BUEHLER® *SUM-MET*™

The Science Behind Materials Preparation

A Guide to Materials Preparation and Analysis

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Warning and Disclaimer

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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 is abrasive material removal 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 5 µm, with SiC paper used exclusively until recently.

Historically, 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 as far as they go; 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 as a paste, slurry or spray. 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 1-µm, or even a 3-µm diamond finish may yield a surface suitable for routine examination, although finer abrasives are usually needed for more critical work, such as failure analysis.

Historically, polishing was 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 9-µm, for example) and the abrasive is added as a slurry or spray. 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 disk, 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.

SAMPLING

The specimens 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. It is rare to encounter excessive sampling as testing costs are usually closely controlled. Inadequate sampling is more likely to occur.

Normally, a specimen 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 cut-off saws, 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 specimen and sectioning is nearly always required to extract the specimens. A classic example of multiple specimen selection is the evaluation of the inclusion content of steels. One specimen is not representative of the whole lot of steel, so sampling becomes 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.

GOALS OF SPECIMEN PREPARATION

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.

- Coarse grinding scratches 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 (optional), (c) grinding, (d) polishing and (e) etching (optional).

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 8-inch (200 mm) diameter platen system with six 1.25-inch (30 mm) 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 the same degree 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.

* Not all of these apply to every specimen.

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-wheel 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-Wheel Cutting

The most commonly used sectioning device in the metallographic laboratory is the abrasive cut-off machine, Figure 1. All abrasive-wheel 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 smooth surface finish 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 summarizes Buehler's recommendations for our machines. Specimens must be fixtured securely during cutting, and cutting pressure should be applied carefully to prevent wheel breakage. Some materials, such as CP (commercial purity) titanium, Figure 2, are quite prone to sectioning damage.

Figure 1. AbrasiMatic 300 Abrasive Cutter
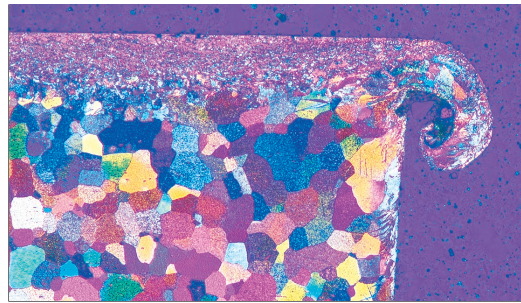
Figure 2. Cutting damage (top) and a "burr" after sectioning of an annealed CP titanium specimen (mod. Weck's reagent, 100X, polarized light plus sensitive tint).

Wheels consist of abrasive particles, chiefly alumina or silicon carbide, and filler in a binder material that 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, abrasives become dull more quickly, and the binder must break-down and release the abrasives when they become dull so that fresh abrasive particles are available to maintain cutting speed and efficiency. Consequently, these wheels are called "consumable" wheels because they wear away with usage. If they do not wear at the proper rate, dull abrasives will rub against the region being cut generating heat and 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 cut-off machine 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

Table 1. Buehler's Abrasive Cutting Recommendations

Recommended Use	Bond	Abrasive	Available Diameters (Inches)	(mm)
General Usage Blades				
Tools Steels 60 HRC andRubber Resin Above Carburized Steels	Al₂O₃	9, 10, 12, 14, 16, 18	230, 250, 300, 350, 400, 455	
Hard Steel 50 HRC	Rubber Resin	Al₂O₃	9, 10, 12, 14, 16, 18	230, 250, 300, 350, 400, 455
Medium Hard Steel 35-50 HRC	Rubber Resin	Al₂O₃	9, 10, 12, 14, 16, 18	230, 250, 300, 350, 400, 455
Soft or Annealed SteelRubber 15-35 HRC 46-90 HRB	Al₂O₃	9, 10, 12, 14, 16, 18	230, 250, 300, 350, 400, 455	
Medium Hard Nonferrous Materials, Uranium, Titanium, Zirconium	Rubber	SiC	9, 10, 12, 14, 16, 18	230, 250, 300, 350, 400, 455
Soft Nonferrous Materials Aluminum, Brass, etc.	Rubber	SiC	9, 10, 12, 14, 16, 18	230, 250, 300, 350, 400, 455
Superalloys	Rubber	Al₂O₃	10, 12, 14, 16, 18	250, 300, 350, 400, 450
Thin Blades to Minimize Kerf Loss and Cutting Deformation				
Tool, Hard Steel, ≤ 45 RC	Rubber	Al₂O₃	5, 9, 10*, 12	130, 230, 250*, 300
Medium Hard, Soft SteelRubber ≥ 45 HRC	Al₂O₃	5, 7, 9, 10, 12, 14	130, 180, 230, 250*, 300, 350	
Hard or Soft Nonferrous Materials	Rubber	SiC	7	180

*Rubber Resin Bond
Refer to Buehler's Consumables Buyers Guide for ordering information, and exact dimensions of arbor sizes, outer diameter, and thickness of Buehler Metabrase, Delta, and Acu-Thin Abrasive Cut-off Wheels

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. Buehler's AcuThin Cut-off Wheels offer the thinnest possible wheels for delicate abrasive sectioning. Buehler also has diamond cut-off blades with resin bonded or Rimlock[1] metal bonded blade forms in sizes from 8-inch (203 mm) to 12-inch (305 mm) 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 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 specimen 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 specimen, and then feeding is halted momentarily, Figure 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.

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

Optimal cutting, with the least damage to the specimen, 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 specimen constant and low. This idea is called the minimum area of contact cutting (MACC) method.

Another option for MACC is the "traverse and increment" cutter design, Figure 3c. 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. The wheel traverse length must be greater than the specimen width. This machine must be programmed for each size part to obtain cutting.

Orbital cutting, Figure 3d, combines the best features of chop, oscillation and traverse-and-increment cutters, while minimizing cutting time. As the wheel is fed into the specimen, 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 specimen. 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

Figure 3. Illustration of cutter types and the wheel path during sectioning

CUTTING STYLE AND WHEEL PATH

Wheel Path

Sample

3a **Chop Cutting**
The traditional form of machine operation. Wheel contact arc is governed by specimen size. Generally a struggle with large/difficult parts.

3b **Chop Cutting with Pulsing**
Wheel contact still governed by specimen size. The pulsing action gives a shock to the wheel causing abrasives to break away. Wheel wear is generally high with this method.

3c **Traverse and Increment**
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. Action is slow.

3d **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.

cutting concept is available in the Delta cutter 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 Saws

Precision saws, Figure 4, are commonly used in metallographic preparation to section materials that are small, delicate, friable, extremely hard, or 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- to 8-inch (76 to 203 mm) in diameter. These blades can be of the non-consumable type, 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 consumable blades 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 2 lists selection criteria for precision saw blades. Buehler's blades for precision saws are available with different abrasive sizes and bonds to provide optimum cutting for a wide variety of applications.

Figure 4. IsoMet 5000 Linear Precision Saw

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 2. Selection Criteria for Precision Saw Blades

BLADE SERIES	3″ x 0.006″ (75mm x 0.2mm)	4″ x 0.012″ (100mm x 0.3mm)	5″ x 0.015″ (125mm x 0.4mm)	6″ x 0.020″ (150mm x 0.5mm)	7″ x 0.025″ (180mm x 0.6mm)	8″ x 0.035″ (200mm x 0.9mm)
Diamond Wafering Blades						
Series 30HC Diamond, for use with plastics, polymers, and rubber			X**		X**	X
Series 20HC Diamond, for aggressive general sectioning of ferrous and nonferrous materials including titanium alloys			X*		X	X
Series 15HC Diamond, for general use with ferrous and nonferrous alloys, copper, aluminum, metal matrix, composites, PC boards, thermal spray coatings, titanium alloys, bone, teeth, and tungsten carbide	X	X	X	X	X	X
Series 20LC Diamond, for use with hard/tough materials structural ceramics, boron carbide, boron nitride, and silicon carbide			X*			X
Series 15LC Diamond, for use with hard/brittle materials structural ceramics, boron carbide, boron nitride, and silicon carbide	X	X	X	X	X	X
Series 10LC Diamond, for use with medium to soft ceramics, electronic packages, unmounted integrated circuits, GaAs, AIN and glass fiber reinforced composites, barium titanate, rare earth oxides, and computer chips	X		X		X*	X
Series 5LC Diamond, for use with soft friable ceramics, electronic packages, unmounted integrated circuits, composites with fine reinforcing media, CaF_2, MgF_2, and carbon composites	X		X			
IsoCut Wafering Blades						
Low Concentration Cubic Boron Nitride (CBN) abrasive blades work well for many tough materials giving significantly shorter cut times	X	X	X	X	X	X
For iron, carbon steels, high alloy steels, cobalt alloys, nickel super-alloys, and lead alloys	X	X	X	X	X	X
High Concentration Cubic Boron Nitride (CBN) for use with Iron and Cobalt Base Alloys, Nickel Base Super Alloys, and Lead Based Alloys	X	X	X	X	X	X
General Usage Abrasive Cut-off Wheels 0.03″ (0.8mm) thick	Bond/Abrasive					
For ferrous materials, stainless steels, cast irons, and thermal spray coatings	R/Al_2O_3				X	
For tough nonferrous metals, aluminum, copper, titanium, uranium, zirconium	R/SiC				X	
AcuThin Abrasive Cut-off Wheels 0.019″ (0.5mm) thick	Bond/Abrasive					
For sectioning small, delicate specimens or where minimal deformation and kerf loss is the primary concern						
Tool, hard steel, ≥ 45 HRC	R/Al_2O_3		X			
Medium hard, soft steel ≤ 45 HRC	R/Al_2O_3		X			

* Alternate blade thickness of 0.020″ (0.5 mm)   ** Alternate blade thickness of 0.030″ (0.8 mm)

For a complete listing of Buehler consumable supplies for use with the Isomet Precision Saws, please refer to Buehler's Consumables Buyers Guide.

MOUNTING OF SPECIMENS

The primary purpose of mounting metallographic specimens is for convenience in handling specimens 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, specimens 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 specimens can be clamped conveniently in sandwich form making this a quick, convenient method for mounting thin sheet specimens. 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 specimen with a thermosetting or thermoplastic mounting material. Common thermosetting resins include phenolic (PhenoCure resin), diallyl phthalate and epoxy (EpoMet resin) while methyl methacrylate (TransOptic resin) is the most commonly used thermoplastic mounting resin. Table 3 lists the characteristics of the hot compression mounting

Table 3. Characteristics of Buehler's Compression (Hot) Mounting Resins

Materials	Least Expensive	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
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	Bronze	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

resins. Both thermosetting and thermoplastic materials require heat and pressure during the molding cycle; but, after curing, mounts made of thermoplastic resins must be cooled under pressure to at least 70 °C while mounts made of thermosetting materials may be ejected from the mold at the maximum molding temperature. However, cooling thermosetting resins under pressure to near ambient temperature before ejection will significantly reduce shrinkage gap formation. Never rapidly cool a thermosetting resin mount with water after hot ejection from the molding temperature. This causes the metal to pull away from the resin producing shrinkage gaps that promote poor edge retention, see Figure 5, because of the different rates of thermal contraction. EpoMet resin, a thermosetting epoxy, provides the best edge retention, Figure 6, of these resins and is virtually unaffected by hot or boiling etchants while phenolic resins are badly damaged.
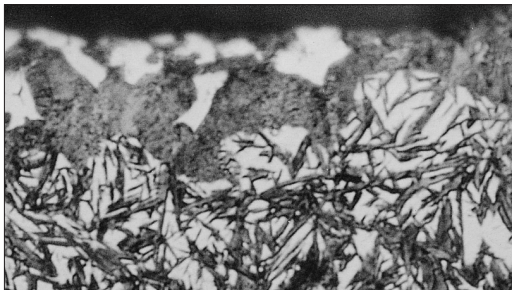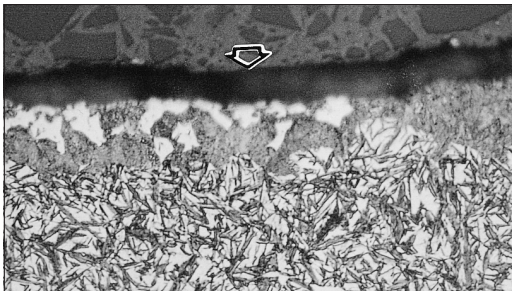

Figure 5. Edge retention of this improperly carbur-ized 8620 alloy steel was degraded by a shrinkage gap between the specimen and the phenolic mount: a) top, 500X; b) bottom, 1000X (2% nital).

Mounting presses vary from simple laboratory jacks with a heater and mold assembly to full automated devices, as shown in Figure 7. 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 "hand" polishing is simplified, as the specimens are easy to hold.
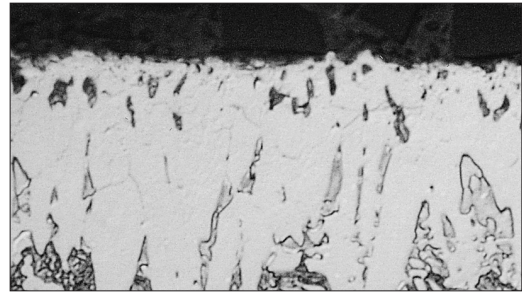

Figure 6. Excellent edge retention of a borided 42CrMo4 alloy steel specimen mounted in EpoMet resin (1000X, 2% nital).

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

Figure 7. SimpliMet 3000 Automatic Mounting Press

Castable Resins for Mounting

Most castable mounting materials require neither pressure nor external heat and are recommended for mounting specimens that are sensitive to heat and/or pressure. Acrylic resins, such as Vari-Dur and SamplKwick resins, are the most widely used castable resins due to their low cost and short curing time. However, shrinkage can be a problem with acrylics. Epoxy resins, 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, (Figure 8), is employed and a low viscosity epoxy, such as EpoThin resin, is used. Epoxies are very suitable for mounting fragile or friable specimens and corrosion or oxidation specimens. Dyes or fluorescent agents may be added to epoxies for the study of porous specimens such as

Figure 8. Vacuum Impregnation Equipment

thermal spray coated specimens. Most epoxies are cured at room temperature, and curing times can vary from 2 to 8 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 4 lists the characteristics of Buehler's castable resins.

Cast epoxy resins provide better edge retention than cast acrylic resins, mainly due to the better adhesion of epoxy to the specimen and their lower shrinkage. Acrylics usually do not bond to the specimen and, because they shrink more during curing, a gap is inevitability formed between specimen and mount. When a shrinkage gap is present, edge retention is usually poor. To improve edge retention with castable mounts, the specimen can be plated, for example, with electroless nickel using the EdgeMet kit, or Flat Edge Filler particles can be added to the resin. To obtain electrical conductivity, Conductive Filler particles can be added to the castable resin.

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

Table 4. Characteristics of Buehler's Castable Resins

Name	Peak Type	Shore D Temperature	Cure Hardness*	Recommended Time	Mold	Comments
EpoThin	Epoxy	80 °F (27 °C)	78 Best Edge Retention	9 hours	Any	Low viscosity, low shrinkage, transparent, best for vacuum impregnation
EpoxiCure	Epoxy	82 °F (28 °C)	82 Best Edge Retention	6 hours	Any	Moderate hardness, transparent, low shrinkage
EpoxiCure NC	Epoxy	82 °F (28 °C)	82 Excellent Edge Retention	6 hours	Any	General purpose epoxy; viscosity 400-600 cps at 77 °F, (25 °C) good adherence to specimen; can be used to vacuum impregnate voids (viscosity can be reduced by warming to 122 ° F (50 °C)); good for heat-sensitive specimens (very low exotherm during polymerization)
EpoKwick	Epoxy	185 °F (85 °C)	82 Good Edge Retention	90 minutes	Sampl-Kup	Faster epoxy, some shrinkage, transparent
EpoColor	Epoxy	175 °F (79 °C)	82 Good Edge Retention	90 minutes	Sampl-Kup	Dye-enhanced epoxy, displays red under darkfield and polarized light
EpoHeat (Transparent)	Epoxy	295 °F (79 °C)	82 Best Edge Retention	90 minutes	Any	Fast, heat cure epoxy system with low shrinkage and low viscosity (32 cps at 176 °F (80 °C)). Excellent for impregnation. Requires oven cure. Golden color.
VariDur 3000	Acrylic	252 °F (122 °C)	90	15-30 minutes	Any	Minimal shrinkage, good edge retention
VariDur	Acrylic	170 °F (77 °C)	85 Fair Edge Retention	10 minutes	Any	Fast cure, harder acrylic opaque, abrasive resistant
SamplKwick	Acrylic	175 °F (79 °C)	80 Poor Edge Retention	5-8 minutes	Any	Very fast cure, translucent, some shrinkage
VariKleer (Transparent)	Acrylic	212 °F (100 °C)	84	10-15 minutes	Sampl-Kup	Designed for use with a pressure vessel, this general use acrylic hardens crystal clear with minimal shrinkage. To achieve crystal clear samples, reusable silicon or EPDM molds should not be used.

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

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" (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 resin using two procedures: a glass mold on a glass plate (insulative) and an aluminum mold on an aluminum plate (conductive). Polymerization produced a maximum exotherm of 132°C using the insulative approach but only 42°C using the conductive approach. Note that 132°C is not much less than the 150°C temperature used in hot compression mounting! Nelson also measured the exotherm produced when an epoxy resin 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 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 resins will exhibit such low exotherms in polymerization. Epoxy resins that cure in short time periods develop much higher exotherms, that can exceed that of acrylic resins. In addition to the speed of curing of the epoxy resin, 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 resin 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 cures fastest in SamplKup plastic molds, slower in phenolic ring forms, and still slower in the reuseable rubber 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 metallographic 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 resin. Plating [2] of a compatible metal on the surface to be protected (electroless nickel, deposited using the EdgeMet Kit 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 9 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 9a, which exhibits poor image contrast between the nickel and the nitrided surface. This is not a problem for the non-plated specimen, Figure 9b.
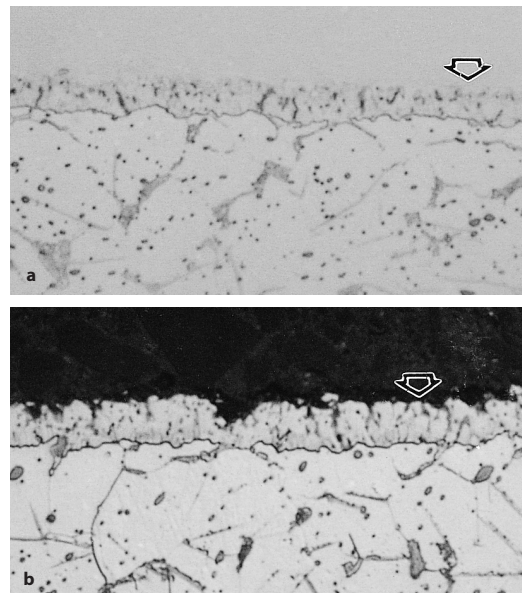

Figure 9. Example of (a, top) poor coating edge visibility due to a lack of contrast between the protective nickel plating and the salt bath nitrided surface (arrow) of 1215 free-machining carbon steel; and, (b, bottom) good contrast and visibility between EpoMet resin and nitrided surface (arrow) plus excellent edge retention (1000X, 2% nital).

