamination
MasterPolish Blended Alumina and Colloidal Silica Suspension	Proprietary viscous blend of high purity 0.05µm alumina and colloidal silica with a slightly basic pH (can be diluted with water)	Proprietary blend has little water and can be used with minerals that are sensitive to warer, such as Mg and most of its alloys; good for most Fe, Ni, or Co based alloys and Metal Matrix Composites (MMC)
MasterPolish 2 Proprietary Chemo-Mechanical Suspension	Proprietary 0.06µm abrasive suspension with a pH of ~10 for chemo-mechanical polishing action	Proprietary blend designed for final polishing of ceramic materials, nitrides, carbides and composites

Table A5: Electrolytic Polishing & Etching Troubleshooting Chart

Problem	Probable Cause	Correction
Center of the specimen is deeply etched	No polishing film at the center of specimen	Increase voltage Decrease agitation Use more viscous electrolyte
Pitting or etching at the edges of specimen	Too vicsous or thick film	Increase voltage Decrease agitation Use more viscous electrolyte
Studge on the surface	Insoluble anode product	Try a new electrolyte Increase temperature Increase voltage
Rough or matte surface	Insufficient or no polishing film	Increase voltage Use more viscous electrolyte
Waviness or streaks on the polished surface	Insufficient time Incorrect agitation Inadequate pre-preparation Too much time	Increase or decrease agitation Better pre-preparation Increase voltage Decrease time
Stains on the polished surface	Attach after polising Current is off	Try less corrosive electrolyte
Unpolished spots (Bulls-eyes)	Gas bubbles	Increase agitation Decrease voltage Tilt the specimen
Phases in the relief	Insufficient polishing time	Increase voltage Better pre-preparation Decrease time
Pitting	Too long polishing Too high voltage	Better pre-preparation Decrease voltage Decrease time Try a different electrolyte