Introduction of new technology has greatly reduced edge preservation problems [5,6]. Mounting presses that cool the specimen to near ambient temperature under pressure produce much tighter mounts. Gaps that form between specimen and

resin are a major contributor to edge rounding, as shown in Figure 5. Staining at shrinkage gaps may also be a problem, as demonstrated in Figure 10. Use of semi-automatic and automatic grinding/polishing equipment, rather than manual (hand) 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 8-inch diameter (200 mm) 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 10. 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 materials. Nevertheless, there are still differences among the polymeric materials used for mounting. Thermosetting resins provide better edge retention than thermoplastic resins. Of the thermosetting resins, 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 resin that contains a filler material. For comparison, Figure 11 shows micrographs of a nitrided 1215 specimen mounted in a phenolic resin (Figure 11a), and in methyl methacrylate (Figure 11b), at 1000X. These specimens were prepared in the same specimen holder as those shown in Figure 9, but neither displays acceptable edge retention at 1000X. Figure 12 shows examples of perfect edge retention, as also demonstrated in Figure 9.
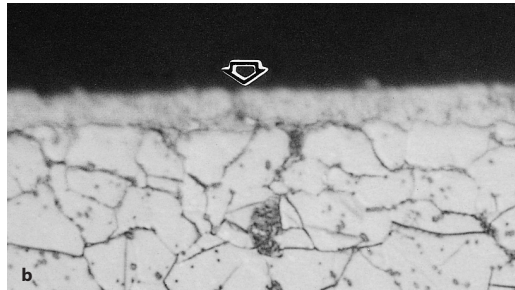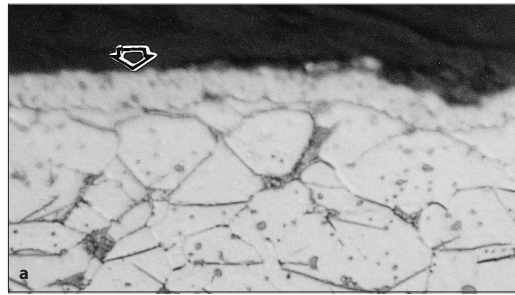

Figure 11. Poor edge retention (arrows) at the salt bath nitrided surface of 1215 free-machining carbon steel mounted in (a, top) phenolic resin and in (b, bottom) methyl methacrylate and polished in the same holder as those shown in Figure 9 (1000X, 2% nital).
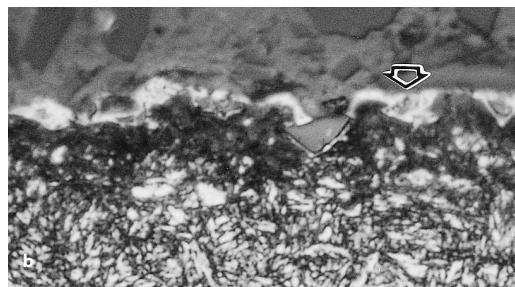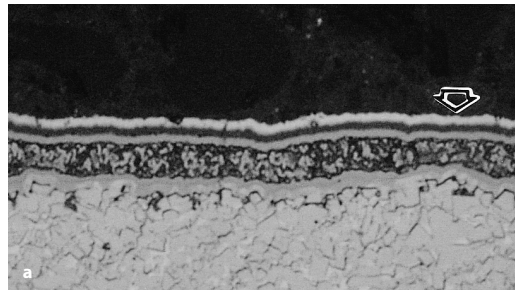
Figure 12. Excellent edge retention for (a, top) complex coated (arrow) sintered carbide insert (1000X, Murakami's reagent) and for (b, bottom) iron nitrided (arrow points to a white iron nitride layer; an embedded shot blasting particle is to the left of the arrow) H13 hot work die steel specimen (1000X, 2% nital). EpoMet was used in both cases.

Very fine aluminum oxide spheres have been added to epoxy mounts 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. Recently, a soft ceramic shot (~775 HV) has been introduced that has grinding/polishing characteristics compatible with metallic specimens placed in the mount. Figure 13 shows an example of edge retention with the Flat Edge Filler soft ceramic shot in an epoxy mount.

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.
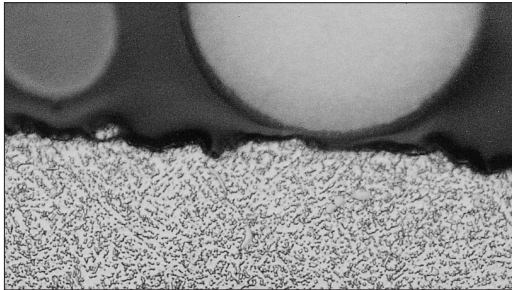
Figure 13. Flat Edge Filler shot was added to Epoxicure resin to improve the edge retention of this annealed H13 hot work die steel specimen (500X, 4% picral).

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

- Electrolytic or electroless Ni plating (e.g., with the EdgeMet Kit) 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 materials are less effective than thermosetting resins. The best thermosetting resin for edge retention is EpoMet G, an epoxy-based resin containing a hard filler material.

- Never hot eject a thermosetting resin 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 "hand" preparation.

- Use the central force mode (defined later in the text) with an automated grinder/polisher as this method provides better flatness than 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 water on the platen and a peripheral hold-down ring, and PSA-backed polishing cloths rather than stretched cloths.

- Apex DGD and UltraPrep metal-bonded or resin-bonded 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, 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 ApexHercules 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 too viscous and boil to easily for vacuum impregnation.

Castable resins are sensitive to shelf life, which can be extended by keeping them in a refrigerator. It is a good practice to date your containers when you get them.

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 to 240 (P180 to P280) is coarse enough to use on specimen surfaces sectioned by an abrasive cut-off wheel. Hack-sawed, bandsawed, or other rough surfaces usually require abrasive grit sizes in the range of 120- to 180-grit. 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 220- or 240-, 320-, 400-, and 600-grit (P240 or P280, P400, P600 and P1200). This sequence is used in the "traditional" approach.

As with abrasive-wheel sectioning, 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 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 grinding stone on a machine such as the PlanarMet (see Figure 17b). 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, damage

depth is high and embedding of alumina abrasive in specimens can be a problem.

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 disks (the traditional type) with grit sizes from about 240- to 9-μm 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 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 6-μm while UltraPrep resin bonded discs are available in three diamond sizes from 30- to 3-μm. Another approach uses a stainless steel woven mesh UltraPlan cloth on a platen charged with coarse diamond, usually in slurry form, for planar grinding. 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 disks. With each of these, an intermediate diamond size, generally 9- to 3-μm, is used.

Grinding Media

The grinding abrasives commonly used in the preparation of metallographic specimens 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 metallography due to its low cutting efficiency. SiC is more readily available as waterproof paper than aluminum oxide. Alumina papers, such as PlanarMet Al 120-grit paper, do have a better cutting rate than SiC for some metals [3]. These abrasives are bonded to paper, polymeric or cloth backing materials of various weights in the form of sheets, 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
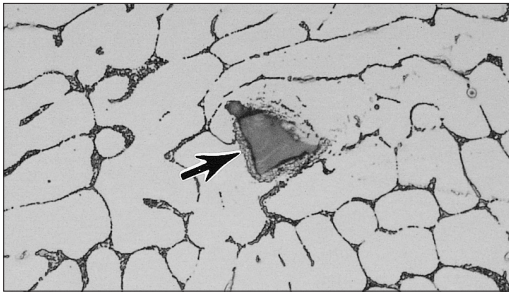
Figure 14. SiC grit particle (arrow) embedded in a 6061-T6 aluminum weldment (500X, aqueous 0.5% HF).

Figure 15. 6μm diamond particles (arrows) embedded in lead (1000X).

readily when grinding soft metals, such as Pb, Sn, Cd and Bi (see Figure 14). 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 15.

CarbiMet 2 SiC paper is made according to the ANSI/CAMI standard (B74. 18-1996) while SiC papers manufactured in Europe are made according to the FEPA standard (43-GB-1984, R 1993). Both
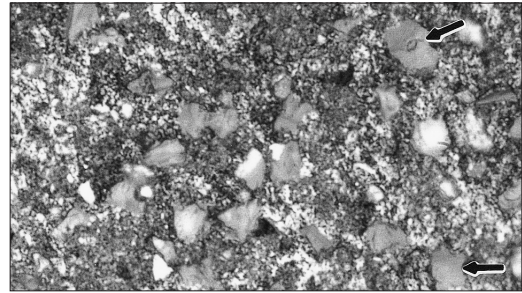
standards use the same methods for sizing the abrasives and the same standards to calibrate these devices (sieving for the coarsest grits, sedimentation grading for intermediate grits (240-600 or P280-P1200), and the electrical resistance method for very fine grit sizes). The grit size numbering systems differ above 180- (P180) grit, but equivalent sizes can be determined using Table 5. As with many standards, they are not mandatory and manufacturers can, and do, make some of their papers to different mean particle

Table 5. European/USA Equivalency Grit Guide

European/USA Equivalency Grit Guide				
FEPA (Europe)		ANSI/CAMI (USA)		
Grit Number	Size(μm)	Grit Number	Size(μm)	Emery Grit
P60	269.0	60	268.0	
P80	201.0	80	188.0	
P100	162.0	100	148.0	
P120	127.0	120	116.0	
P180	78.0	180	78.0	3
P240	58.5	220	66.0	2
P280	52.2	240	51.8	
P320	46.2			
P360	40.5	280	42.3	1
P400	35.0	320	34.3	0
P500	30.2	360	27.3	
P600	25.8	400	22.1	00
P800	21.8			
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.

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.

Grinding Equipment

Stationary grinding papers, often used by students, but uncommon in industrial use, are supplied in strips or rolls, such as for use with the HandiMet 2 roll grinder. 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.

Belt grinders, such as the SurfMet I (Figure 16), are usually present in most laboratories. An alternative approach is to use a high speed disc grinder, such as the SuperMet grinder (Figure 17a). These types of devices use coarse abrasive papers from 60- to 240-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 macro-etched, or for removing

Figure 16. SurfMet I belt grinder

Figure 17a. SuperMet grinder

sectioning damage. Manual grinding work is performed on rotating "wheels"; that is, motor-driven platens, Figure 17a and Figure 18, upon which the grinding paper is attached.

Alternately, in the cases where large sections need to be ground to planarity or a high degree of stock removal is necessary, then a planar grinder that uses a grinding stone should be considered (figure 17b). Such devices can quickly bring even quite
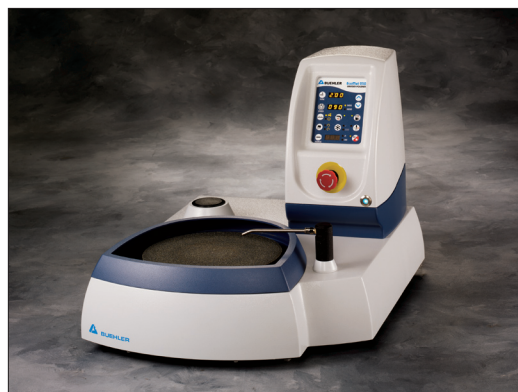

Figure 17b. PlanarMet stone grinder

Figure 18. EcoMet 250 grinder/polisher

large sections of un-mounted material to a planar condition with a good surface finish relatively quickly and so are ideal for macro work

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 disks 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 slurries. 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.

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 metallographic interpretation, both qualitative and quantitative. The polishing technique used should not introduce extraneous structures such as disturbed metal (Figure 19), pitting (Figure 20), dragging out of inclusions, "comet tailing" (Figure 21), staining (Figure 22) or relief
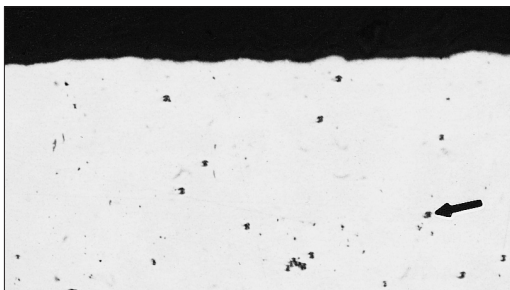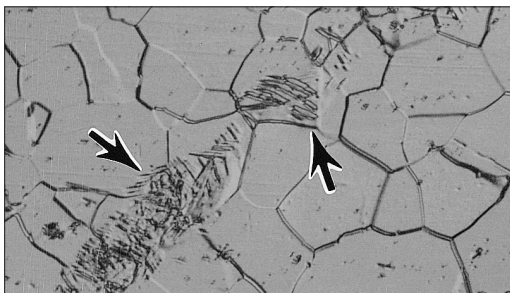

Figure 19. (top) Preparation damage (arrows) in annealed CP titanium (500X, DIC, Kroll's reagent).
Figure 20. (bottom) Pitting (arrow) on the surface of a cold-drawn brass (Cu-20% Zn) specimen (100X)
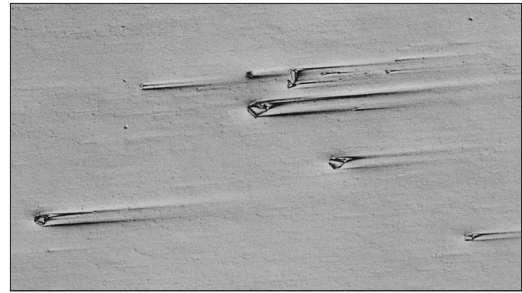

Figure 21. "Comet tails" at large nitrides in an annealed H13 hot work die steel specimen (200X, DIC).

(height differences between different constituents, or between holes and constituents (Figure 23 and 24). Polishing usually is conducted in several stages. Traditionally, coarse polishing generally was conducted with 6- or 3-μm 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 polishing step may be followed by polishing with 1-μm 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.
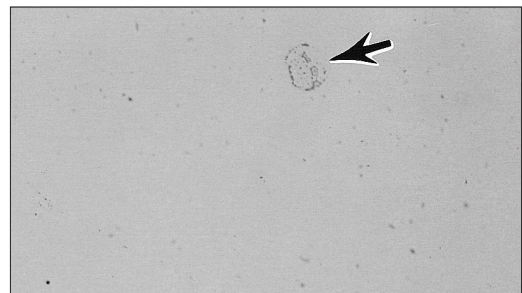
Figure 22. Staining (arrow) on the surface of a of Ti-6% Al-2% Sn-4% Zr-2% Mo prepared specimen (200X).

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 sensi-
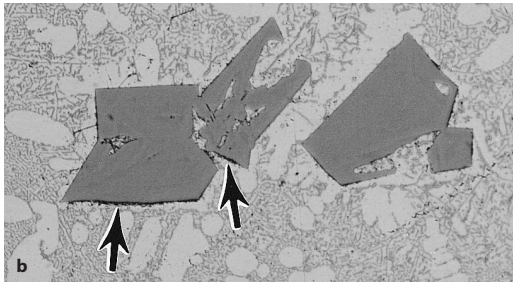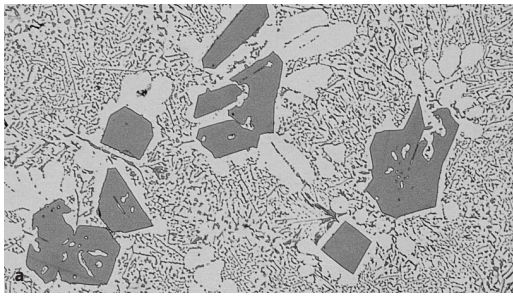
Figure 23. Examples of freedom from relief (a, top) and minor relief (b, bottom) at the edges of large primary hypereutectic silicon particles in an as-cast Al-19.85% Si specimen (200X, aqueous 0.5% HF).
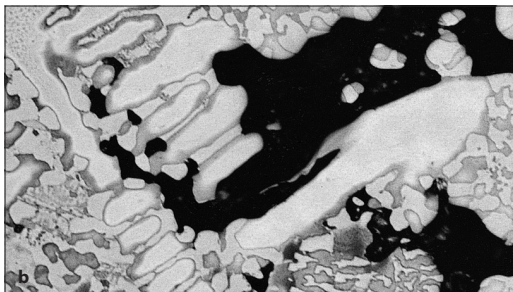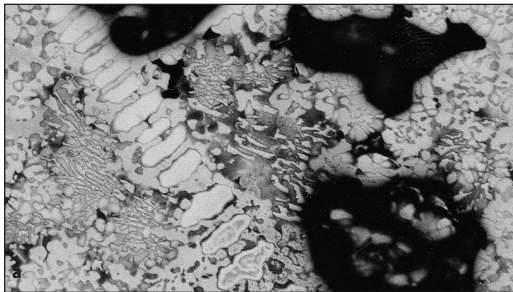
Figure 24. Differences in relief control in a brazed specimen containing shrinkage cavities: a) (top) poor control; and, b) (bottom) good control (100X, glyceregia).

tive adhesive (PSA) 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.

Electrolytic Polishing

Electrolytic polishing can be used to prepare specimens with deformation free surfaces. The technique offers reproducibility and speed. In most cases, the published instructions for electrolytes tell the user to grind the surface to a 600-grit finish and then electropolish for about 1- to 2-minutes. However, the depth of damage after a 600 (P1200) grit finish may be several micrometres but most electropolishing solutions remove only about 1-μm per minute. In this case, the deformation will not be completely removed. In general, electropolished surfaces tend to be wavy rather than flat and focusing may be difficult at high magnifications. Further, electropolishing tends to round edges associated with external surfaces, cracks or pores. In two-phase alloys, one phase will polish at a different rate than another, leading to excessive relief. In some cases, one phase may be attacked preferentially and inclusions are usually attacked. Consequently, electrolytic polishing is not recommended for failure analysis or image analysis work, except possibly as a very brief step at the end of a mechanical polishing cycle to remove whatever minor damage may persist. Electropolishing has been most successful with soft single-phase metals and alloys, particularly where polarized light response needs to be maximized.

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 metallographers 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 metallographer's hands, and the equipment.

Figure 25. EcoMet 300 grinder/polisher and AutoMet 300 power head

Automated Polishing

Mechanical polishing can be automated to a high degree using a wide variety of devices ranging from relatively simple systems, Figure 25, to rather sophisticated, minicomputer, or microprocessor controlled devices, Figure 26. Units also vary in capacity from a single specimen to a half dozen or

Figure 26. BuehlerVanguard 2000 fully automatic specimen preparation system

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 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 metallographers 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 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 metallographic 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 6 lists Buehler's line of polishing cloths, their characteristics and applications.

Table 6. Cloth Selection Guide

Premium Cloths	Recommended μm Size Range	Abrasive Types	Cloth Characteristics	Usage Guide	Applications
UltraPad	15 - 3	Diamond	Hard Woven, No Nap with High Material Removal	Used to Replace Multiple SiC Grinding Steps	Ferrous Materials and Thermal Spray Coatings
UltraPol*	15 - 1	Diamond	Hard Woven, No Nap	Excellent Surface Finish Used to Retain Flatness in Medium to Hard Specimens	Minerals, Coal, and Ceramics, Inclusion Retention in Steels, and Refractory Metals
Nylon*	15 - 1	Diamond	Medium-Hard Woven, No Nap	Used to Retain Flatness and Hard Phases	Ferrous Materials, Sintered Carbides and Cast Irons
TexMet P	15 - 3	Diamond	Hardest Perforated Non-woven Pad for High Material Removal	Used for Material Removal and Flatness of Hard Specimens	Ceramics, Carbides, Petrographic, Hard Metals, Glass, Metal Matrix Composites
TexMet	15 - 3	Diamond, Al_2O_3	Hard Non-woven Pad used for Durability	Used for Harder Specimens and Increased Flatness	Hard Ferrous Materials and Ceramics
TriDent*	9 - 1	Diamond	Softer, Durable, Woven Synthetic, No Nap	Used to Maximize Flatness and Retain Phases while Providing Excellent Surface Finish	Ferrous and Nonferrous Metals, Microelectronics, Coatings
VerduTex*	15 - 1	Diamond	Durable, Medium Hard Synthetic Silk	Used for Rough and Final Polishing	Ferrous and Nonferrous Composite Material and Plastic (CFK, GFK)
VelTex	9 - 1	Diamond	Short-napped, Synthetic Velvet	Ideal for Final Polishing	Steel. Nonferrous and Soft Metals
WhiteFelt	6 - 0.02	Diamond, Al_2O_3, SiO_2	Soft and Durable Matted Wool Cloth	General Usage for Intermediate to Fine Steps	Ferrous and Nonferrous Materials
MicroCloth	5 - 0.02	Diamond, Al_2O_3, SiO_2	Synthetic Cloth with Medium Nap	Most Popular General Usage Final Polishing Cloth	Ferrous and Nonferrous Metals, Ceramics, Composites, PCB's, Cast Irons, Cermets, Plastics, Electronics
MasterTex	1 - 0.05	Al_2O_3, SiO_2	Soft Synthetic Velvet with Low Nap	Softer Final Polishing Cloth	Soft Nonferrous and Microelectronic Packages
ChemoMet	1 - 0.02	Al_2O_3, SiO_2	Soft Synthetic Pad, Micro-nap	General Usage Pad that Removes Smear Metal from Tough Materials during Chemomechanical Polishing	Titanium, Stainless Steels, Lead/Tin Solders, Electronic Packages, Soft Nonferrous Metals, Plastics

Interchangable in many of the following methods

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 oxide may be used, although these are used infrequently. With the exception of diamond, these abrasives are normally suspended in distilled-water, but if the metal to be polished is not compatible with water, other suspensions such as ethylene glycol, alcohol, kerosene or glycerol, may be required. The diamond abrasive should be extended only with the carrier recommended by the manufacturer. Besides the water-based diamond suspension, Buehler also offers oil-based suspensions that are needed to prepare materials sensitive to water.