Figure A3. A Guide to Equivalent Loads for Different Mount Sizes
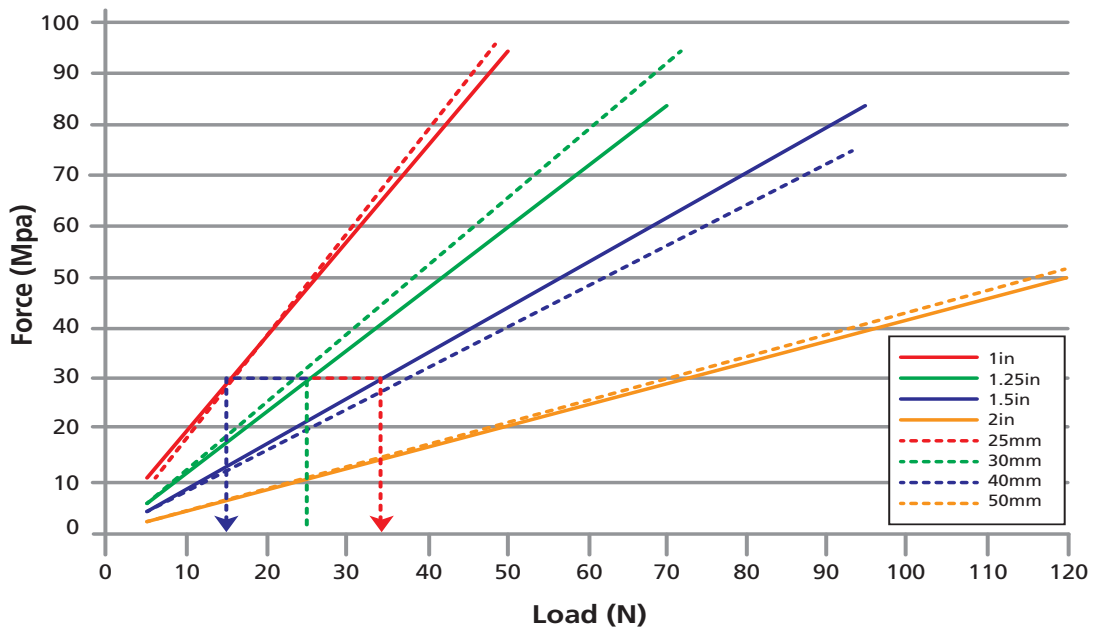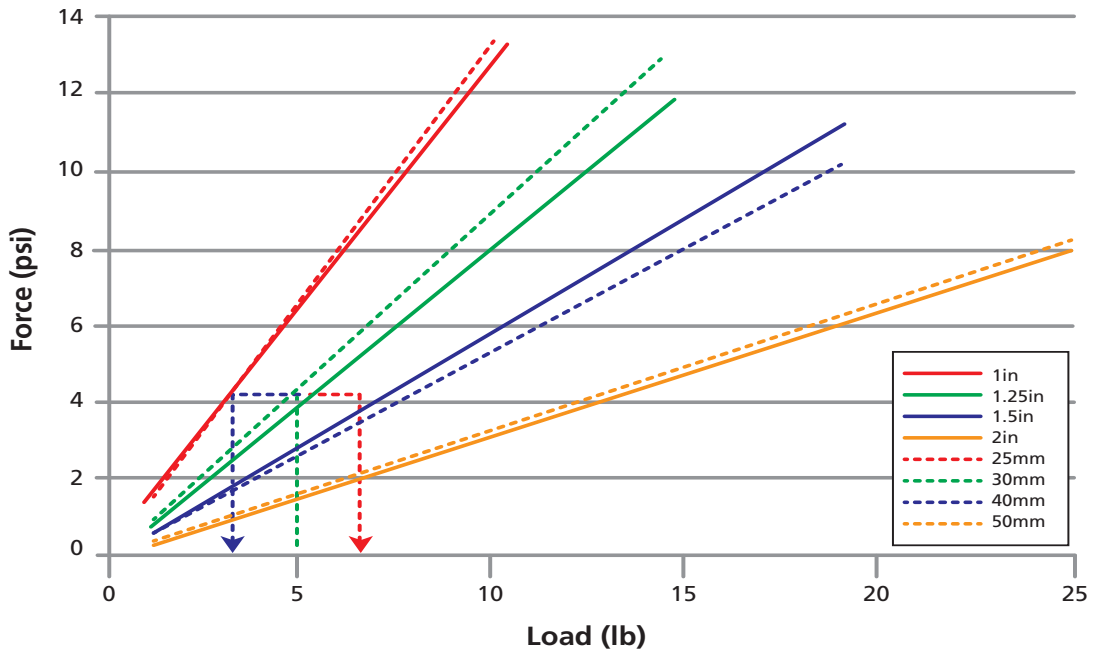
The two graphs plot the load applied per specimen, and the resulting pressure, as a function of mold diameter for both Imperial and metric units. An example of the use of the graphs is shown. Assume that we have a practice developed using 1.25in diameter mounts that calls for 5 lbs. load per specimen. We want to use this method with mounts that are 1in in diameter. How much load should we use? Draw a line vertically at 5 lbs. (shown in green) until it intersects the line for a 1.25in diameter mount. Then draw a horizontal line (shown in blue) from the intersection point to the line for 1in diameter mounts. Then, draw a vertical line (shown in blue) down to the load axis. This point is about 3 lbs. So, we would use 3 lbs per specimen for 1in mounts. In similar manner, if we want to use 1.5in diameter specimens, the graph would suggest that we use about 7 lbs. per specimen (red lines).

Table A5: Preparation Troubleshooting Guide

Defect	Probable Cause	Suggested Remedy
Comet Tails	Poorly bonded very hard phase in softer matrix, or pores in matrix results in unidirectional grooves emanating from particles of hole.	Use hard, napless cloths; reduce applied pressure; reduce step times; impregnate pores with epoxy or wax; use complementary rotation. In manual preparation, avoid unidirectional grinding.
Edge Rounding	Shrinkage gaps between specimen and mount are main problem and, when present, avoid napped cloths and long polishing times.	Avoid gaps by cooling thermosetting mount under pressure after polymerization. Use EpoMet resin (least prone to shrinkage gaps); of castable resins, epoxy is best (adding Flat Edge Filler particles to epoxy improves edge retention); use central force rather than individual force; use Apex Hercules rigid grinding discs; use napless cloths.
Embedding	Hard abrasive tends to embed in soft metals; finer particles are more likely to embed then larger particles.	Coat SiC abrasive paper with parafin wax (candlewax is best, soaps are also effective); beeswax is less effective; reduce applied pressure rpm and times; avoid finer SiC abrasive papers; avoid using diamond slurries with fine diamond sizes (for ≤3µm diamond, paste embeds less than slurries). SiC embedded in soft metals (e.g. Pb) can be removed by polishing with alumina slurries
Pull Outs	Excessive grinding time on worn SiC paper; excessive polishing time on napped cloths; excessive pressure; and inadequate lubrication lead to pull out of second-phase particles.	Grind no more than 60 seconds on a sheet of SiC paper; use napless cloths; reduce applied pressure; use proper degree of lubrication.
Relief	Use of napped cloths, long polishing times and low pressure create height differences between matrix and second-phase particles.	Use napless cloths, higher pressures and shorter times. If relief is observed using contra rotation in last step, repeat the las step using complementary rotation.
Scratches	Contamination of grinding or polishing surfaces by coarser abrasives; pull out of hard particles from matrix or broken pieces of brittle materials; or inadequate grinding or polishing times.	Maintain clean operating conditions, washing hands and equipment between steps; for cracked or porous specimens, use ultrasonic cleaning after each step (especially polishing); execute each step thoroughly (avoid short cutting methods); when using the Apex magnetic disc system, store the discs in a clean drawer or cabinet and scrub surfaces if contamination is believed to have occurred; and high pressures can leave a heavy scratch pattern.
Smear	Flow of softer metals may be caused by inadequate lubrication, excessive pressures or rpms.	Use proper degree of lubrication, lower pressures and rpms; use a medium napped cloth on final step; lightly etch the specimen after final polishing and repeat the last step; use the vibratory polisher to remove smeared surface metal.
Stain	Stains around second-phase particles can be induced by interactions between abrasive and particle, perhaps affected by water quality; or they can occur due to inadequate cleaning or improper solvents used after the abrasive step.	Some staining issues are unique to the specimen but most result from using impure tap water (switch to distilled water; sometimes de-ionized water is needed) or inadequate drying. When using colloidal silica, which can be hard to remove, direct the water jet onto the cloth and wash the specimens (and cloth) for the last 6-10 seconds of the polishing cycle. This makes subsequent cleaning easy. Otherwise, scrub the surface with water containing a detergent solution using cotton. Then, rinse with alcohol and blow dry with hot air. Compressed air systems can contain impurities, such as oils, that can stain the surface.

ASTM METALLOGRAPHY STANDARDS

ASTM Metallography Standards

A247: Visual Classification of Graphite in the Microstructure of Cast Iron

A892: Defining and Rating the Microstructure of High Carbon Bearing Steels

B390: Evaluating Apparent Grain Size and Distribution of Cemented Tungsten Carbides

B588: Measurement of the Thickness of Transparent or Opaque Coatings by Double-Beam Interference Microscope Technique

B657: Metallographic Determination of Microstructure in Cemented Tungsten Carbide

B681: Measurement of Thickness of Anodic Coatings on Aluminum and of Other Transparent Coatings on Opaque Surfaces Using the Light-Section Microscope

B748: Measurement of the Thickness of Metallic Coatings by Measurement of Cross Section with a Scanning Electron Microscope

B795: Determining the Percentage of Alloyed or Unalloyed Iron Contamination Present in Powder Forged Steel Parts