Diamond abrasives were introduced to metallographers commercially in the late 1940s. However, their use in metallography goes back to at least 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
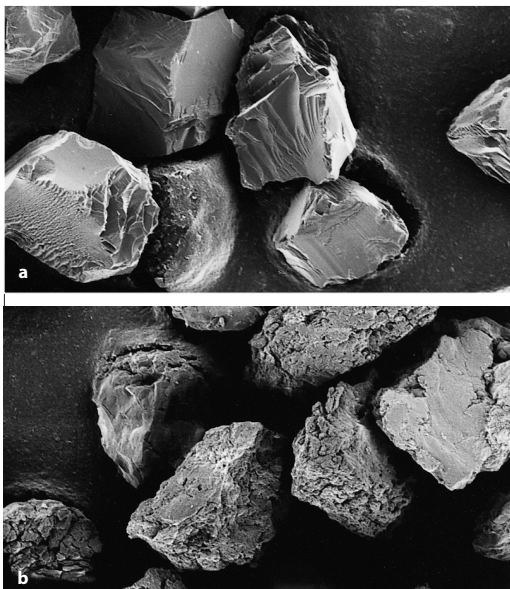
Figure 27. Comparison of monocrystalline (a, top) and polycrystalline (b, bottom) synthetic diamond grain shapes (SEM, 450X).

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 paste use synthetic polycrystalline diamonds. Figure 27 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 9.5. The silica particles are actually nearly spherical in shape, Figure 28, the polishing action is slow,
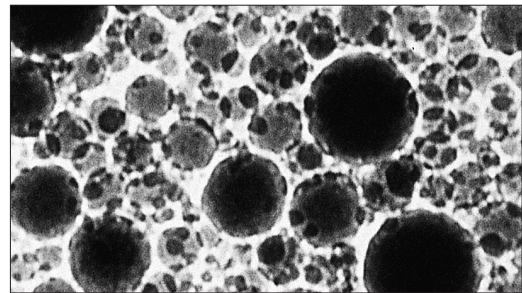

Figure 28. Amorphous silica particles in colloidal silica (TEM, 300,000X).

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 (for 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 abrasive 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.

For routine examinations, a fine diamond abrasive, such as 1-μm, may be adequate as the last preparation step. Traditionally, aqueous fine alumina powders and slurries, such as the MicroPolish II deagglomerated alumina powders and suspensions, have been used for final polishing with medium nap cloths. Alpha alumina (0.3-μm size) and gamma alumina (0.05-μm 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 (basic pH around 9.5) 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 29, are often used for final polishing, particularly with more difficult to prepare materials, for image analysis studies, or for publication quality work.

Figure 29. VibroMet 2 vibratory polisher

HELPFUL HINTS FOR GRINDING/POLISHING

Specimens that contain cracks or pores that are not filled with epoxy may require ultrasonic cleaning to remove abrasive and debris from the openings to avoid contaminating the next step.

Excessive ultrasonic cleaning vibrations can damage the structure of certain soft metals and alloys, particularly precious metals.

To remove a worn out tightly adhering cloth, soak the platen under hot water for a few minutes. This loosens the glue bond and allows you to pull it off more easily.

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 grits, 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 7.

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 rigid, as other polishing cloths may be substituted and one or more of the polishing steps might be omitted. 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 metallography.

Contemporary Methods

New concepts and new preparation materials have been introduced that enable metallographers 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 one more than 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 sheets of SiC are needed then the use of a more aggressive / more durable surface such as ZirMet paper or diamond grinding discs (Apex DGD, Apex DGD Color, UltraPrep diamond discs or UltraPlan stainless steel mesh cloth (charged with diamond)

Table 7. The Traditional Procedure for Preparing Most Metals and Alloys

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	120- (P120) grit SiC water cooled	6 (27)	240-300 Comp.	Until Plane
CarbiMet 2 abrasive discs	220- or 240- (P240 or P280) grit SiC water cooled	6 (27)	240-300 Comp.	1:00
CarbiMet 2 abrasive discs	320- (P400) grit SiC water cooled	6 (27)	240-300 Comp.	1:00
CarbiMet 2 abrasive discs	400- (P600) grit SiC water cooled	6 (27)	240-300 Comp.	1:00
CarbiMet 2 abrasive discs	600- (P1200) grit SiC water cooled	6 (27)	240-300 Comp.	1:00
Canvas cloth	6-μm MetaDi diamond paste with MetaDi fluid extender	6 (27)	120-150 Comp.	2:00
Billiard or Felt cloths	1-μm diamond paste with MetaDi fluid extender	6 (27)	120-150 Comp.	2:00
MicroCloth cloth	Aqueous 0.3-μm α – alumina MicroPolish slurry	6 (27)	120-150 Comp.	2:00
MicroCloth cloth	Aqueous 0.05-μm γ - alumina MicroPolish slurry	6 (27)	120-150 Comp.	2:00

Comp. = Complementary (platen and specimen holder both rotate in the same direction)

may be used. In the case that a lot of material has to be removed then a stone grinding device such as the PlanarMet should be considered. With this huge array of products to choose from, how can the metallographer 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 pads, such as TexMet, 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 0.1-µm 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 slurries 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 (head moves in the direction opposite to the platen) is preferred as the slurry 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 8 to 10. 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 ApexHercules 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 9.

The planar grinding step could also be performed using a 45-µm metal bonded, or a 30-µm resin bonded, UltraPrep disc or with the ApexHercules H rigid grinding disc and 15- or 30-µm diamond, depending upon the material.

Table 8. Generic Four-Step Contemporary Procedure for Many Metals and Alloys

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	120- to 240- (P120 to P280) grit SiC water cooled	6 (27)	240-300 Comp.	Until Plane***
UltraPol cloth	9-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	5:00
TexMet Pad or TriDent cloth	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	4:00
MicroCloth, VelTex or ChemoMet cloths	~0.05-µm MasterMet colloidal silica or MasterPrep alumina suspensions	6 (27)	120-150 Contra**	2:00

* Plus MetaDi fluid extender as desired

** With heads that rotate at >100 rpm, contra may spray the abrasive above the splash guard

*** Until Plane = All specimens in the holder are ground to a common plane and the damage from sectioning has been removed

Comp. = Complementary (platen and specimen holder both rotate in the same direction)

Contra = platen and specimen holder rotate in opposite directions

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.

The ApexHercules 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 30-μm resin bonded diamond disc, or with a second ApexHercules S disc and 15- or 30-μm diamond, depending upon the metal or alloy. For some very difficult metals and alloys, a 1-μm 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 9. Four-Step Contemporary Procedure for Steels Using a Rigid Grinding Disc

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs waterproof paper	120- to 240- (P120 to P280) grit SiC, water cooled	6 (27)	240-300 Comp.	Until Plane
ApexHercules H rigid grinding disc	9-μm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	5:00
TexMet Pad or TriDent cloth	3-μm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	4:00
MicroCloth, VelTex or ChemoMet cloths	~0.05-μm MasterMet colloidal silica or MasterPrep alumina suspensions	6 (27)	120-150 Contra	2:00

*Plus MetaDi fluid extender as desired
Comp.= Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

Table 10. Four-Step Contemporary Procedure for Nonferrous Metals Using a Rigid Grinding Disc

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/ Direction	Time (min:sec)
CarbiMet 2 abrasive discs waterproof paper	220- to 320- (P280 or P400) grit SiC water cooled	5 (22)	240-300 Comp.	Until Plane
ApexHercules S rigid grinding disc	6-μm MetaDi Supreme diamond suspension*	5 (22)	120-150 Comp.	5:00
TriDent cloth	3-μm MetaDi Supreme diamond suspension*	5 (22)	120-150 Comp.	4:00
MicroCloth, VelTex or ChemoMet cloths	~0.05-μm MasterMet colloidal silica or MasterPrep alumina suspensions	5 (22)	120-150 Contra	2:00

*MetaDi fluid extender as desired
Comp.= Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

PROCEDURES FOR SPECIFIC MATERIALS

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 periodic table of the elements 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. The periodic table (pg. 29) has the various groups color coded for convenience.

Of course, metallographers 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 Circuit Boards

• Microelectronic Devices

• Plastics and Polymers

The procedures discussed in this book were developed and tested extensively using machines with an 8-inch (200 mm) diameter platen with six 1.25-inch (32 mm) diameter specimens in a 5-inch (125 mm) diameter holder. These procedures produced the same results when the specimens were prepared using a 10-inch (250 mm) or a 12-inch (300 mm) diameter platen. When using the 8-inch (200 mm) platen and a 5-inch (125 mm) 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 7-inch (175 mm) diameter

specimen holder is used with the 12-inch (300 mm) 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 1.5-inch (25, 32 or 38 mm) 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 1-inch (25 mm), six 1.25-inch (32 mm) or three 1.5-inch (38 mm) diameter specimens, then we can adjust the pressures relative to the standard values in the tables (for six 1.25-inch (32 mm) diameter specimens) using an 8 inch (200 mm) diameter platen format machine. A comparison of the cross-sectional areas would suggest that for the six 1-inch (25 mm) specimens, the pressures could be reduced to 65% of the standard values while for three 1.5-inch (38 mm) 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 12-inch (250 or 300 mm) diameter platen, respectively, compared to an 8-inch (200 mm) platen. This suggests that the times could be reduced to 80 or 67% of that used for the 8-inch (200 mm) diameter platen when using 10- or 12-inch (250 or 300 mm) 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.

PERIODIC TABLE OF ELEMENTS

1 H																	2 He
3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
55 Cs	56 Ba	57 La*	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
87 Fr	88 Ra	89 Ac**															

* Lanthanides   ** Actinides

Lanthanide and Actinide Elements

58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu
90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr

Thermally Spray Coated Specimens
Sintered Carbides, Ceramics, and Composites

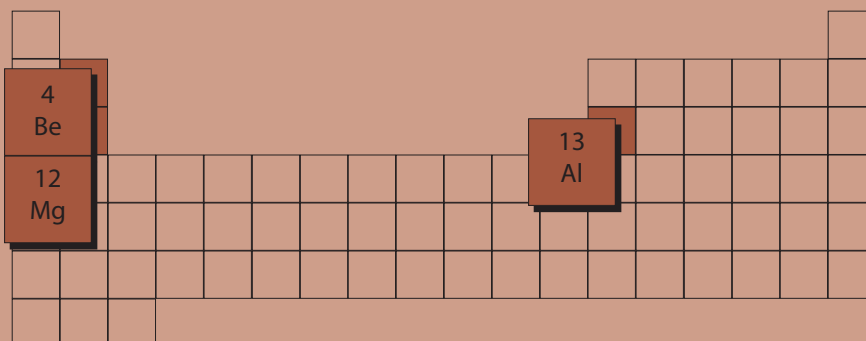**Printed Circuit Boads**
Electronic Materials and Polymers

Metal

Metalloid

Nonmetal

LIGHT METALS: Al, Mg and Be

LIGHT METALS: Al, Mg and Be

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 below. 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.

240- or 320-grit (P280 and P400) SiC waterproof paper may also be used for the planar grinding step. An UltraPol cloth produces better surface

Table 11. Five-Step Procedure for Aluminum Alloys

| Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	220 to 320 (P240 to P400) grit SiC water cooled	5 (22)	240-300 Comp.	Until Plane
UltraPol or UltraPad cloths	9-µm MetaDi Supreme diamond*	5 (22)	120-150 Comp.	5:00
TriDent cloth	3-µm MetaDi Supreme suspension*	5 (22)	120-150 Comp.	4:00
TriDent cloth	1-µm Metadi paste*	5 (22)	120-150 Comp.	2:00
MicroCloth, VelTex or ChemoMet cloth	~0.05-µm Mastermet colloidal silica	5 (22)	120-150 Contra	1:30

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = platen and specimen holder rotate in opposite directions

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 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.
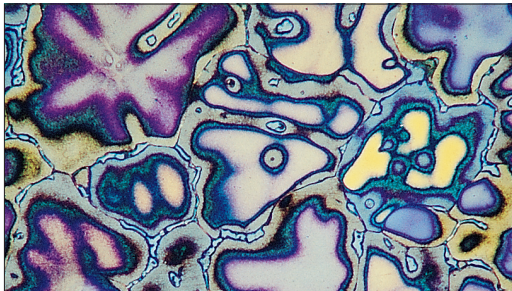
Dendritic segregation in as cast 206 aluminum revealed by color etching with Weck's reagent (polarized light, 200X).

For many aluminum alloys, excellent results can be obtained using a four step procedure, such as shown below in Table 12. This procedure retains all of the intermetallic precipitates observed in aluminum and its alloys and minimizes relief. Synthetic napless cloths may also 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.
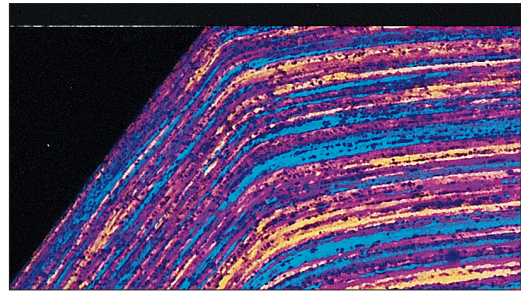
Deformed, elongated grain structure of extruded 6061-F aluminum after shearing revealed by anodizing with Barker's reagent (polarized light, 100X).

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.

Table 12. Four-Step Procedure for Aluminum Alloys

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/ Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	220- to 320- (P240 to P400) grit SiC water cooled	5 (22)	240-300 Comp.	Until Plane
UltraPol cloth	6-μm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	6:00
TriDent cloth	3-μm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	4:00
MicroCloth VelTex or ChemoMet cloth	~0.05-μm MasterMet colloidal silica or MasterPrep alumina suspensions	6 (27)	120-150 Contra	2:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = platen and specimen holder rotate in opposite directions

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 13.

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

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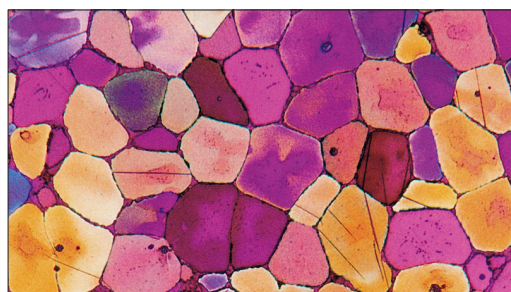
As-cast microstructure of a magnesium alloy with 2.5% of rare earth elements, 2.11% Zn and 0.64% Zr showing a film of the rare earth elements in the grain boundaries, alloy segregation within grains ("coring" revealed by color variations within grains) and a few mechanical twins in the grains (acetic-picral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

Table 13. Four-Step Procedure for Magnesium Alloys

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	220- to 320- (P240 to P400) grit SiC* water cooled	5 (22)	200-250 Comp.	Until Plane
TexMet pad	9-µm MetaDi oil-based diamond slurry	5 (22)	120-150 Contra	6:00
TexMet pad	3-µm MetaDi oil-based diamond slurry	5 (22)	120-150 Contra	5:00
TexMet pad	1-µm MetaDi oil-based diamond slurry	5 (22)	120-150 Contra	4:00
ChemoMet cloth	~0.05-µm MasterPolish or MasterPrep alumina	6 (26)	120-150 Contra	1:30-3:00

*SiC surfaces were coated with wax to minimize embedment
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = platen and specimen holder rotate in opposite directions

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 14 shows a four-step practice for beryllium.
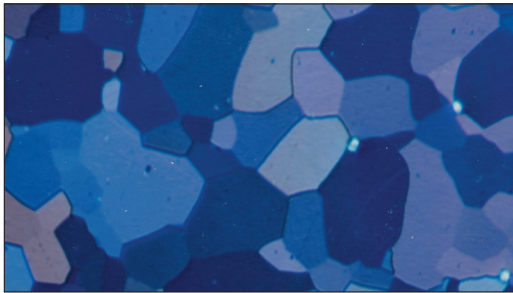
Grain structure of wrought, P/M beryllium (unetched, cross-polarized light, 100X).

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.

Helpful Hints For Magnesium

For polishing, apply diamond paste and use lapping oil, or use Buehler's oil-based MetaDi diamond suspensions, available down to a 0.10-µm size, with a medium nap cloth.

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 14. Four-Step Procedure for Beryllium

| Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	220- to 320- (P240 to P400) grit SiC water cooled	4 (18)	240-300 Comp.	Until Plane
UltraPol cloth	6-µm MetaDi Supreme diamond suspension*	4 (18)	120-150 Comp.	5:00
TriDent cloth	3-µm MetaDi Supreme diamond suspension*	4 (18)	120-150 Comp.	4:00
MicroCloth VelTex or ChemoMet cloth	~0.05-µm MasterMet colloidal silica	3 (13)	80-120 Contra	2:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

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

|						
					50 Sn	51 Sb
	30 Zn				82 Pb	83 Bi
	48 Cd					
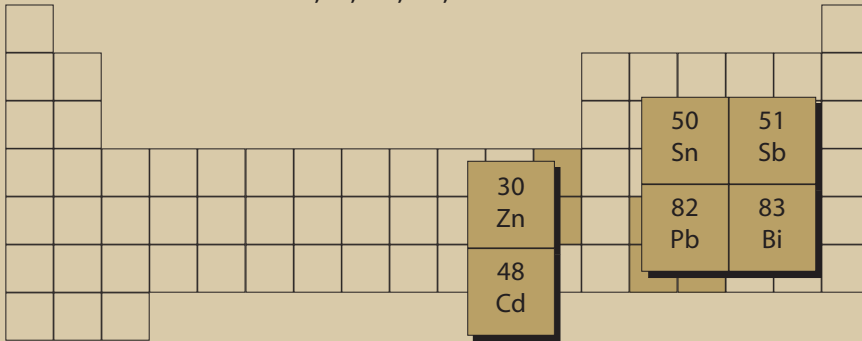
LOW-MELTING POINT METALS: Sb, Bi, Cd, Pb, Sn and Zn

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. 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. 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
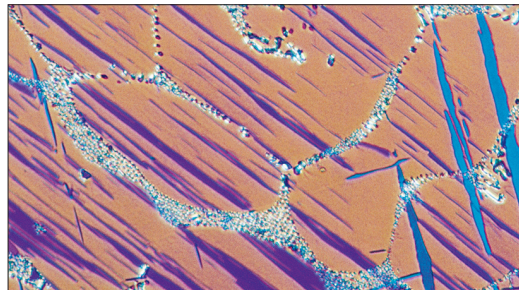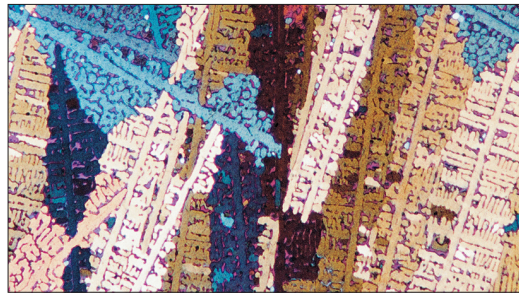
(top) Cadmium dendrites and a Cd-Bi eutectic in an as-cast Cd – 20% Bi alloy (unetched, crossed polarized light plus sensitive tint, 50X). (bottom) As-cast microstructure of Zn – 0.1% Ti – 0.2% Cu alloy exhibiting mechanical twins and a three-phase eutectic (alpha-Zn, Cu-Zn and $Zn_{15}Ti$) in a cellular pattern (Palmerton reagent, crossed polarized light plus sensitive tint, 200X).
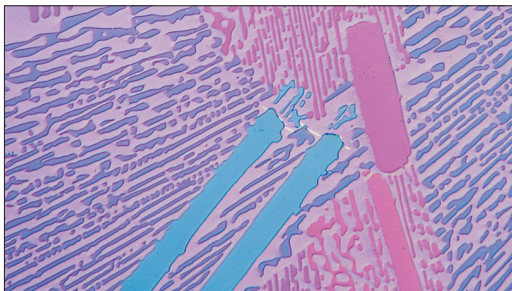
Proeutectic dendrites of Cu_2Sb surrounded by a eutectic mixture of antimony and Cu_2Sb, in an as-cast Sb – 30% Cu specimen (unetched, polarized light, 200X).

prepare. Due to their low melting points, and low recrystallization temperatures, 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.

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 15.

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 1-μm alumina step will remove any embedded silicon carbide particles much more effectively than a diamond abrasive step.

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 (*Wear*, Vol. 16, 1970, p. 351-358) 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.