B796: Nonmetallic Inclusion Level of Powder Forged Steel Parts

B847: Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross section

C664: Thickness of Diffusion Coating

E3: Preparation of Metallographic Specimens

E7: Standard Terminology Relating to Metallography

E45 Determining the Inclusion Content of Steel

E82: Determining the Orientation of a Metal Crystal

E112: Determining Average Grain Size

E340: Macroetching Metals and Alloys

E381: Macroetch Testing Steel Bars, Billets, Blooms, and Forgings

E384: Microindentation Hardness of Materials

E407: Microetching Metals and Alloys

E562: Determining Volume Fraction by Systematic Manual Point Count

E766: Calibrating the Magnification of a Scanning Electron Microscope

E768: Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel

E807: Metallographic Laboratory Evaluation

E883: Reflected-Light Photomicrography

E930: Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size)

E975: X-Ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation

E986: Scanning Electron Microscope Beam Size Characterization

E1077: Estimating the Depth of Decarburization of Steel Specimens

E1122: Obtaining JK Inclusion Ratings Using Automatic Image Analysis

E1180: Preparing Sulfur Prints for Macrostructural Examination

E1181: Characterizing Duplex Grain Sizes

E1245: Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis

E1268: Assessing the Degree of Banding or Orientation of Microstructures

E1351: Production and Evaluation of Field Metallographic Replicas

E1382: Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis

E1508: Quantitative Analysis by Energy-Dispersive Spectroscopy

E1558: Electrolytic Polishing of Metallographic Specimens

E1920: Metallographic Preparation of Thermal Spray Coatings

E1951: Calibrating Reticles and Light Microscope Magnifications

E2014: Metallographic Laboratory Safety

E2015: Preparation of Plastics and Polymeric Specimens for Microstructural Examination

F1854: Stereological Evaluation of Porous Coatings on Medical Implants

ASTM/ISO STANDARDS

ASTM Metallography Standards

B578: Microhardness of Electroplated Coatings

B721: Microhardness and Case Depth of Powder Metallurgy Parts

C730: Knoop Indentation Hardness of Glass

C849: Knoop Indentation Hardness of Ceramic Whitewares

C1326: Knoop Indentation Hardness of Advanced Ceramics

C1327: Vickers Indentation Hardness of Advanced Ceramics

E10: Brinell Hardness of Metallic Materials

E18: Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials

E92: Vickers Hardness of Metallic Materials

E140: Standard Hardness Conversion Tables for Metals

E448: Scleroscope Hardness Testing of Metallic Materials

E1842: Macro-Rockwell Hardness Testing of Metallic Materials

ISO Metallography Standards

ISO643: Steels – Micrographic Determination of the Ferritic or Austenitic Grain Size

ISO945: Cast Iron: Designation of Microstructure of Graphite

ISO1083: Spheroidal Graphite Cast Iron – Classification

ISO1463: Metallic and Oxide Coatings – Measurement of Coating Thickness – Microscopical Method

ISO2624: Copper and Copper Alloys – Estimation of Average Grain Size

ISO2639: Steel – Determination and Verification of the Effective Depth of Carburized and Hardened Cases

ISO3754: Steel – Determination of Effective Depth of Hardening After Flame or Induction Hardening

ISO3763: Wrought Steels – Macroscopic Methods for Assessing the Content of Non-Metallic Inclusions

ISO3887: Steel, Non-Alloy and Low Alloy – Determination of Depth of Decarburization

ISO4499: Hardmetals: Metallographic Determination of Microstructure

ISO4524-1: Metallic Coatings – Test Methods for Electrodeposited Gold and Gold Alloy Coatings – Part 1: Determination of Coating Thickness

ISO4964: Steel – Hardness Conversions

ISO4967: Steel – Determination of Content of Non-Metallic Inclusions – Micrographic Method Using Standard Diagrams

ISO4968: Steel – Macrographic Examination by Sulphur Print (Baumann Method)

ISO4970: Steel – Determination of Total or Effective Thickness of Thin Surface-Hardened Layers

ISO5949: Tool Steels and Bearing Steels – Micrographic Method for Assessing the Distribution of Carbides Using Reference Photomicrographs

ISO9042: Steels – Manual Point Counting Method for Statistically Estimating the Volume Fraction of a Constituent with a Point Grid