Table 15. Six-Step Procedure for Low-Melting Point Metals

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	320- (P400) grit SiC water cooled (wax coated)*	4 (18)	150-250 Comp.	Until Plane
CarbiMet 2 abrasive discs	400- (P600) grit SiC water cooled (wax coated)*	4 (18)	150-250 Comp.	0:30
CarbiMet 2 abrasive discs	600- (P1200) grit SiC water cooled (wax coated)*	4 (18)	150-250 Comp.	0:30
CarbiMet 2 MicroCut abrasive discs	800- (P1500) grit SiC water cooled (wax coated)*	4 (18)	150-250 Comp.	0:30
MicroCloth or VelTex cloths	1-μm MicroPolish II deagglomerated alumina suspension	5 (22)	120-150 Comp.	4:00-5:00
MicroCloth or VelTex cloths	MasterPrep 0.05-μm alumina suspension**	4 (18)	80-150 Contra	4:00

*Rub candle wax lightly across revolving disc prior to grinding
**See vibratory polish recommendation in text
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

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

22 Ti	23 V	24 Cr
40 Zr	41 Nb	42 Mo

|			
72 Hf	73 Ta	74 W	75 Re
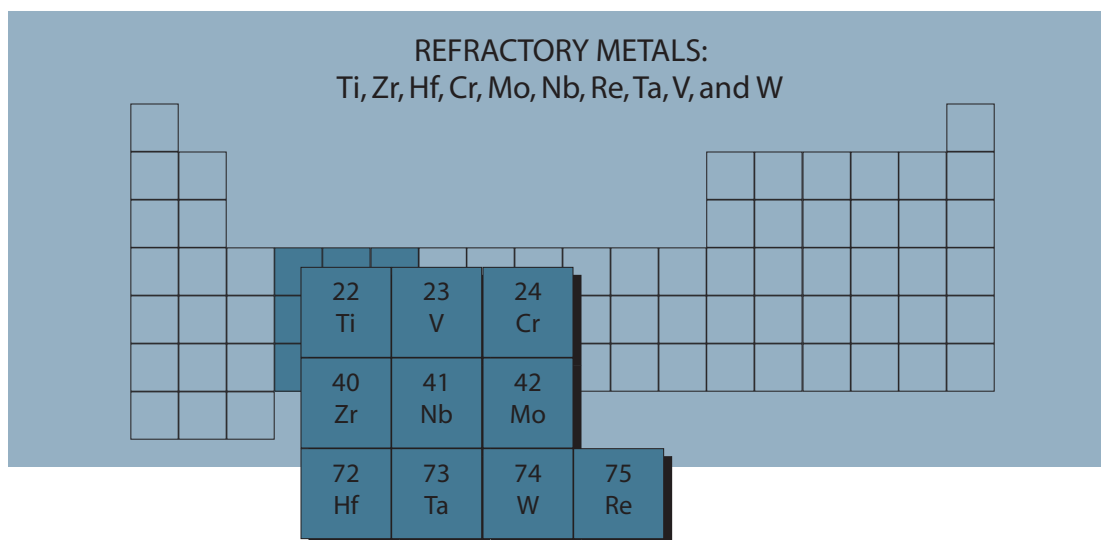
REFRACTORY METALS: Ti, Zr, Hf, Cr, Mo, Nb, Re, Ta, V and W

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
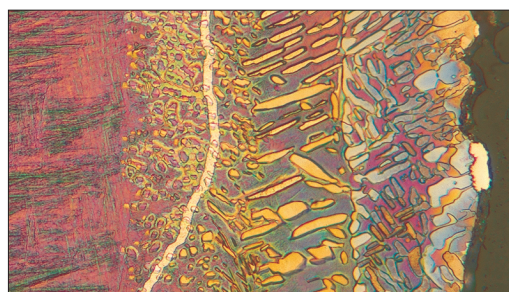
Alpha at the surface of heat treated (1038 °C, water quench) Ti – 3% Cr alloy after tint etching with Beraha's reagent (polarized light, 500X).

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,

Table 16. Three-Step Procedure for Titanium and Titanium Alloys

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	320- (P400) grit SiC, water cooled	6 (27)	240-300 Comp.	Until Plane
UltraPol cloth	9-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Contra	10:00
MicroCloth or VelTex cloths	~0.05-µm MasterMet colloidal silica plus attack polish agent**	6 (27)	120-150 Contra	10:00

*Plus Metadi fluid extender as desired   **See text for agent
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
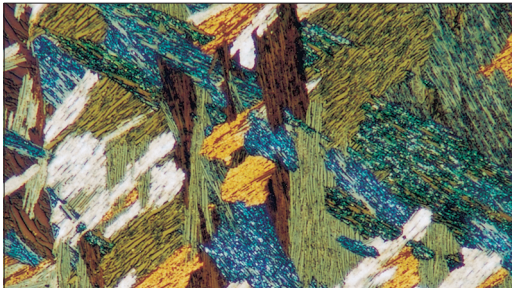Contra = Platen and specimen holder rotate in opposite directions

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 1-µm diamond step, but this is usually unnecessary, see Table 16.

A number of attack polishing agents have been used. The simplest is a mixture of 10 mL hydrogen peroxide (30% concentration – avoid skin contact) and 50 mL 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

Basket-weave alpha-beta structure of as-cast Ti – 6% Al – 4% V revealed by heat tinting (polarized light, 100X).

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. Alternatively, attack polishing additions can be made to the final polishing abrasive slurry, or vibratory polishing may be employed. Table 17 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 5 mL of a chromium trioxide solution (20 g CrO_3 to 100 mL water) added to 95 mL colloidal silica or MasterpPrep alumina slurry. Additions of oxalic, hydrofluoric or nitric acids have also been used. All of these attack polishing addi-

Table 17. Four-Step Procedure for Zirconium and Hafnium

| Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	320- (P400) grit SiC water cooled	5 (22)	200-250 Comp.	Until Plane
UltraPol or UltraPad cloths	9-µm MetaDi Supreme diamond suspension*	5 (22)	150-200 Contra	5:00
TriDent cloth	3-µm MetaDi Supreme diamond suspension*	5 (22)	150-200 Contra	3:00
MicroCloth, VelTex or ChemoMet cloths	~0.05-µm MasterMet colloidal silica plus attack polish agent**	6 (27)	120-150 Contra	7:00

*Plus MetaDi fluid extender as desired   **See text for agent
Comp. = Complementary (platen and specimen holder both rotate in the same direction
Contra = Platen and specimen holder rotate in opposite directions

tions 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 25 mL lactic acid, 15 mL nitric acid and 5 mL 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 15 (add a 5- or 3-μm alumina step) is needed, as diamond is ineffective. Use the chromium trioxide attack polish with the alumina and conclude with Kelly's chemical polish.
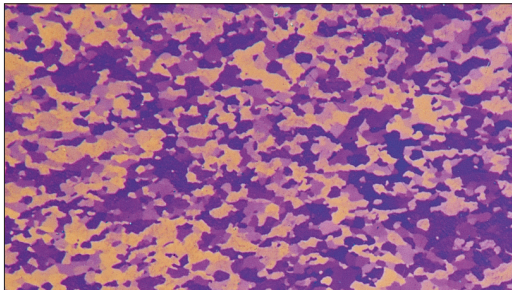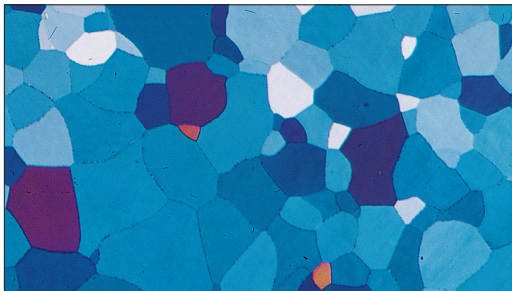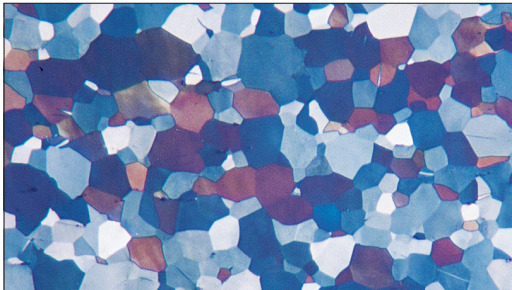
(top) Equiaxed grain structure of wrought zirconium (unetched, crossed polarized light, 100X). (middle) Equiaxed grain structure of wrought hafnium (unetched, crossed polarized light plus sensitive tint, 100X). (bottom) Fine grain recrystallized microstructure of wrought Zr - 1.14% Cr strip in the as-polished condition at 200X using polarized light plus sensitive tint.
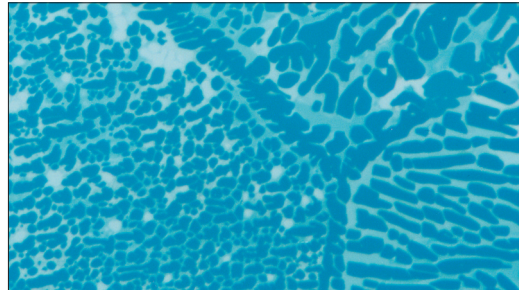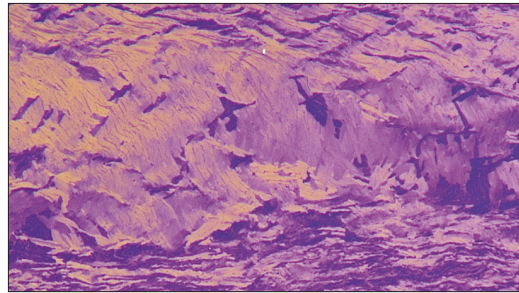
(top) Cold worked microstructure of wrought zirconium alloy XL (Zr - 3% Sn - 0.8% Nb - 0.8% Mo) in the as-polished condition at 200X with polarized light plus sensitive tint. (bottom) Microstructure (α-Zr + AlZr$_3$) of wrought Zr - 8.6% Al in the as-polished condition at 200X with polarized light.

## 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

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.
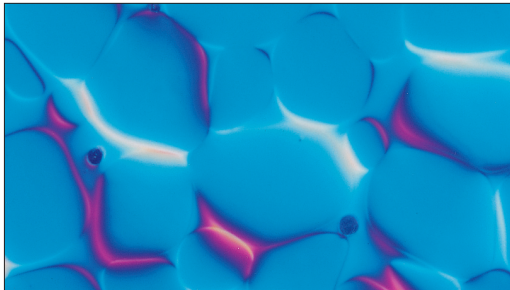
Microstructure of powder-made W – 5.6% Ni – 2.4% Fe revealing tungsten grains surrounded by a nickel-iron matrix (unetched, Nomarski DIC, 200X).

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 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. 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.

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 18.

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 5 mL chromium trioxide solution (20 g CrO_3 in 100 mL water) to 95 mL MasterMet colloidal silica. Avoid skin contact as this is a strong oxidizing solution. A number of chemical
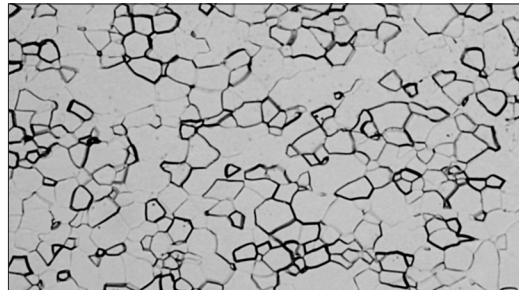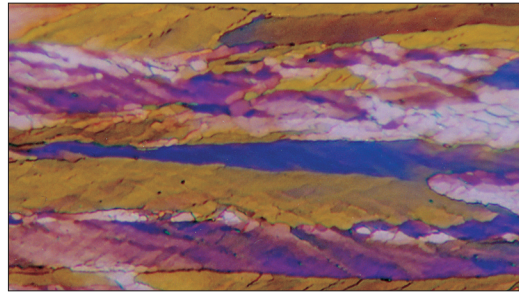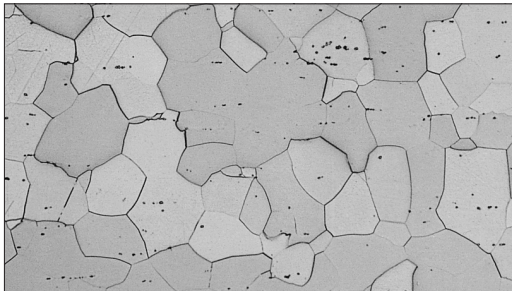
Table 18. Four-Step Procedure for Refractory Metals

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	320- (P400) grit SiC water cooled	6 (27)	150-250 Comp.	Until Plane
UltraPol or UltraPad cloths	9-μm MetaDi Supreme diamond suspension*	6 (27)	150-200 Contra	10:00
TexMet pad TriDent cloth	3-μm MetaDi Supreme diamond suspension*	6 (27)	150-200 Contra	8:00
MicroCloth, VelTex or ChemoMet cloths	~0.05-μm MasterMet colloidal silica plus attack polish agent**	6 (27)	120-150 Contra	5:00

*Plus Metadi fluid extender as desired   **See text for agent

Comp. = Complementary (platen and specimen holder both rotate in the same direction)

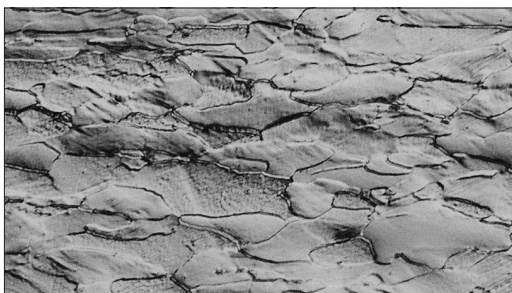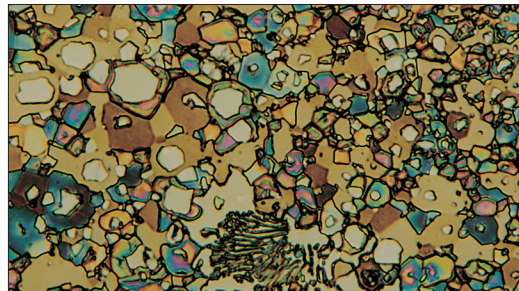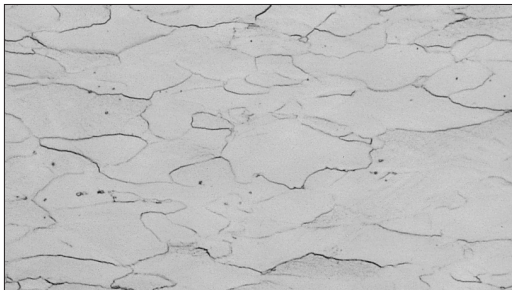Contra = Platen and specimen holder rotate in opposite directions

polishing solutions have been suggested [2]. For Nb, V and Ta, use a solution consisting of 30 mL water, 30 mL nitric acid, 30 mL hydrochloric acid and 15 mL hydrofluoric acid. Swab or immerse at room temperature. An alternative chemical polish for Nb, V and Ta consists of 120 mL water, 6 g ferric chloride, 30 mL hydrochloric acid and 16 mL 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.

(top) 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 (middle) bright field and (bottom) Nomarski DIC which reveals the cold work much better (Murakami's reagent, 500X).
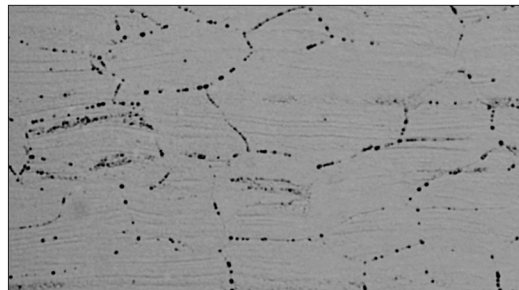

(top) Non-recrystallized grain structure of wrought pure molybdenum, longitudinal plane (500X, polarized light, etch of water, hydrogen peroxide (30% conc) and sulfuric acid in 7:2:1 ratio).
(bottom) Equiaxed alpha grain structure in wrought pure vanadium (200X, etch: glycerol-nitric acid-hydrofluoric acid, 1:1:1ratio).


(top) 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). (bottom) 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: Fe

| 26 |
| Fe |
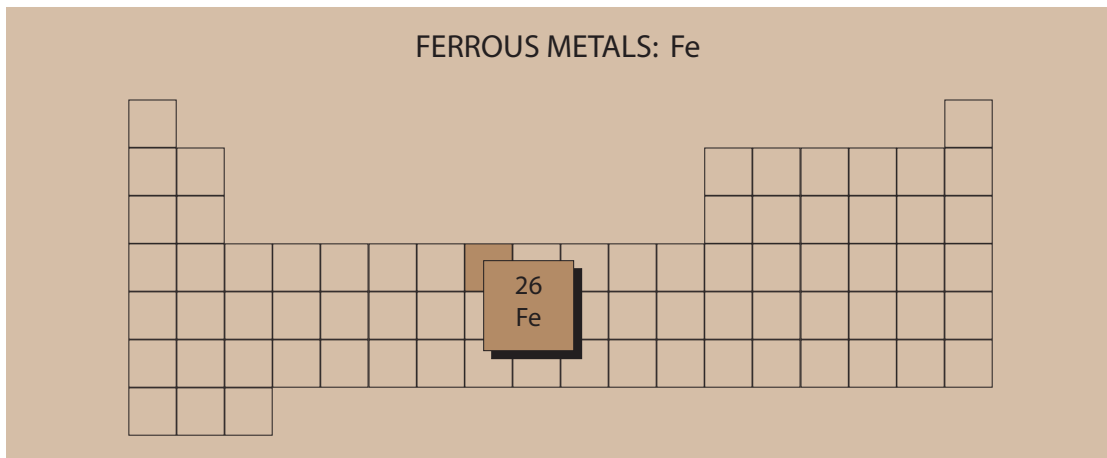
FERROUS METALS

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. 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.
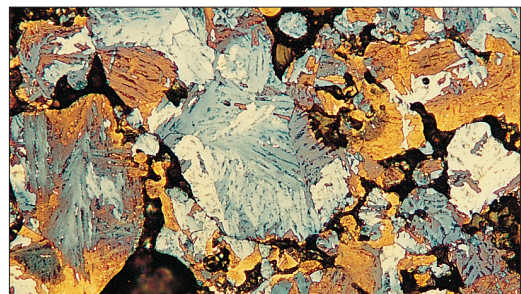
Lath martensitic microstructure of an unconsolidated powder metallurgy steel gear containing substantial porosity (Klemm's I reagent, polarized light, 200X).

Table 19. Four-Step Procedure for Hard Steels using the Buehler Hercules H disc

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
Apex DGD or UltraPrep metal-bonded disc	45-µm diamond, water cooled	6 (27)	240-300 Comp.	Until Plane
ApexHercules H rigid grinding disc	9-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	5:00
TriDent cloth	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	3:00
MicroCloth, VelTex or ChemoMet cloths	MasterPrep 0.05-µm alumina suspension, or MasterMet colloidal silica	6 (27)	120-150 Contra	2:00

*Plus Metadi fluid extender desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

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 9 presented a variation of the method given in Table 19.

This practice is also useful for cast iron specimens including graphitic cast irons. 240 (P280) grit SiC paper can be substituted for the UltraPrep disc or another ApexHercules H disc can be used for planar grinding with 30-μm MetaDi Supreme diamond suspension. 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 30-μm resin bonded Ultra-Prep disc, or 240- (P280) grit SiC paper, for step 1. The ApexHercules S disc can be used in place of the ApexHercules H disc but this is not usually necessary. However, with either disc, use 6-μm MetaDi Supreme for the second step instead of 9-μm, as shown in Table 20.

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 etch ing, follow

Table 20. Four-Step Procedure for Soft Steels using the BuehlerHercules S disc

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
Apex DGD or UltraPrep resin-bonded disc	30-μm diamond, water cooled	6 (27)	240-300 Comp.	Until Plane
ApexHercules S rigid grinding disc	6-μm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	5:00
TriDent cloth	3-μm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	3:00
MicroCloth, VelTex or ChemoMet cloths	MasterPrep 0.05-μm alumina suspension, or MasterMet colloidal silica	6 (27)	120-150 Contra	2:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

Table 21. Three-Step Procedure for Steels

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	120- to 320- (P120 to P400) grit SiC, water cooled	6 (27)	240-300 Comp.	Until Plane
ApexHercules H or S rigid grinding disc	3-μm MetaDi Supreme Diamond Suspension*	6 (27)	120-150 Comp.	5:00
MicroCloth VelTex or ChemoMet cloths	MasterPrep 0.05-μm Alumina Suspension, or MasterMet Colloidal Silica	6 (27)	120-150 Contra	5:00

*Plus MetaDi Fluid extender as desired
Comp. = Complementary (platen and specimen holder rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

this practice with a brief vibratory polish using the cloths and abrasives in the last step.

Many steels, particularly the harder steels, can be prepared in three steps with excellent results. A recommended practice is given in Table 21.