ISO9220: Metallic Coatings – Measurement of Coating Thickness – Scanning Electron Microscope Method

ISO14250: Steel – Metallographic Characterization of Duplex Grain Size and Distribution

ISO/OTHER NATIONAL STANDARDS

ISO Hardness Standards

ISO1355: Metallic Materials – Hardness Test – Calibration of Standardized Blocks to be Used for Rockwell Superficial Hardness Testing Machines (Scales 15N, 30N, 45N, 15T, 30T and 45T)

ISO4516: Metallic and Related Coatings – Vickers and Knoop Microhardness Tests

ISO4545: Metallic Materials – Hardness Test – Knoop Test

ISO4546: Metallic Materials – Hardness Test – Verification of Knoop Hardness Testing Machines

ISO4547: Metallic Materials – Hardness Test – Calibration of Standardized Blocks to be Used for Knoop Hardness Testing Machines

ISO6441-1: Paints and Varnishes – Determination of Microindentation Harness – Part 1: Knoop Hardness by Measurement of Indentation Length

ISO6441-1: Paints and Varnishes – Determination of Microindentation Harness – Part 2: Knoop Hardness by Measurement of Indentation Depth Under Load

ISO6507-1: Metallic Materials – Hardness Test – Vickers Test – Part I: HV 5 to HV 100

ISO6507-2: Metallic Materials – Hardness Test – Vickers Test – Part 2: HV 0, 2 to Less Than HV 5

ISO6508: Metallic Materials – Hardness Test – Rockwell Test (Scales A, B, C, D, E, F, G, H, K)

ISO9385: Glass and Glass-Ceramics – Knoop Hardness Test

ISO10250: Metallic Materials – Hardness Testing – Tables of Knoop Hardness Values for Use in Tests Made on Flat Surfaces

ISO14271: Vickers Hardness Testing of Resistance Spot, Projection and Seam Welds (low load and microhardness)

Other National Standards

France

NFA04-10: Determination of the Ferritic and Austenitic Grain Size of Steels

Germany

DIN50150: Testing of Steel and Cast Steel; Conversion Table for Vickers Hardness, Brinell Hardness, Rockwell Hardness and Tensile Strength

DIN50192: Determination of the Depth of Decarburization

DIN50600: Testing of Metallic Materials; Metallographic Micrographs; Picture Scales and Formats

DIN50601: Metallographic Examination; Determination of the Ferritic or Austenitic Grain Size

DIN50602: Metallographic Examination; Microscopic Examination of Special Steels Using Standard Diagrams to Assess the Content of Non-Metallic Inclusions

SEP1510: Microscopic Test of Steels for Grain Size by Comparison with Standard Charts

SEP1570: Microscopical Examination of Special Steels for Non-Metallic Inclusions Using Standard Micrograph Charts

SEP1572: Microscopic Testing of Free-Cutting Steels for Non-Metallic Sulphide Inclusions by Means of a Series of Pictures

Italy

UNI3137: Extraction and Preparation of Samples

UNI3138: Macrographic Analysis

UNI3245: Microscopic Examination of Ferrous Materials - Determination of Austenitic or Ferritic Grain Size of Plain Carbon and Low-Alloy Steels

UNI4227: Determination of Metallographic Structures

UNI4389: Nonferrous Metals and Their Alloys: Determination of the Dimension of Their Crystal Grains

OTHER NATIONAL STANDARDS

Japan

JISB7724: Brinell Hardness – Verification of Testing Machine

JISB7725: Vickers Hardness – Verification of Testing Machines

JISB7730: Rockwell Hardness Test – Calibration of Standardized Blocks

JISB7734: Knoop Hardness Test – Verification of Testing Machines

JISB7735: Vickers Hardness Test – Calibration of the Reference Blocks

JISB7736: Brinell Hardness Test – Calibration of Standardized Blocks

JISG0551: Methods of Austenite Grain Size Test for Steel

JISG0552: Method of Ferrite Grain Size Test for Steel

JISG0553: Macrostructure Detecting Method for Steel, Edition 1

JISH0501: Methods for Estimating Average Grain Size of Wrought Copper and Copper Alloys

JISR1610: Testing Method for Vickers Hardness of High Performance Ceramics

JISR1623: Test Method for Vickers Hardness of Fine Ceramics at Elevated Temperatures

JISZ2243: Brinell Hardness Test – Test Method

JISZ2244: Vickers Hardness Test – Test Method

JISZ2245: Rockwell Hardness Test – Test Method

JISZ2251: Knoop Hardness Test – Test Method

JISZ2252: Test Methods for Vickers Hardness at Elevated Temperatures

Poland

PN-57/H-04501: Macroscopic Examination of Steel. The Deep Etching Test.

PN-61/H-04502: Reagents for Macrostructure Tests of Iron Alloys

PN-61/H-04503: Reagents for Microstructure Tests of Iron Alloys

PN-63/H-04504: Microstructure Testing of Steel Products. Iron Carbide. Ghost Structure. Widmanstätten's Structure.

PN-66/H-04505: Microstructure of Steel Products. Templates and Evaluation.

PN-75/H-04512: Nonferrous Metals. Reagents for Revealing Microstructure.

PN-75/H-04661: Gray, Spheroidal Graphite and Malleable Cast Iron. Metallographic Examination. Evaluation of Microstructure

PN-76/H-04660: Cast Steel and Iron. Microscopic Examination. Sampling and Preparation of Test Pieces.

PN-84/H-04507/01: Metals. Metallographic Testing of Grain Size. Microscopic Methods for Determination of Grain Size.

PN-84/H-04507/02: Metals. Metallographic Testing of Grain Size. Methods of Revealing the Prior-Austenitic Grains in Non-Austenitic Steels.

PN-84/H-04507/03: Metals. Metallographic Testing of Grain Size. Macroscopic Method of Revealing the Prior-Austenitic Grain Size by the Fracture Method.

PN-84/H-04507/04: Metals. Metallographic Testing of Grain Size. A Method of Testing for Overheating of Steel.

PN-87/H-04514: Steel, Cast Steel, Cast Iron. Macrostructure Examination. Baumann's Test.

Russia

GOST801: Standard for Ball and Roller Bearing Steel

GOST1778: Metallographic Methods of Determination of Nonmetallic Inclusions

GOST5639: Grain Size Determination

Sweden

SIS111101: Estimating the Average Grain Size of Metals

SIS111102: Estimating the Austenitic Grain Size of Ferritic and Martensitic Steels

SIS111111: Methods for Assessing the Slag Inclusion Content in Steel: Microscopic Methods

SIS111114: Determination of Slag Inclusions – Microscopic Methods – Manual Representation

SIS111116: Steel – Method for Estimation of the Content of Non-Metallic Inclusions – Microscopic Methods – Jernkontoret's Inclusion Chart II for the Assessment of Non-Metallic Inclusions

Sweden (continued)

SIS110340: Hardness Test – Vickers Test HV 0, 2 to HV 100 – Direct Verification of Testing Machines

SIS1103 41: Hardness Test – Vickers Test HV 0,2 to HV 100 – Indirect Verification of Testing Machines Using Standardized Blocks

SIS1103 42: Hardness Test – Vickers Test HV 0,2 to HV 100 – Direct Verification of Standardizing Machine for Calibration of Standardized Blocks

SIS110343: Hardness Test – Vickers Test HV 0,2 to HV 100 – Calibration of Standardized Blocks

SIS112516: Metallic Materials – Hardness Test – Vickers Test HV 5 to HV 100

SIS112517: Metallic Materials – Hardness Test – Vickers Test HV 0,2 to Less Than HV 5

SIS117020: Determination of the Depth of Decarburization in Steel

United Kingdom

BS860: Tables for Comparison of Hardness Scales.