For soft alloys, use 240- or 320- (P280 or P400) grit SiC paper; for 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. Planar grinding can also be performed using 45-μm metal-bonded or 30-μm resin-bonded UltraPrep diamond discs. For softer steels, use the ApexHercules S disc for best results. The UltraPol 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 ApexHercules S disc or the Ultra-Pol cloth for best results. Start with 120- (P120) grit SiC paper only if it is a very hard martensitic stainless steel, such as type 440C. For the martensitic grades, planar grinding can be performed using a 45-μm metal-bonded diamond UltraPrep

disc. For softer stainless steels, use the 30-μm resin-bonded diamond UltraPrep disc for planar grinding. Another alternative is a second Apex Hercules disc, either H or S, depending upon the hardness of the grade, and a 30-μm MetaDi Supreme polycrystalline diamond suspension. The solution annealed austenitic stainless steels and the fully ferritic stainless steels are the most difficult to prepare. It may be helpful to add a 1-μm 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 22.
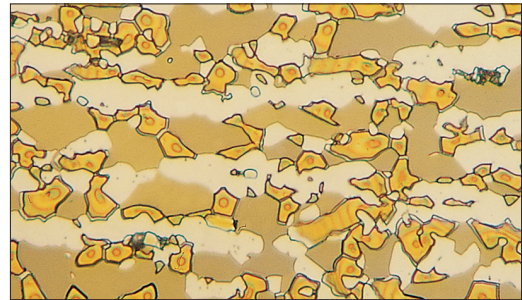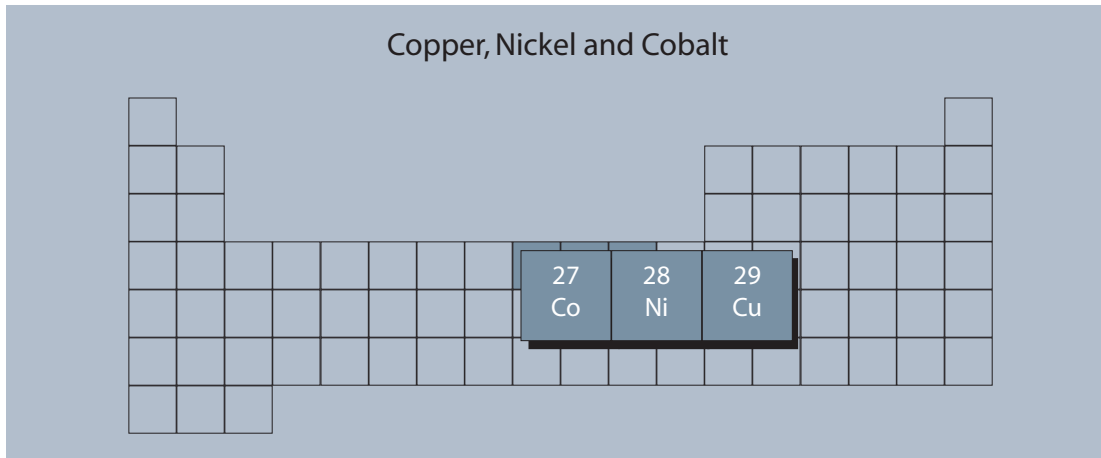

Microstructure of a duplex (ferrite tan, austenite white) stainless steel held at 816º C for 48 hours which forms sigma phase (orange particles) revealed by electrolytic etching with aqueous 20% NaOH at 3 V dc, 9 s (500X).

Table 22. Four-Step Contemporary Procedure for Stainless Steels and Maraging Steels

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	120- to 240- (P120 to P280) grit SiC, water cooled	6 (27)	240-300 Comp.	Until Plane
ApexHercules S rigid grinding disc or UltraPol cloth	9-μm MetaDi Supreme Diamond Suspension*	6 (27)	120-150 Comp.	5:00
TriDent cloth	3-μm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	5:00
MicroCloth, VelTex or ChemoMet cloths	MasterPrep 0.05-μm alumina suspension, or MasterMet colloidal silica	6 (27)	120-150 Contra	2:30

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

Copper, Nickel and Cobalt

| 27 | 28 | 29 |
| Co | Ni | Cu |

COPPER, NICKEL and COBALT

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 23.

Planar grinding can be performed using the 45- or 15-µm metal-bonded or the 30-µm resin-bonded UltraPrep discs. Use the resin-bonded disc for the soft copper grades and copper alloys.
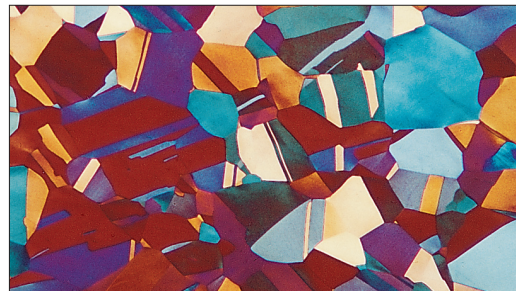
Alpha grains containing annealing twins in phosphorous-deoxidized arsenical bronze that was annealed and lightly cold drawn (Klemm's I reagent, polarized light, 50X).

Table 23. Five-Step Procedure for Cu and Cu Alloys

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	220- to 320- (P240 to P400) grit SiC water cooled	5-6 (22-27)	240-300 Comp.	Until Plane
UltraPol cloth or ApexHercules S rigid grinding disc	6-µm MetaDi Supreme diamond suspension*	5-6 (22-27)	120-150 Comp.	5:00
TriDent cloth or TexMet pads	3-µm MetaDi Supreme diamond suspension*	5-6 (22-27)	120-150 Comp.	3:00
TriDent cloth	1-µm MetaDi Supreme Diamond Suspension*	5-6 (22-27)	120-150 Comp.	2:00
MicroCloth, VelTex or ChemoMet cloths	~0.05-µm MasterMet colloidal silica or MasterPrep alumina suspensions	5-6 (22-27)	120-150 Contra	1:30-2:00

*Plus MetaDi Fluid extender as desired
Comp.= Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

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 super-alloys. Solution annealed superalloys are always more difficult to prepare than age hardened superalloys. Age hardened superalloys can be prepared using the ApexHercules H disc; for all other nickel alloys, use the ApexHercules 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 24.

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 speci-mens, or when color etching is being performed, a 1-µm 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 30-µm resin-bonded UltraPrep diamond disc or with 240- (P280) or 320- (P400) grit SiC papers with equal success, see Table 25.

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

Table 24. Four-Step Procedure for Nickel-Based Superalloys and Ni-Cr-Fe Alloys

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	220- to 240- (P240 to P280) grit SiC water cooled	6 (27)	240-300 Comp.	Until Plane
ApexHercules H or ApexHercules S rigid grinding disc	9-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	5:00
Trident cloth	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	5:00
MicroCloth, VelTex ChemoMet cloths	MasterPrep 0.05-µm alumina suspension or MasterMet colloidal silica	6 (27)	120-150 Contra	2:00-5:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

Table 25. Five-Step Procedure for Ni, Ni-Cu and Ni-Fe Alloys

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
UltraPrep resin-bonded disc	30-µm diamond water cooled	5 (22)	200-300 Comp.	Until Plane
UltraPol cloth or ApexHercules S rigid grinding disc	9-µm MetaDi Supreme diamond suspension*	6 (27)	100-150 Comp.	5:00
TriDent cloth or TexMet pads	3-µm MetaDi Supreme diamond suspension*	6 (27)	100-150 Comp.	3:00
TriDent cloth	1-µm MetaDi Supreme diamond suspension*	6 (27)	100-150 Comp.	2:00
MicroCloth, VelTex or ChemoMet cloths	~0.05-µm MasterMet colloidal silica or MasterPrep alumina suspensions	6 (27)	80-150 Contra	1:30-2:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

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.
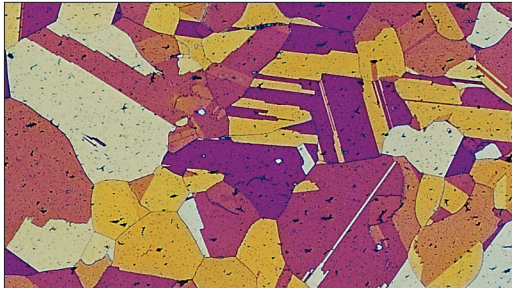

Alpha grains containing annealing twins of solution annealed and double aged Waspaloy nickel-based superalloy (Beraha's reagent, 100X).

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 26.

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 40 mL lactic acid, 30 mL hydrochloric acid and 5 mL nitric acid (immerse). A wide variety of Co-based alloys have been prepared with the above method without need for chemical polishing. The 1-µm diamond step could be eliminated for routine work.
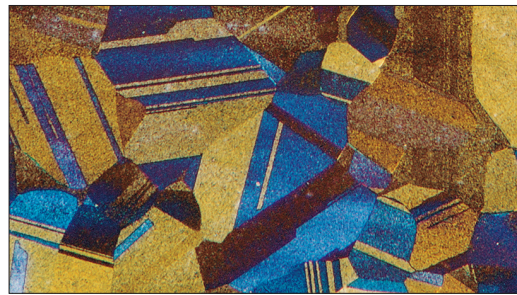
Equiaxed grain structure of Elgiloy (Co – 20% Cr – 15% Fe – 15% Ni – 7% Mo – 2% Mn – 0.15% C – 0.05% Be) after hot rolling and annealing revealing annealing twins (Beraha's reagent, crossed polarized light plus sensitive tint, 100X).
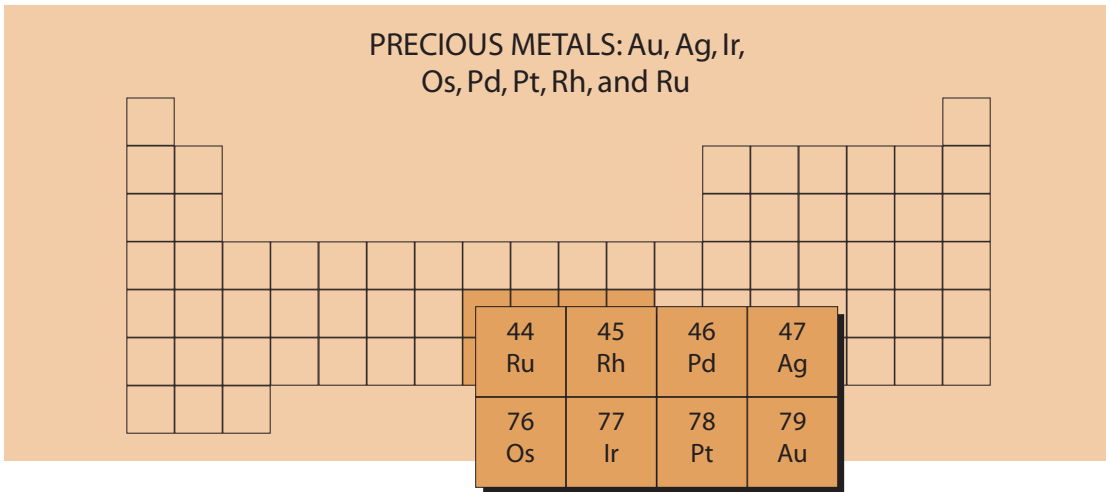
Table 26. Five-Step Procedure for Cobalt

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (Waterproof Paper)	220- to 320- (P240 to P400) grit SiC water cooled	6 (27)	250-300 Contra	Until Plane
UltraPol cloth or ApexHercules S rigid grinding disc	9-µm MetaDi Supreme diamond suspension*	6 (27)	100-150 Contra	5:00
TexMet pad	3-µm MetaDi Supreme diamond suspension*	6 (27)	100-150 Contra	5:00
TriDent cloth or TexMet pads	1-µm MetaDi Supreme diamond suspension*	6 (27)	100-150 Contra	3:00
MicroCloth, VelTex or ChemoMet cloths	~0.05-µm MasterMet colloidal silica or MasterPrep alumina suspensions	6 (27)	80-120 Contra	2:00-3:00

*Plus Metadi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

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

| 44 Ru | 45 Rh | 46 Pd | 47 Ag |
| 76 Os | 77 Ir | 78 Pt | 79 Au |

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 Microelectronic Devices). 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. 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
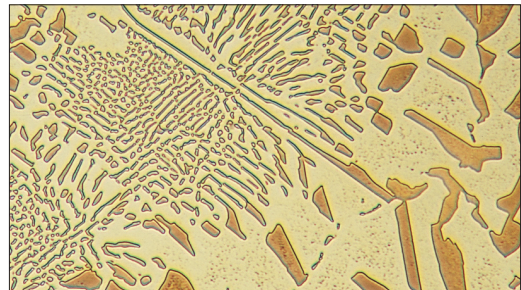
Eutectic microstructure of Ag – 28% Cu where Klemm's reagent has colored the copper particles and the silver phase is uncolored (500X).

Table 27. Stewart's Manual Procedure for Precious Metals

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	220- to 240- (P240 to P280) grit SiC water cooled	moderate	400	Until Plane
CarbiMet 2 abrasive discs	320- (P400) grit SiC water cooled	moderate	400	1:00
CarbiMet 2 abrasive discs	400- (P600) grit SiC water cooled	moderate	400	1:00
CarbiMet 2 abrasive discs	600- (P1200) grit SiC water cooled	moderate	400	1:00
TriDent cloth	6-µm MetaDi Supreme diamond suspension	moderate	400	2:00
MasterTex cloth	1-µm MetaDi Supreme diamond suspension	moderate	400	2:00

Comp. = Complementary (platen and specimen holder both rotate in the same direction)

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 is shown in Table 27.

This can be followed by a brief vibratory polish using colloidal silica on MasterTex, ChemoMet or MicroCloth pads to further enhance the quality of the preparation; but, a 1-μm 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. TexMet pads are 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 I cloth and MasterPrep alumina. Due to their excellent corrosion resistance, colloidal silica is not effective as an abrasive for precious metals. The ChemoMet pad has many fine pores to hold the abrasive. The cycle is given in Table 28.

For 18 karat gold and higher (≥75% Au), it is necessary to use an attack polish agent in the final step. An aqueous solution of 5 g CrO_3 in 100 mL water works well. Mix 10 mL of the attack polish agent with 50 mL of MasterPrep suspension. This will thicken, so add about 20-30 mL 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.
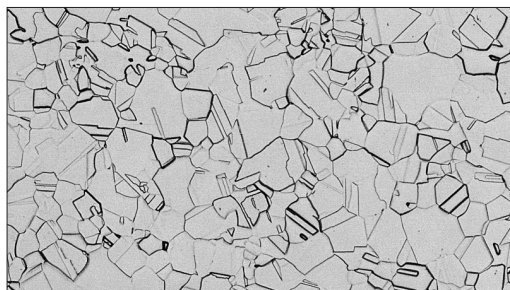
Equiaxed grain structure of cold rolled and annealed 18-karat gold (Neyoro 28A: 75% Au – 22% Ag – 3% Ni) revealing annealing twins (equal parts 10% NaCN and 30% H_2O_2, 50X).

Table 28. Five-Step Automated Procedure for Precious Metals

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs	220- to 320- (P240 to P400) grit water cooled	3 (13)	150-240 Comp.	Until Plane
TexMet pad	9-μm MetaDi II diamond paste*	3 (13)	150-240 Comp.	5:00
TexMet pad	3-μm MetaDi II diamond paste*	3 (13)	150-240 Comp.	3:00
TexMet pad	1-μm MetaDi II diamond paste*	3 (13)	150-240 Comp.	2:00
ChemoMet cloth	MasterPrep alumina suspension	6 (26)	100-150 Comp.	2:00

* Use water as the lubricant, but keep the pad relatively dry
Comp. = Complementary (platen and specimen holder both rotate in the same direction)

THERMALLY-SPRAY COATED SPECIMENS

Thermally sprayed 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 and use 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 13-15). A variety of procedures can be used. The ApexHercules H rigid grinding disc produces exceptional edge flatness for these

Table 29. Four-Step Procedure for TSC and TBC Specimens with Metallic Coatings

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
ApexHercules H rigid grinding disc	30-µm MetaDi Supreme diamond suspension*	5 (22)	240-300 Contra	Until Plane
ApexHercules H rigid grinding disc	6-µm MetaDi Supreme diamond suspension*	5 (22)	150-200 Contra	4:00
TriDent cloth	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	3:00
MicroCloth, VelTex or ChemoMet cloths	MasterPrep 0.05-µm alumina suspension or MasterMet colloidal silica	4 (18)	120-150 Contra	2:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

Table 30. Alternate Four-Step Procedure for TSC and TBC Specimens with Metallic Coatings
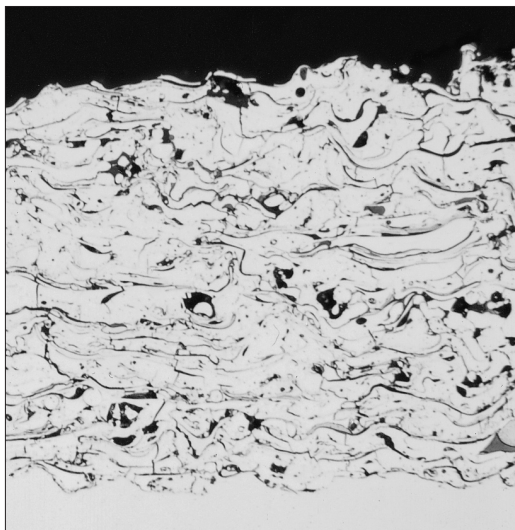
Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
ApexHercules H rigid grinding disc	30-µm MetaDi Supreme diamond suspension*	5 (22)	240-300 Contra	Until Plane
UltraPol cloth	9-µm MetaDi Supreme diamond suspension*	5 (22)	150-200 Contra	4:00
TriDent cloth	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	3:00
MicroCloth, VelTex or ChemoMet cloths	MasterPrep 0.05-µm alumina suspension or MasterMet colloidal silica	4 (18)	120-150 Contra	2:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

specimens. Two four step procedures for TSC and TBC specimens using the Apex Hercules H rigid grinding disc for specimens with metallic coatings and one procedure for specimens with ceramic coatings are given, see Tables 29-31.

The 30-µm resin bonded, or the 45-µm metal bonded UltraPrep diamond discs, can be substituted for the planar grinding step. (Table 29 and 30). UltraPol or UltraPad cloths can be used in the second step. (Table 29 and 31).

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).
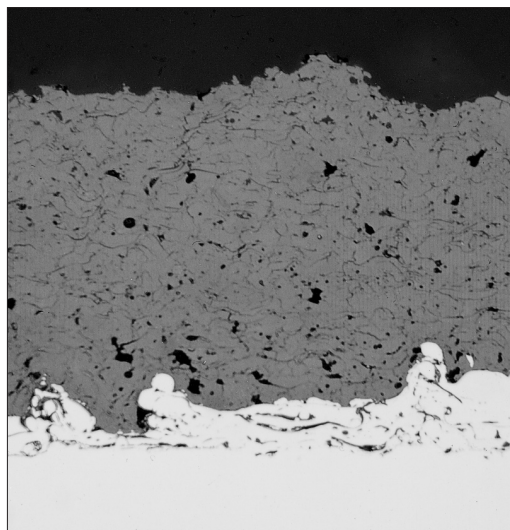
Microstructure of a steel substrate covered by two thermally-sprayed layers, a NiAl bond coat and yittria-zirconia top coat (unetched, 100X). The bond coat contains pores, linear detachments and inclusions while the top coat is quite porous.

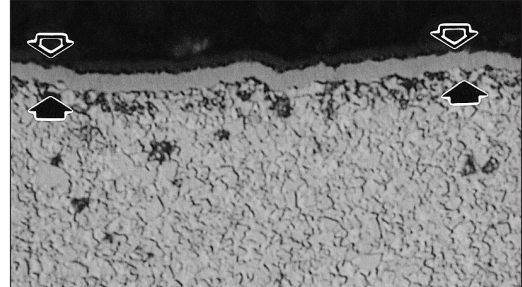**Table 31. Four-Step Procedure for TSC and TBC Specimens with Ceramic Coatings**

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
Apex DGD or UltraPrep metal-bonded disc	45-µm disc water cooled	5 (22)	240-300 Contra	Until Plane
ApexHercules H rigid grinding disc	9-µm MetaDi Supreme diamond suspension*	5 (22)	150-180 Contra	4:00
TexMet pad or TriDent cloth	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	3:00
MicroCloth, ValTex or ChemoMet cloths	MasterPrep 0.05-µm alumina suspension or MasterMet colloidal silica	8 (36)	120-150 Contra	2:30

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

SINTERED CARBIDES

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). 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 ApexHercules H rigid grinding discs. A further option would be the use of the traditional metal-bonded diamond discs.

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).

Table 32. Four-Step Procedure for Sintered Carbides using UltraPrep Discs

| Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
Apex DGD or UltraPrep metal-bonded disc	45-µm diamond water cooled	6 (27)	240-300 Contra	5:00**
Apex DGD or UltraPrep metal-bonded disc	9-µm diamond water cooled	6 (27)	240-300 Contra	4:00
TriDent cloth, UltraPol cloth, TexMet pad or Nylon cloth	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Contra	3:00
TriDent, UltraPol or Nylon cloths	~0.05-µm MasterMet colloidal silica or MasterPrep alumina suspensions	6 (27)	120-150 Contra	2:00

*Plus MetaDi fluid extender as desired

** Grind longer than "Until Plane"

Comp. = Complementary (platen and specimen holder both rotate in the same direction)

Contra = Platen and specimen holder rotate in opposite directions

Table 33. Four-Step Procedure for Sintered Carbides using ApexHercules H Discs

| Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
ApexHercules H rigid grinding disc	30-µm MetaDi Supreme diamond suspension*	6 (27)	240-300 Contra	5:00**
ApexHercules H rigid grinding disc	9-µm MetaDi Supreme diamond suspension*	6 (27)	240-300 Contra	4:00
TriDent cloth, UltraPol cloth, TexMet pad or Nylon cloth	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	3:00
MicroCloth, VelTex or ChemoMet cloths	~0.05-µm MasterMet colloidal silica or MasterPrep alumina suspensions	6 (27)	120-150 Contra	2:00

*Plus MetaDi fluid extender as desired
** Grind longer than "Until Plane"
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

If a greater amount of material must be removed in the planar grinding step, use either the 30-μm resin bonded or the 45-μm metal-bonded UltraPrep disc for step one, in Table 33.
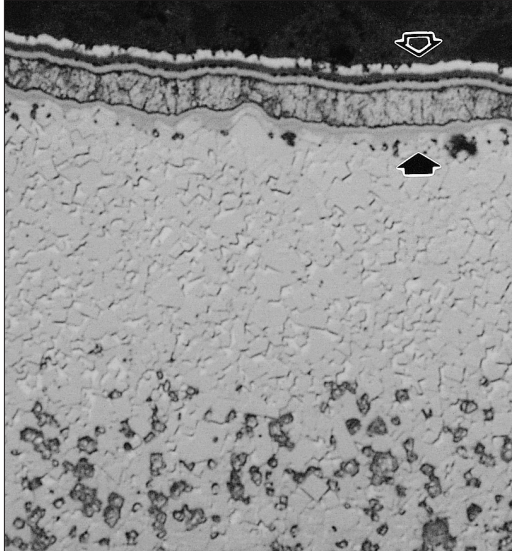
Microstructure at the surface of a multilayered, CVD-coated WC - 8% Co cutting tool. The arrows point to the CVD layers of TiCN, TiN, TiC and alumina. The region below the coatings is made higher in Co to improve crack resistance and complex carbide forming elements (Ta, Ti and Nb) are added to the matrix (dark spots at bottom) for wear resistance (Murakami's reagent, 1000X).

The final step in these two procedures, Tables 32 and 33, 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 3-μm diamond step. For routine examination there is little need to go beyond the third step. It is also possible to use a 1-μm diamond step, similar to step three, but for 2 minutes, as an alternative fourth step.
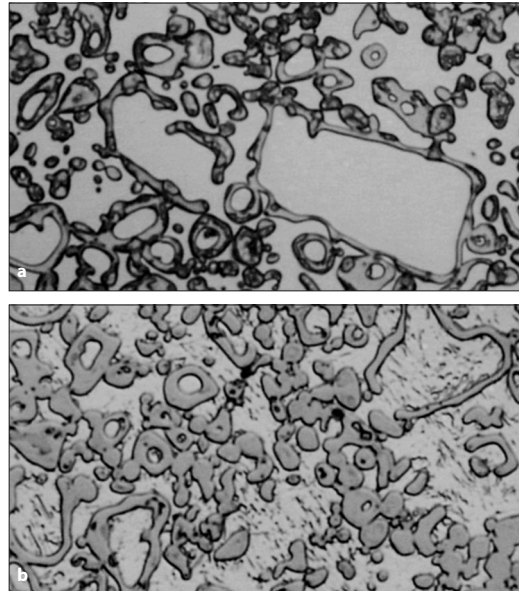
Microstructure of cold isostatically pressed and age hardened Ferro-Titanit Nikro 128 (Fe - 13.5% Cr - 9% Co - 4% Ni - 30% TiC) cutting tool;
a) etched with Murakami's reagent at room temperature to darken the TiC; and b) electrolytic etched with aqueous 1% chromium trioxide at 2 V dc, 30 s to reveal the martensitic matrix (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 nap-less surface, such as TriDent, TexMet or UltraPol pads. To see the phase boundaries between WC particles and the cobalt binder, use a medium nap surface, such as MicroCloth, MasterTex pads.

CERAMICS

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.
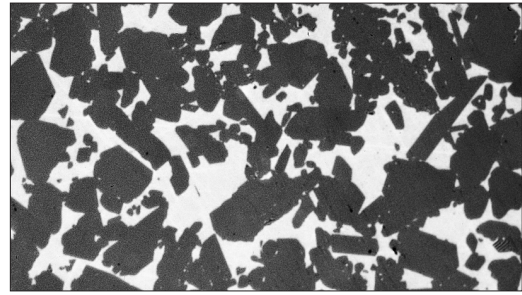
Microstructure of hot-pressed silicon nitride (binder unknown) (unetched, 500X).

Table 34. Four-Step Procedure Suitable for Most Ceramic Materials

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
Apex DGD or UltraPrep metal-bonded disc	45-µm diamond MetaDi fluid for coolant	8 (36)	120-150 Comp.	Until Flat
ApexHercules H rigid grinding disc	9-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	4:00
ApexHercules S rigid grinding disc	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	3:00
TexMet, Nylon or UltraPol cloths	1-µm MetaDi Supreme diamond suspension* or MasterPolish 2 suspension	10 (44)	120-150 Contra	5:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

**Table 35. Four-Step Procedure Suitable for Most Ceramic Materials**

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
Apex DGD or UltraPrep metal-bonded disc	45-µm diamond MetaDi fluid for coolant	8 (36)	120-150 Contra	Until Flat
UltraPad cloth	15-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	6:00
UltraPad cloth or TexMet pad	6-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	4:00
TexMet or Nylon or UltraPol cloths	3-µm MetaDi Supreme diamond suspension and MasterMet colloidal silica (50:50 simultaneously)	8 (36)	120-150 Contra	4:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

Ceramics

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 34 and 35.

MasterPolish 2 suspension is specifically formulated for final polishing ceramic materials and will yield a better surface finish than 1-μm diamond.
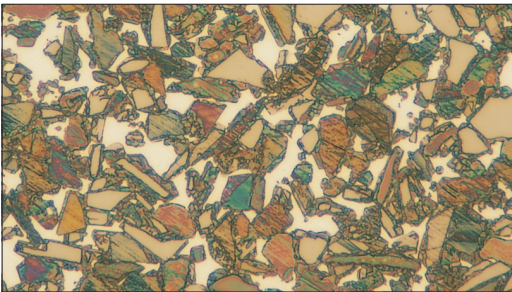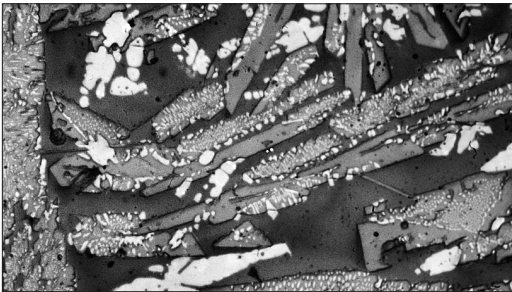
(top) Microstructure of an alumina (matrix), zirconia (gray), silica (white) refractory material (unetched, 200X). (bottom) Silicon - SiC ceramic etched electrolytically with 10% oxalic acid (500X).
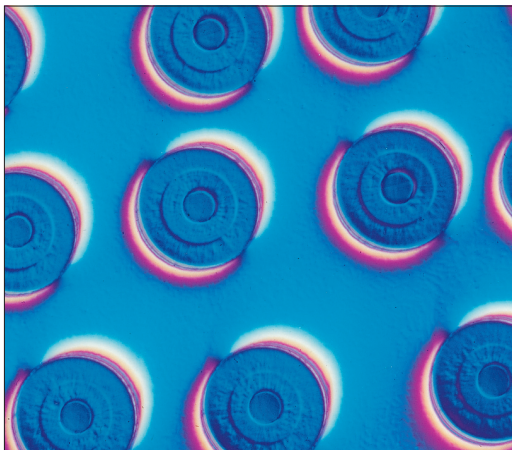
COMPOSITES

Composites cover a wide range of compositions, general grouped under the subcategories metal-matrix composites (MMC), polymer-matrix composites (PMC) and ceramic-matrix composites (CMC). Consequently, preparation schemes are quite difficult to generalize due to the extreme range of materials employed, differences in hardness and grinding/polishing characteristics; so, relief control is a big issue. Pull out problems are also very common, especially with PMCs. Sectioning frequently produces considerable damage that must be removed in the initial preparation step. Mounting with castable epoxy resin along with vacuum impregnation is frequently performed.

(top) Microstructure of a metal-matrix composite, SiC in a CP titanium matrix (unetched, Nomarski DIC, 100X).
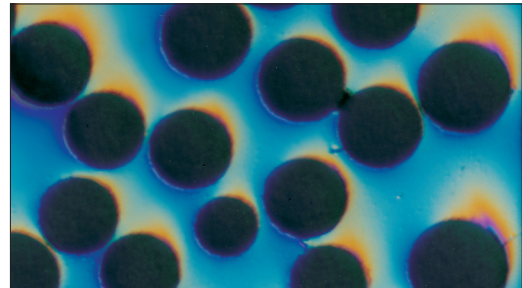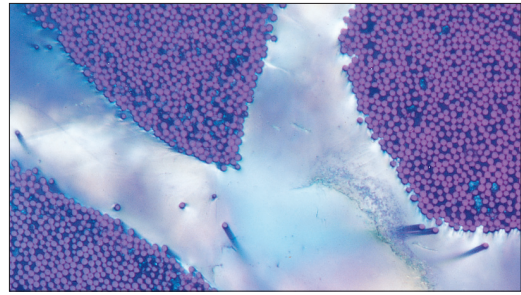
(top) Microstructure of a polymer-matrix composite, graphite fabric-reinforced polysulfone (unetched, polarized light, 100X). (bottom) Microstructure of alumina fibers in an aluminum - lithium alloy matrix (500X, as-polished, Nomarski DIC).
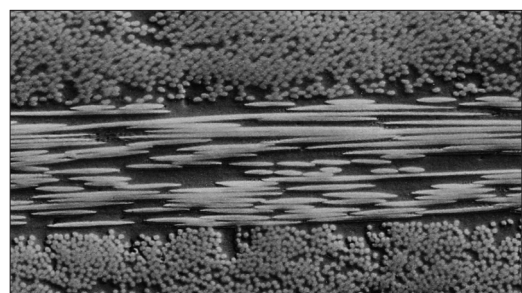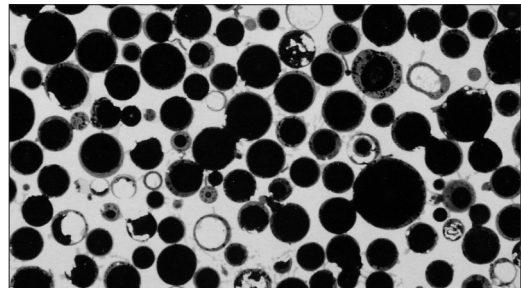
(top) Synthetic foam made with hollow ceramic spheres added to 7075 aluminum (100X original magnification, magnification bar is 100 μm long). Etched with aqueous 0.5% HF. (bottom) Carbon-reinforced polymer composite (original at 100X; magnification bar is 100 μm long). Specimen is as-polished and viewed with Nomarski DIC illumination.

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 5 LC and 10 LC 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.

Composites

Table 36. Four-Step Procedure for Metal-Matrix Composites

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
UltraPrep resin-bonded disc	30-µm diamond water cooled	5 (22)	240-300 Contra	Until Plane
UltraPol, or UltraPad cloths	9-µm MetaDi Supreme diamond suspension*	5 (22)	150-180 Contra	4:00
TriDent cloth	3-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Comp.	3:00
ChemoMet cloth	~0.05-µm MasterMet colloidal silica or MasterMet alumina suspension	6 (27)	120-150 Contra	2:00

*Plus MetaDi fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

Table 37. Four-Step Procedure for Polymer-Matrix Composites

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	320- (P400) grit SiC water cooled	4 (18)	150-240 Comp.	Until Plane
UltraPol, or UltraPad cloths	6-µm MetaDi Supreme diamond suspension*	4 (18)	120-150 Comp.	4:00
TexMet pad, TriDent or Nylon cloths	3-µm MetaDi Supreme diamond suspension*	5 (22)	120-150 Comp.	4:00
ChemoMet or MicroCloth cloths	~0.05-µm MasterMet colloidal silica or MasterPrep alumina suspension	6 (27)	120-150 Comp.	2:00-4:00

*Plus MetaDi Fluid extender as desired
Comp. = Complementary (platen and specimen holder both rotate in the same direction)

**Table 38. Four-Step Procedure for Ceramic-Matrix Composites**

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
UltraPrep metal-bonded disc	45-µm diamond MetaDi fluid for coolant	6 (27)	240-300 Contra	Until PLane
UltraPol cloth, UltraPad cloth, or TexMet pad	15-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Contra	4:00
UltraPol cloth, TriDent cloth, or TexMet pad	6-µm MetaDi Supreme diamond suspension*	6 (27)	120-150 Contra	3:00
UltraPol, ChemoMet or Nylon cloths	1-µm MetaDi Supreme diamond suspension and ~0.05-µm MasterMet colloidal silica (50:50 simultaneously)	6 (27)	120-150 Contra	2:00

*Plus MetaDi Fluid extender as desired
Contra = Platen and specimen holder rotate in opposite directions

PRINTED CIRCUIT BOARDS

The vast majority of printed circuit boards (PCB'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. 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 PCB's generally do not complicate the preparation methods due to the fact that extremely hard and brittle materials are not
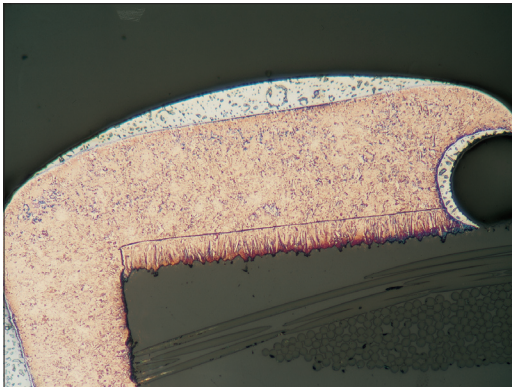
Micrograph showing a plated-through hole in a printed circuit board (resin and fibers) with copper foil, electroless-plated copper and Pb-Sn solder (equal parts ammonium hydroxide and 3% hydrogen peroxide, 200X).

commonly found in the boards. This changes, however, when 'populated' boards with ceramic or semiconductor components must be sectioned. Please refer to methods of preparation for ceramics or microelectronic materials for information on these cases. A generalized method for non populated PCB's of both the rigid and flex varieties is presented in Table 39.

Quality control of manufactured PCB'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, numerous specimens must be prepared. This is easiest when coupons are taken from PCB's and are ganged together in precise alignment. In this manner, the through holes in all of the coupons can be sectioned at one time, with one set of consumables. The Buehler NelsonZimmer System allows this type of automation with few adjustments to the method listed below.

HELPFUL HINTS FOR PRINTED CIRCUIT BOARDS

To improve penetration of acrylic mounting material into the through-holes of a PCB specimen, dip the "pinned" PCB 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.

Table 39. Five-Step Procedure for Non-Populated Printed Circuit Boards

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	180- (P180) grit SiC water cooled	5 (22)	120-150 Contra	Until Plane
CarbiMet 2 abrasive discs	320- (P400) grit SiC water cooled	5 (22)	120-150 Contra	1:30
CarbiMet 2 abrasive discs	600- (P1200) grit SiC water cooled	5 (22)	120-150 Contra	1:30
TexMet pad or, TriDent cloth	3-μm MetaDi II diamond paste*	5 (22)	150-240 Contra	1:30
MicroCloth cloth	0.05-μm MasterPrep alumina suspension	5 (22)	100-150 Contra	1:15

*Plus MetaDi Fluid extender as desired
Contra = Platen and specimen holder rotate in opposite directions

ELECTRONIC MATERIALS

The term,'microelectronic 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, we must focus on a few individual materials, and develop a philoso-

Aluminum circuitry on silicon adjacent to mineral filled epoxy packaging, (1000x).

phy of preparation in which we give our attention specifically to the materials of interest.

First and foremost in the class of 'microelectronic 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 40 illustrates a typical process of preparation.

Table 40. Three-Step Procedure for Preparing Silicon in Microelectronic Devices

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive abrasive disc (waterproof paper)	600 (P1200) grit SiC water cooled	Manual	100-150	Until Plane
TexMet pad	3-μm diamond MetaDi II diamond paste/ MasterPrep as lubricant	7 (31)	100-150 Contra	2:00
ChemoMet pad	0.02-μm MasterMet 2 colloidal silica	5 (22)	100-150 Contra	1:30

Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions
Note: The first step is manual so that you can monitor the progress of grinding relative to the feature of interest. The remaining steps are accomplished using a system that can apply individual force to the back of the spceimen. The first step can be automated if there is a significant amount of material to remove.

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 chemo-mechanical 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 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 section listing preparation methods for copper and nickel).

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, we can 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 40. (During the 3-μm polishing step, the best results are obtained by using diamond paste.
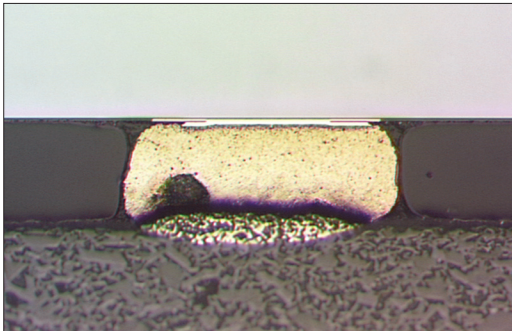
High temperature (96% Pb) solder under flip chip on ceramic substrate, (200x).

Table 41. Three-Step Procedure for High-Temperature Solder on Ceramic Devices

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive disc (waterproof paper)	600 (P1200) grit SiC water cooled	7 (31)	100-150 Comp.	Until Plane
TexMet pad	3-μm MetaDi II diamond paste/ MasterPrep as lubricant	7 (31)	100-150 Contra	Until all embedded 9-μm diamond is removed
ChemoMet pad	0.02-μm MasterMet 2 colloidal silica	5 (22)	100-150 Contra	2:00

*Apply sparingly
Comp. = Complementary(platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

Pastes contain a waxy base that reduces diamond 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.)
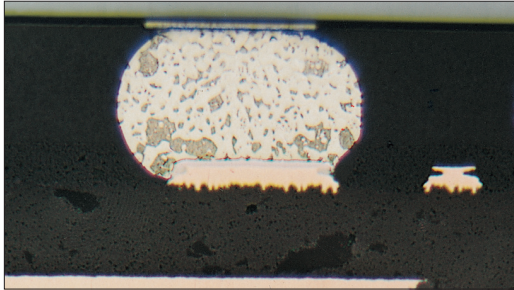
Silicon flip chip, solder balls, on PC board, (200x).

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 ApexHercules 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 pads, 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 41 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 chemo- mechanical 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.
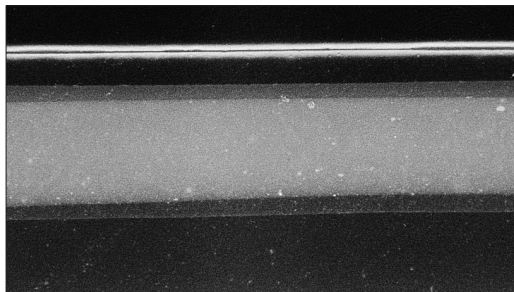
Micrograph showing multi-layered polymer meat casing mounted in epoxy (dark field, 100X).

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.