BS4490: Methods for Micrographic Determination of the Grain Size of Steel

BS5710: Macroscopic Assessment of the Non-Metallic Inclusion Content of Wrought Steels

BS6285: Macroscopic Assessment of Steel by Sulphur Print

BS6286: Measurement of Total or Effective Thickness of Thin Surface-Hardened Layers in Steel

BS6479: Determination and Verification of Effective Depth of Carburized and Hardened Cases in Steel

BS6481: Determination of Effective Depth of Hardening of Steel after Flame or Induction Hardening

BS6533: Macroscopic Examination of Steel by Etching with Strong Mineral Acids

BS6617: Determination of Decarburisation in Steel

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EDUCATIONAL COURSES

More thorough testing of increasingly complex materials, to include microstructural analysis, is being driven by industry and consumer requirements for improved product quality and reliability. Existing methods for specimen preparation can be insufficient to produce excellent results in higher through-put environments. The desire for speed, consistency and quality have created demand for improved preparation and analysis techniques. Besides offering solutions for this purpose, the training courses offered by Buehler enable individuals to obtain or improve the necessary preparation skills.

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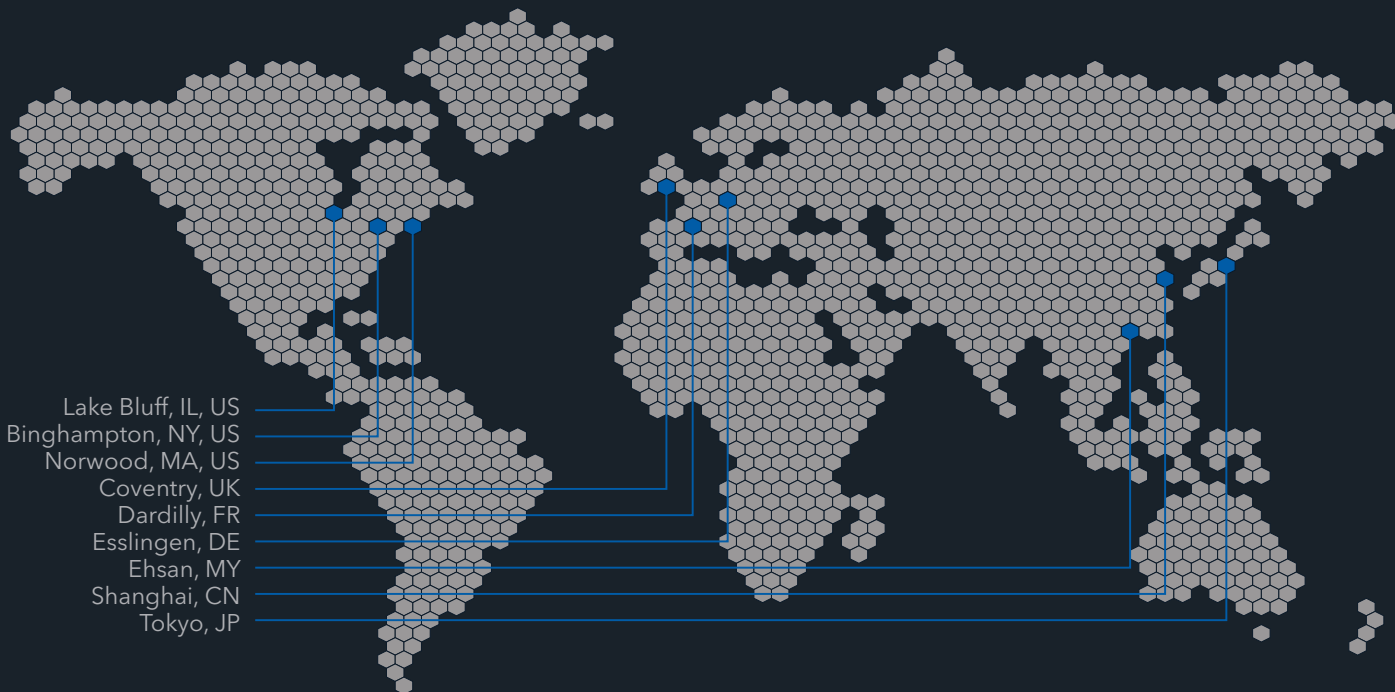
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