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.
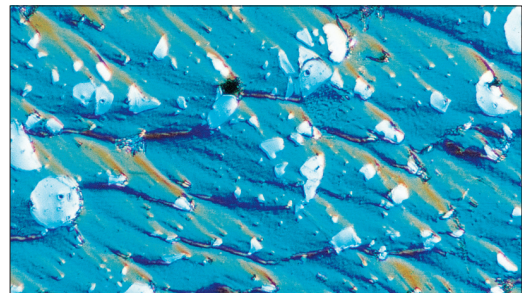
Micrograph of high-density polyethylene containing a filler (white particles) (unetched, Nomarski DIC, 100X).

Table 42. Generic Procedure for Preparing Plastic and Polymeric Specimens

Surface	Abrasive/ Size	Load Lb. (N)/ Specimen	Base Speed (rpm)/Direction	Time (min:sec)
CarbiMet 2 abrasive discs (waterproof paper)	320- (P400) grit SiC water cooled	4 (18)	200-250 Contra	Until Plane
CarbiMet 2 abrasive discs	400- (P600) grit SiC water cooled	4 (18)	200-250 Contra	0:30
CarbiMet 2 abrasive discs	600- (P1200) grit SiC water cooled	4 (18)	200-250 Contra	0:30
CarbiMet 2 MicroCut abrasive discs	800- (P1500) grit SiC water cooled	4 (18)	200-250 Contra	0:30
TexMet pad or TriDent cloth	3-μm MetaDi II diamond paste and MetaDi fluid	5 (22)	100-120 Comp.	4:00
MasterTex cloth	0.05-μm MasterPrep alumina suspension	3 (13)	100-120 Contra	4:00

Comp. = Complementary (platen and specimen holder both rotate in the same direction)
Contra = Platen and specimen holder rotate in opposite directions

Polymers

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 42 contains a generic procedure for preparing many plastic and polymeric materials. Depending upon the material and the surface roughness after sectioning, it may be possible to start grinding with 400- (P600) or even 600- (P1200) grit SiC paper.

If flatness is critical, the final step can be altered. Use a TexMet pad with MasterPrep alumina abrasive slurry at 10 lbs. pressure (44N) per specimen, 120 rpm, contra rotation, 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 30 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.
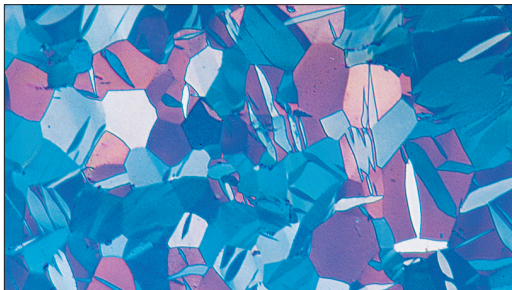

Figure 30. Mechanical twins at the surface of hot worked and cold drawn high-purity zirconium viewed with polarized light (200X).

Etching Procedures

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 0.2-0.3-μm 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. Table 43 lists 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
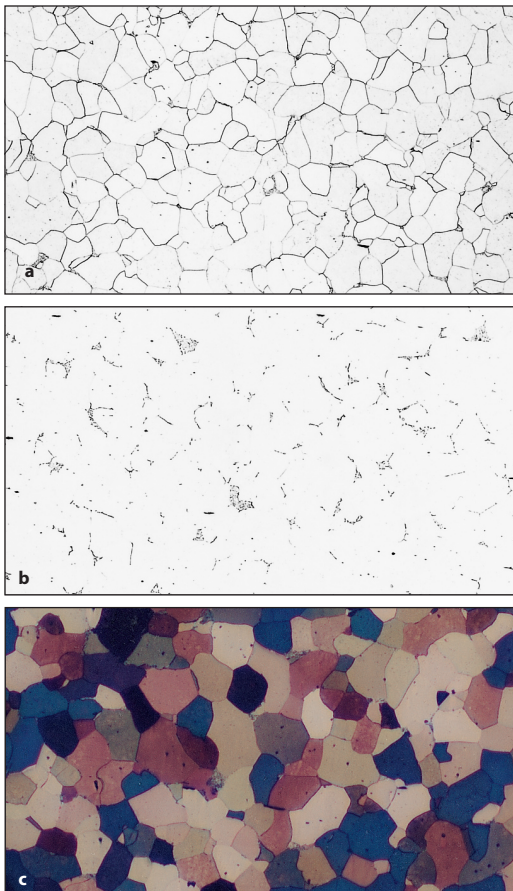

Figure 31. Microstructure of low-carbon sheet steel etched with (a, top) 2% nital, (b, middle) 4% picral; and (c, bottom) Beraha's reagent (100 mL water, 10 g $Na_2S_2O_3$ and 3 g $K_2S_2O_5$) at 100X.

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 31 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 32 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 32a, surrounded by pearlite. Etching with boiling alkaline sodium picrate, Figure 32b, colored the
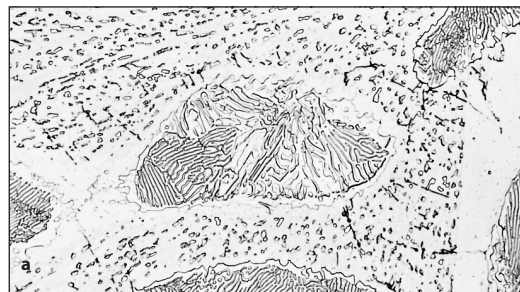

Figure 32a. The ternary eutectic (α-Fe_3C-Fe_3P) in gray cast iron revealed by etching (a, top) in picral and nital to "outline" the phases

cementite phase only, including in the surrounding pearlite (a higher magnification is required to see the very finely spaced cementite that is more lightly colored). Etching with boiling Murakami's reagent, Figure 32c, 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.
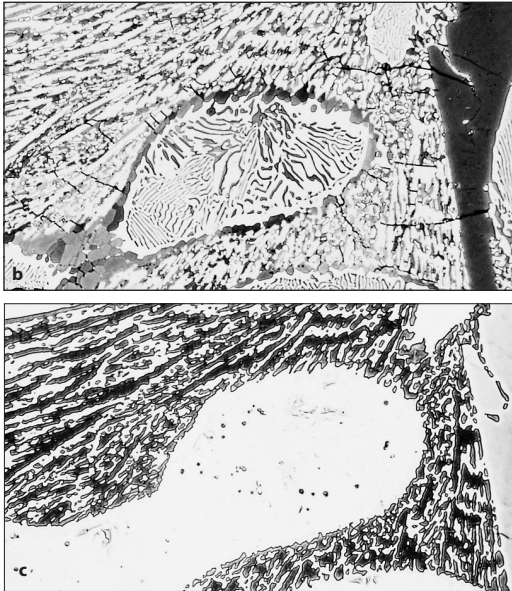

Figure 32b. (top) boiling alkaline sodium picrate to color the cementite. Figure 32c. (bottom) boiling Murakami's reagent to color the phosphide (200X).

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 33 shows 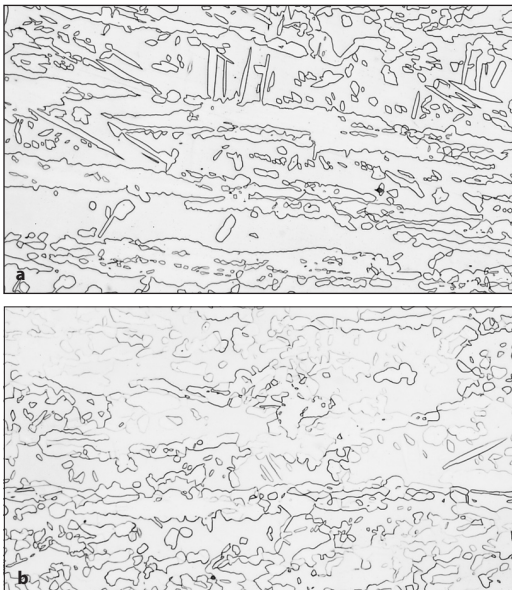examples of the use of a number of popular etchants to reveal the microstructure of a dual phase stainless steel in the hot rolled and annealed condition. Figure 33a shows a well delineated structure when the specimen was immersed in ethanolic 15% HCl for 30 minutes. All of the phase boundaries are clearly revealed, but there is no discrimination between ferrite and austenite. Twin boundaries in the austenite are not revealed. Glyceregia, a

Figure 33. Microstructure of a duplex stainless steel revealed using (a, top) alcoholic 15% HCl by immersion (30 min.); and, with (b, bottom) glyceregia by swabbing (2 min.) at 200X.
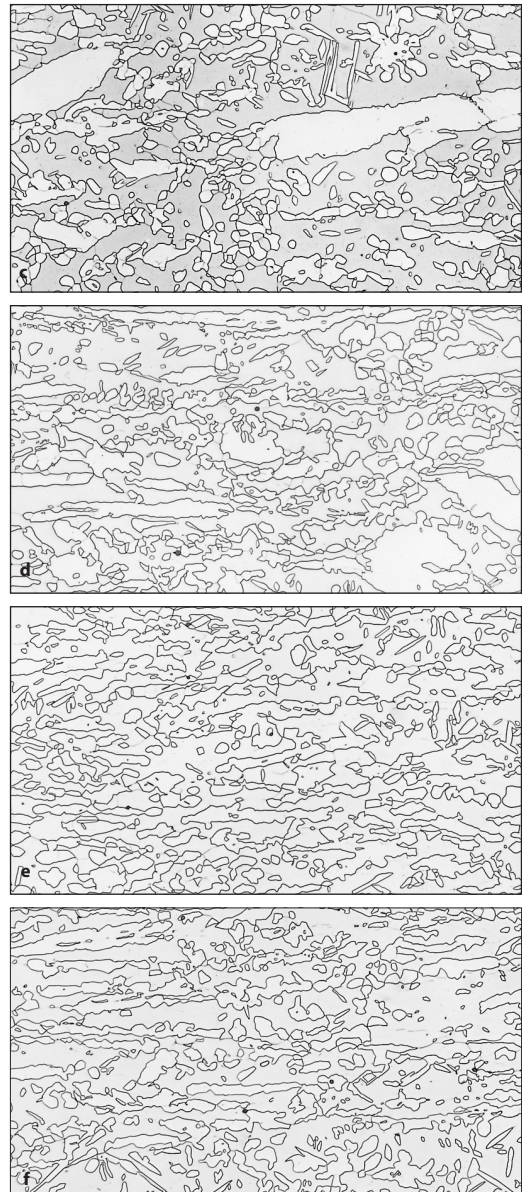

Figure 33. Microstructure of a duplex stainless steel revealed by electrolytic etching with (c) aqueous 60% HNO_3 (1 V dc, 20 s); with (d) aqueous 10% oxalic acid (6 V dc, 75 s); with (e) aqueous 10% CrO_3 (6 V dc, 30 s); and, with (f) aqueous 2% H_2SO_4 (5 V dc, 30 s) at 200X.

etchant for stainless steels, was not suitable for this grade, Figure 33b, as it appears to be rather orientation sensitive. Many electrolytic etchants have been used for etching stainless steels, but only a few have selective characteristics. Of the four shown in Figures 33c 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 33g 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 33g, 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 33i.
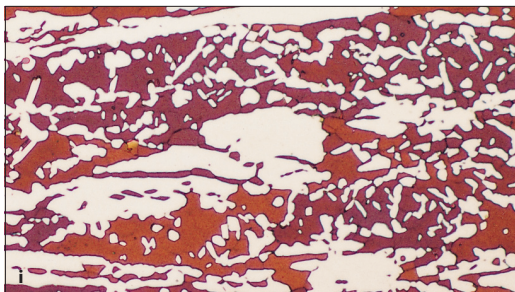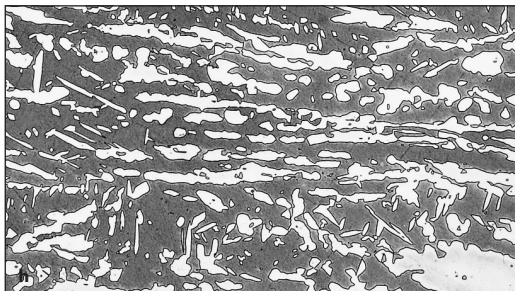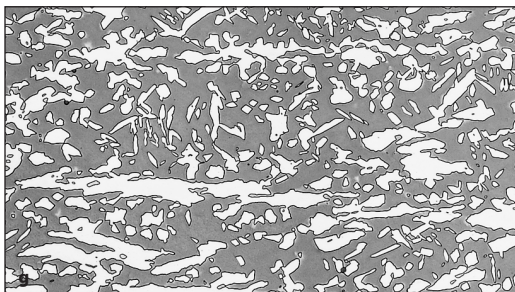

Figure 33. Selective coloring of ferrite in a duplex stainless steel by electrolytic etching with (g) aqueous 20% NaOH (4 V dc, 10 s) and with (h) aqueous 10 N KOH, (3 V dc, 4 s); and by immersion color etching (i) with Beraha's reagent (100 mL water, 10 mL HCl and 1 g $K_2S_2O_5$) at 200X.

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 34 illustrates coloring of beta phase in Naval Brass (UNS 46400) using Klemm's I reagent. Selective etching has a long historical record for identifica-
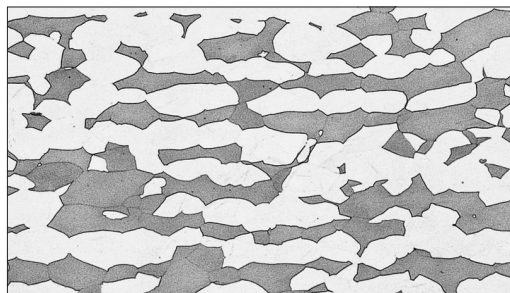
Figure 34. Beta phase colored in Naval Brass (Cu-39.7% Zn-0.8% Sn) by immersion color etching with Klemm's I reagent (200X).

tion of intermetallic 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 35 shows selective coloration of theta phase, $CuAl_2$, in the Al-33% Cu eutectic alloy. As a final example, Figure 36 illustrates the structure of a
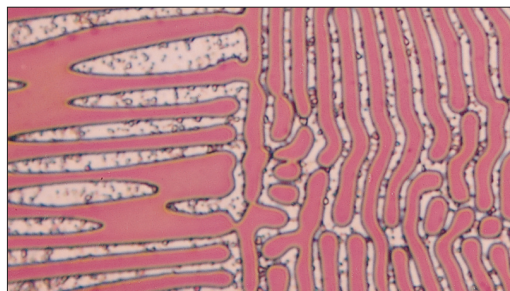
Figure 35. Theta phase, $CuAl_2$, colored in the α-Al/CuAl$_2$ eutectic in an as-cast Al-33% Cu specimen by immersion color etching with the Lienard and Pacque etch (200 mL water, 1 g ammonium molybdate, 6 g ammonium chloride) at 1000X.

simple WC-Co sintered carbide, cutting tool. In the as-polished condition, Figure 36a, the cobalt binder can be seen faintly against the more grayish tungsten carbide grains. A few particles of graphite are visible. In Figure 36b, 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 36c, attacks the cobalt and provides good uniform contrast for measurement of the cobalt binder phase. Follow ing 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 36d.
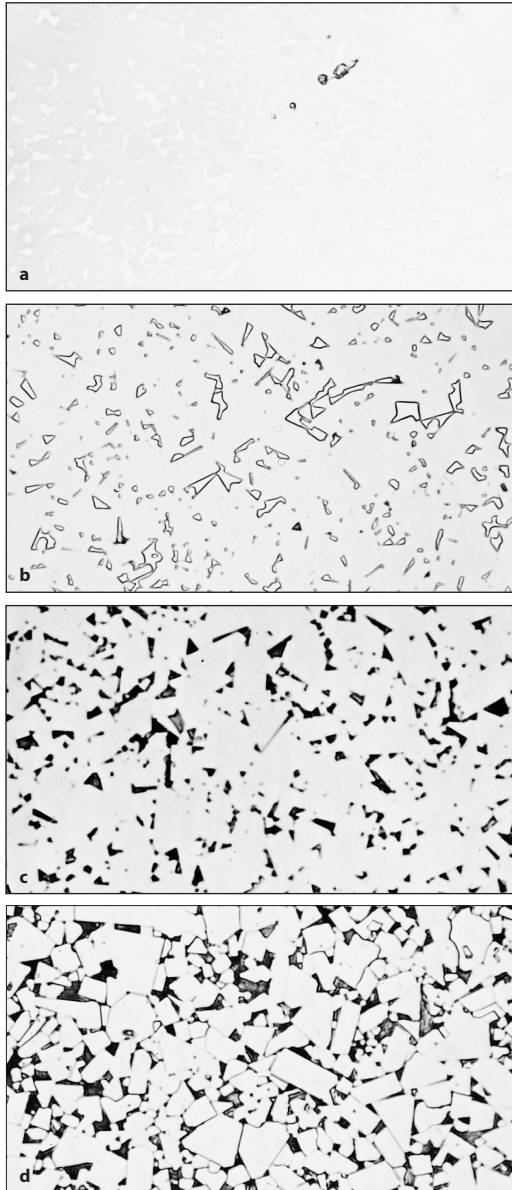

Figure 36. Microstructure of WC-Co cutting tool: a) as-polished revealing graphite particles; b) relief polished revealing the cobalt binder phase; c) after immersion in Chaporova's etch (HCl saturated with FeCl₃) to attack and "darken" the cobalt; and, d) after (c) plus Murakami's reagent to outline the WC grains (1000X).

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 33, 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 37 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.
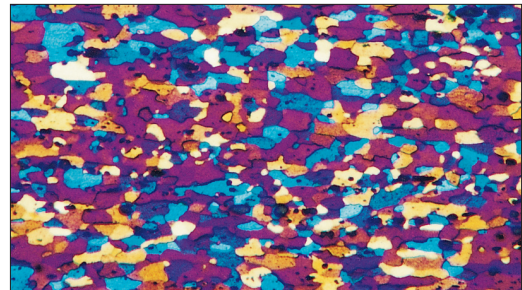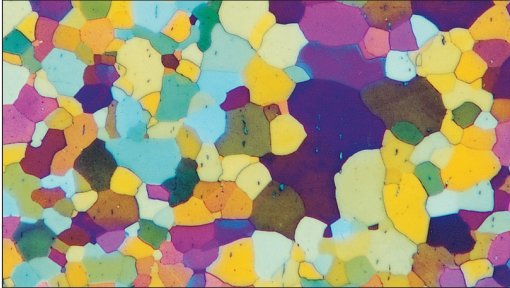

Figure 37. Grain structure of annealed 5754 aluminum sheet revealed by anodizing with Barker's reagent (30 V dc, 2 min.) and viewing with polarized light plus sensitive tint (100X).

Heat Tinting

Although not commonly utilized, heat tinting [2] is an excellent method for obtaining color contrast between constituents or grains. An 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 30-500 nm. 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 38 shows the grain structure of CP titanium revealed by heat tinting.

Figure 38. Grain structure of annealed CP titanium revealed by heat tinting on a hot plate (100X, polarized light plus sensitive tint).

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.

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.

Table 43. Commonly Used Etchants for Metals and Alloys[3].

LIGHT METALS - Aluminum and Alloys

Composition	Comments
1. 95 mL water 2.5 mL HNO_3 1.5 mL HCl 1.0 mL 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. 90-100 mL water 0.1-10 mL HF	General-purpose reagent. Attacks $FeAl_3$, other constituents outlined. The 0.5% concentration of HF is very popular.
3. 84 mL water 15.5 mL HNO_3 0.5 mL HF 3 g CrO_3	Graff and Sargent's etchant, for grain size 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/in^2, 30-45 V dc. For most alloys and tempers, 20 seconds at 1 A/in^2 and 30 V dc at 20 °C is sufficient. Stirring not needed. Rinse in warm water, dry. Use polarized light; sensitive tint helpful.

Magnesium and Alloys

Composition	Comments
5. 25 mL water 75 mL 3-5 ethylene glycol 1 mL 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.
6. 19 mL water 60 mL ethylene glycol 20 mL acetic acid 1 mL 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.
7. 100 mL ethanol 10 mL water 5 g picric acid	For Mg and alloys. Use fresh. Immerse specimen for 15-30 seconds. Produces grain contrast.

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

Composition	Comments
8. 100 mL water 30 mL HCl 2 g $FeCl_3$	For Sb, Bi and alloys. Immerse specimen up to a few minutes.
9. 100 mL water 25 mL HCl to a 8 g $FeCl_3$	For Sb-Pb, Bi-Sn, Cd-Sn, Cd-Zn, and Bi-Cd alloys. Immerse specimen up few minutes.
10. 95-99 mL ethanol 1-5 mL 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 a stain forms, wash in 10% alcoholic HCl.
11. 100 mL water 10 g ammonium molybdate 10 g citric acid	Pollack's reagent for Pb and alloys. Immerse specimen 15-30 seconds. Other compositions used are: 100 mL: 9 g: 15 g and 100 mL: 10 g: 25 g.
12. 100 mL water 2 mL HCl 10 g $FeCl_3$	For Sn-based babbitt metal. Immerse specimen up to 5 minutes.
13. 200 mL water 40 g CrO_3 3 g Na_2SO_4	Palmerton reagent for pure Zn and alloys. Immerse specimen up to 3 minutes. Rinse in 20% aqueous CrO_3.
14. 200 mL water 10 g CrO_3 1 g Na_2SO_4	Modified Palmerton reagent for Zn die-casting alloys. Immerse specimen for several seconds, rinse in 20% aqueous CrO_3.

[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.

Table 43. Commonly Used Etchants for Metals and Alloys[3].

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

Composition	Comments
15. 100 mL water 1-3 mL HF 2-6 mL HNO$_3$	Kroll's reagent for Ti alloys. Swab specimen 3-10 seconds or immerse specimen 10-30 seconds.
16. 200 mL water 1 mL HF	For Ti, Zr and alloys. Swab or immerse specimen. Higher concentrations can be used but are prone to staining problems.
17. 30 mL lactic acid 15 mL HNO$_3$ 30 mL HF	For Ti alloys. Swab specimen up to 30 seconds. Decomposes, do not store. Good for alpha-beta alloys.
18. 30 mL HCl 15 mL HNO$_3$ 30 mL HF	For Zr, Hf, and alloys. Swab specimen 3-10 seconds, or immerse specimen up to 120 seconds.
19. 45 mL H$_2$O (H$_2$O$_2$ or glycerol) 45 mL HNO$_3$ 8-10 mL 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.
20. 60 mL HCl 20 mL 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.
21. 30 mL HCl 45 mL glycerol 15 mL HNO$_3$	Modified "Glyceregia". For Cr and alloys. Immerse specimen up to a few minutes.
22. 100 mL water 10 g KOH or NaOH 10 g K$_3$Fe(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.
23. 70 mL water 20 mL H$_2$O$_2$ (30%) 10 mL H$_2$SO$_4$	For Mo alloys. Immerse specimen 2 minutes. Wash with water and dry; immersion produces colors, swabbing produces grain-boundary etch.
24. 10-20 mL glycerol 10 mL HNO$_3$ 10 mL HF	For Mo and Mo-Ti alloys. Immerse specimen for up to 5 minutes.
25. 100 mL water 5 g K$_3$Fe(CN)$_6$ 2 g KOH	For Mo-Re alloys. Use at 20 °C by immersion.
26. 50 mL acetic acid 20 mL HNO$_3$ 5 mL HF	For Nb, Ta and alloys. Swab specimen 10-30 seconds.
27. 50 mL water 14 mL H$_2$SO$_4$ 5 mL HNO$_3$	DuPont Nb reagent. For Nb-Hf and Nb alloys.
28. 50 mL water 50 mL HNO$_3$ 1 mL HF	For Nb-Zr and Nb-Zr-Re alloys. Swab specimen.
29. 30 mL lactic acid 10 mL HNO$_3$ 5 mL HF	For Re and W-Re alloys. Swab specimen.
30. 10 mL HF 10 mL HNO$_3$ 10-30 mL glycerol	For V and alloys; grain-boundary etch for Ta alloys. Swab specimen. Equal parts used for Ta and high Ta alloys.

IRON and STEEL

Composition	Comments
31. 90-99 mL methanol or ethanol 1-10 mL 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 60 seconds.

Table 43. Commonly Used Etchants for Metals and Alloys[3].

Composition	Comments
32. 100 mL ethanol 4 g picric acid	Picral. Recommended for structures consisting of ferrite and carbide. Does not reveal ferrite grain boundaries. Addition of about 0.5-1% zephiran chloride improves etch rate and uniformity.
33. 100 mL ethanol 5 mL HCl 1 g picric acid	Vilella's reagent. Good for ferrite-carbide structures. Produces grain contrast for estimating prior austenite grain size. Results best on martensite tempered at 572-932 °F (300-500 °C). Occasionally reveals prior-austenite grain boundaries in high alloy steels. Outlines constituents in stainless steels. Good for tool steels and martensitic stainless steels.
34. 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 and bainitic steels. Many wetting agents have been used, sodium tridecylbenzene sulfonate is one of most successful (the dodecyl version is easier to obtain and works as well). Use at 20-100 °C. Swab or immerse sample for 2-60 minutes. Etch in ultrasonic cleaner (see ref.2, pg. 219-223). Additions of 0.5g $CuCl_2$ 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.
35. 150 mL water 50 mL HCl 25 mL HNO$_3$ 1 g CuCl$_2$	Modified Fry's reagent. Used for 18% Ni maraging steels, martensitic and PH stainless steels.
36. 100 mL water 25 g NaOH 2 g 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.
37. 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.
38. 100 mL ethanol 100 mL HCl 5 g 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 20 °C by immersion or swabbing. Can be stored.
39. 15 mL HCl 10 mL acetic acid 5 mL HNO$_3$ 2 drops glycerol	Acetic glyceregia. Mix fresh; do not store. Use for high alloy stainless steels.
40. 100 mL water 10 g K$_2$Fe(CN)$_6$ 10 g KOH or NaOH	Murakami's reagent. Usually works better on ferritic stainless grades than on austenitic grades. Use at 20 °C for 7-60 seconds: reveals carbides si sigma faintly attacked with etching up to 3 minutes. Use at 80°C (176°F) 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.
41. 100 mL water 10 g oxalic acid	Use for stainless steels at 6 V dc. Carbides revealed 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.
42. 100 mL water 20 g NaOH	Used to color ferrite in martensitic, PH or dual-phase stainless steels. Use at 3-5 V dc, 20 °C, 5 seconds, stainless steel cathode. Ferrite outlined and colored tan.
43. 40 mL water 60 mL 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 1.4 V dc, 2 minutes (see ref. 2, pgs. 235, 238 and 239).

COPPER, NICKEL and COBALT: Copper and Alloys

Composition	Comments
44. 25 mL NH$_4$OH 25 mL water (optional) 25-50 mL H$_2$O$_2$ (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.
45. 100 mL water 10 g 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.

Table 43. Commonly Used Etchants for Metals and Alloys[3].

Composition	Comments
46. 100 mL water 3 g ammonium persulfate 1 mL NH$_4$OH	General purpose etch for Cu and alloys, particularly Cu-Be alloys.
47. 70 mL water 5 g Fe(NO$_3$)$_3$ 25 mL HCl	Excellent general purpose etch, reveals grain boundaries well. Immerse specimen 10-30 seconds.

Nickel and Alloys

Composition	Comments
48. 5 g FeCl$_3$ 2 mL HCl 99 mL ethanol	Carapella's etch for Ni and Ni-Cu (Monel) alloys. Use by immersion or swabbing.
49. 40-80 mL ethanol 40 mL HCl 2 g 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.
50. 50 mL water 50 mL HCl 10 g 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.
51. 15 mL HCl 10 mL glycerol 5 mL HNO$_3$	"Glyceregia", for superalloys and Ni-Cr alloys. Swab specimen for 5-60 seconds. Mix fresh. Do not store. Use under a hood.
52. 60 mL glycerol 50 mL HCl 10 mL 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 specimen 10-60 seconds.

Cobalt and Alloys

Composition	Comments
53. 60 mL HCl 15 mL water 15 mL acetic acid 15 mL HNO$_3$	For Co and alloys. Mix fresh and age 1 hour before use. Immerse spe specimen for up to 30 seconds. Do not store.
54. 200 mL ethanol 7.5 mL HF 2.5 mL HNO$_3$	General etch for Co and alloys. Immerse specimen 2-4 minutes.
55. 50 mL water 50 mL HCl 10 g CuSO$_4$	Marble's reagent, for Co high temperature alloys. Immerse or swab spe specimen for up to 1 minute.
56. 80 mL lactic acid 10 mL H$_2$O$_2$ (30%) 10 mL HNO$_3$	For Co alloys. Use by swabbing.

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

Composition	Comments
57. 60 mL HCl 40 mL 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.
58. 60 mL HCl 20 mL HNO$_3$	Aqua regia for pure gold, Pt and alloys, some Rh alloys. Use boiling for up to 30 minutes.
59. 1-5 g CrO$_3$ 100 mL HCl	For Au, Ag, Pd and alloys. Swab or immerse specimen up to 60 seconds.
60. 30 mL water 25 mL HCl 5 mL HNO$_3$	For pure Pt. Use hot, immerse specimen up to 5 minutes.
61. Conc. HCl	For Rh and alloys. Use at 5 V ac, 1-2 minutes, graphite cathode, Pt lead wires.

Table 43. Commonly Used Etchants for Metals and Alloys[3].

62.	Solution a
	  100 mL water
	    40 g 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.

63.	50 mL NH$_4$OH 20 mL 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.

64.	Solution a
	  10 g NaCN
	  100 mL 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 the desired etch level is obtained. If a brown stain forms, swab with a to remove it.

SINTERED CARBIDES

	Composition	Comments
65.	100 mL water 10 g KOH or NaOH 10 g 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.
66.	97 mL water 3 mL 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.
67.	15 mL water 30 mL HCl 15 mL HNO$_3$ 15 mL acetic acid	For WC, TiC, TaC, and Co in sintered carbides. Use at 20 °C for 5-30 seconds.
68.	100 mL H$_2$O 3 g FeCl$_3$	To darken Co (or Ni) binder phase. Mix fresh. Swab 10 seconds.

CERAMICS and NITRIDES

	Composition	Comments
69.	Phosphoric acid	For alumina, Al$_2$O$_3$, and silicon nitride, Si$_3$N$_4$. Use by immersion at 250 °C for up to a few minutes for Al$_2$O$_3$ or up to 15 minutes for Si$_3$N$_4$.
70.	100 mL water 15 mL HNO$_3$	For magnesia, MgO. Use by immersion at 25-60 °C for several minutes.
71.	100 mL water 5 g NH$_4$ FHF 4 mL HCl	For titania, TiO$_2$. Immerse up to a few minutes.
72.	50 mL water 50 mL H$_2$SO$_4$	For zirconia, ZrO$_2$. Use by immersion in boiling solution for up to 5 minutes.
73.	HCl	For calcia, CaO, or MgO. Immerse for up to 6 minutes.
74.	HF	For Si$_3$N$_4$, BeO, BaO, MgO, ZrO$_2$ and Zr$_2$O$_3$. Immerse for up to 6 minutes.

PLASTICS and POLYMERS

	Composition	Comments
75.	100 mL water 60 g CrO$_3$	For polypropylene (PP). Immerse for up to several hours at 70°C.
76.	HNO$_3$	For polyethylene (PE). Immerse for up to a few minutes.
77.	Xylol 70 °C.	Reveals spherolites in polyethene (PE). Immerse for up to a few days at
78.	70 mL water 30 mL HCl	For polyoxymethylene (POM). Immerse up to 20 seconds.
79.	Xylene	For polyamid (PA) and polyethylene (PE). Use at 70 °C for 60 seconds. For Nylon 6, use at 65-70 °C for 2-3 minutes. For Nylon 6, use at 75 °C for 3-4 minutes.

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 39 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.
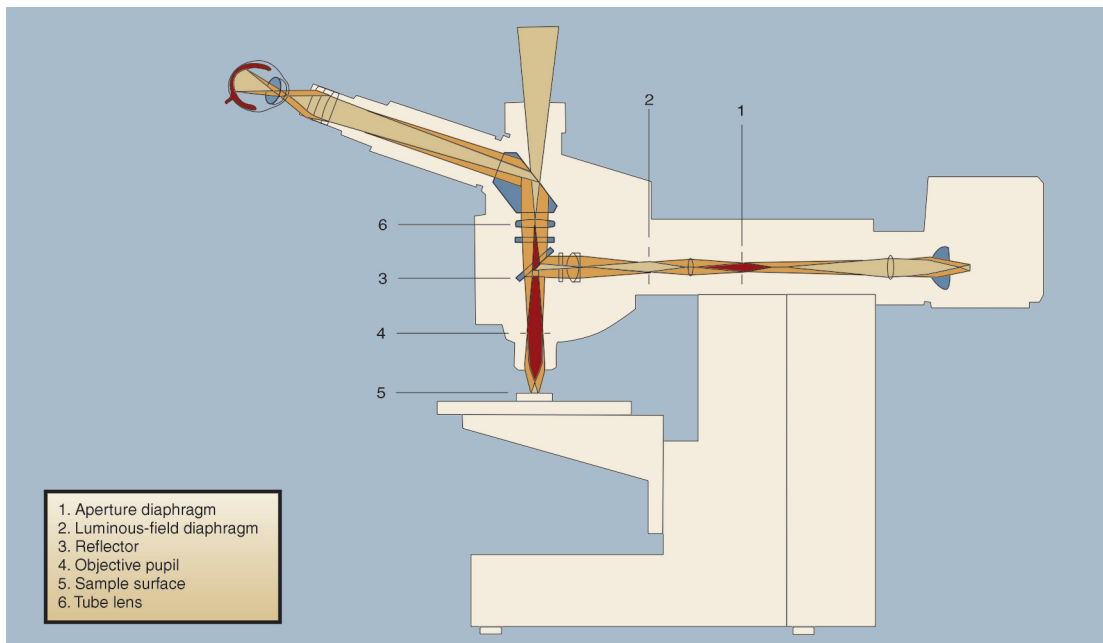

1. Aperture diaphragm
2. Luminous-field diaphragm
3. Reflector
4. Objective pupil
5. Sample surface
6. Tube lens

Figure 39. Schematic diagram showing the light path through an upright reflected light microscope operating with bright field illumination.

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 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.

Semiapochromatic 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 250 mm (10-inch) from the eye. The eyepiece magnifies this image, permitting achievement of useful magnifications. The standard eyepiece has a 24 mm diameter field of view; wide field eyepieces for plano-type objectives have a 30 mm 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 10 mm 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 20 mm, 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 automated to produce a direct digital readout of the measurement, which is accurate to approximately 1-μm.

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. 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 40 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 0.2-μm. To see lines or points spaced 0.2-μm apart, the required magnification is determined by dividing by the resolving power of the human eye, which is difficult to determine under observation conditions. Abbe used a value of 0.3 mm at a distance of 250 mm – the distance from the eye for optimum vision. This gives 1500x. For light with a mean wavelength of 0.55-μm, 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 550 nm, the lowest magnification that takes full advantage of the NA of the objective is 500 times the NA.
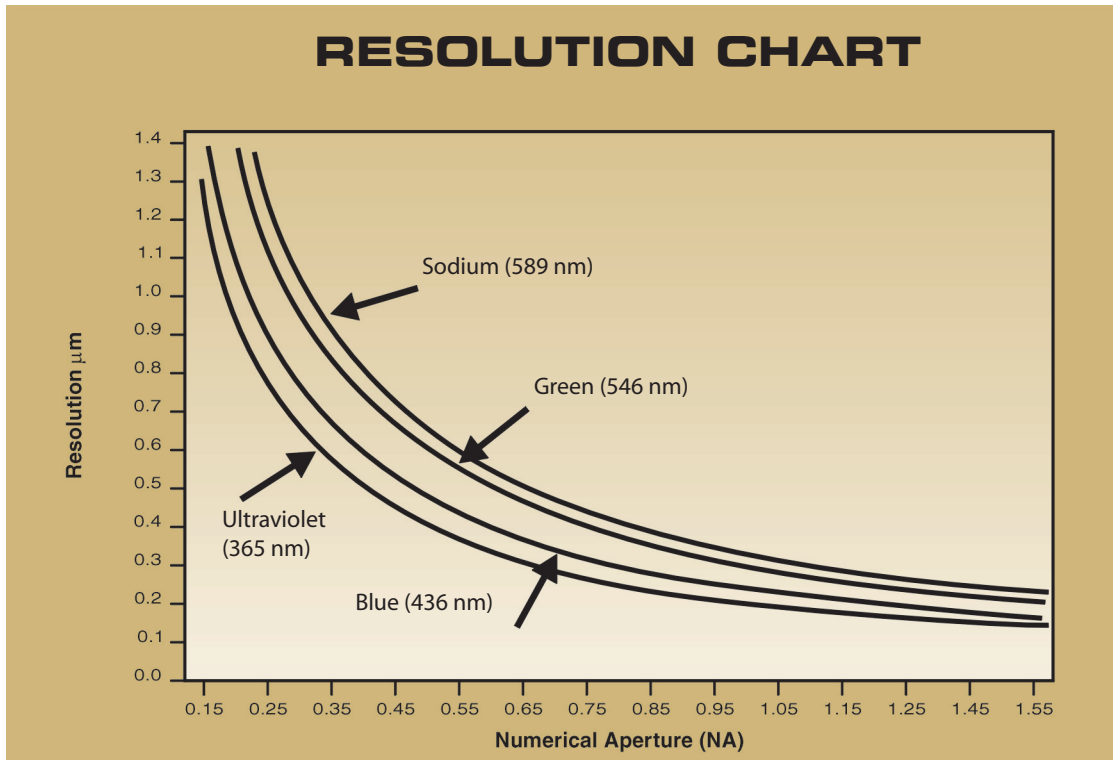
RESOLUTION CHART

Figure 40. Influence of objective numerical aperture and light wavelength on the resolution of the light microscope.

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:

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

where n is the refractive index of the medium between the specimen and the objective ($n \sim 1.0$ 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 41.

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
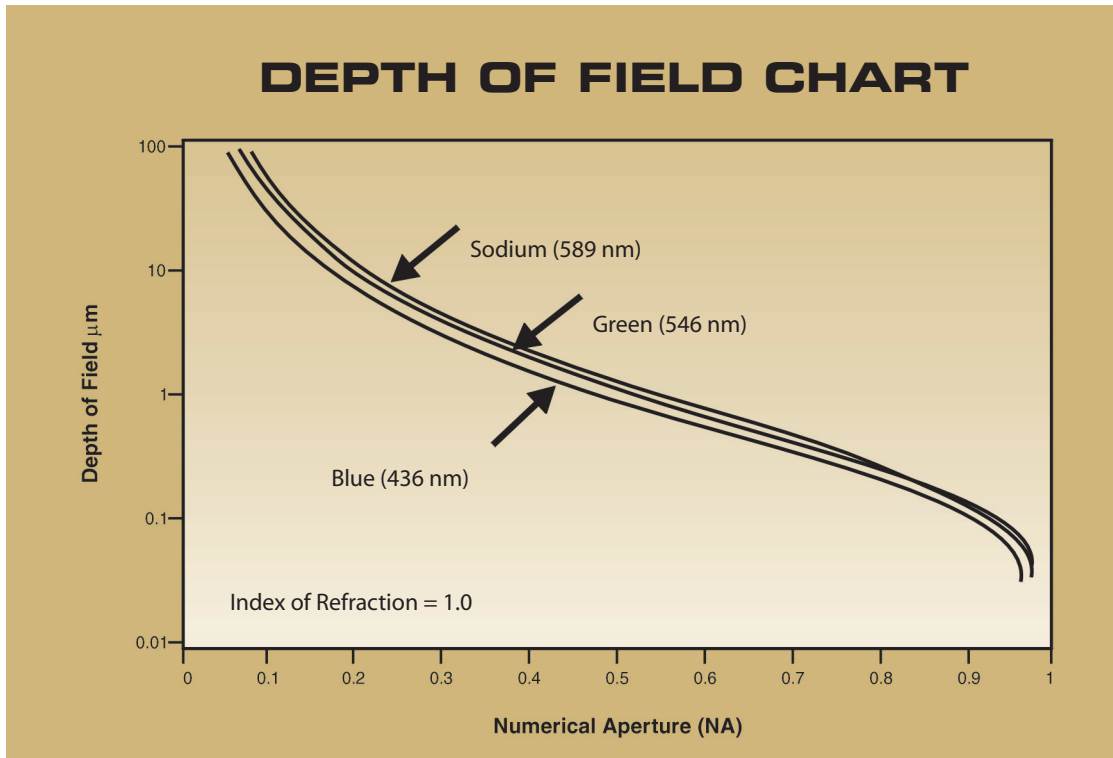
DEPTH OF FIELD CHART

Sodium (589 nm)

Green (546 nm)

Blue (436 nm)

Index of Refraction = 1.0

Depth of Field μm

Numerical Aperture (NA)

Figure 41. Influence of the objective numerical aperture and light wavelength on the depth of field of the light microscope.

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 42. 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

1. Light source
2. Color-neutral beam splitter
3. Reflected-light objective
4. Sample surface
5. Tube lens
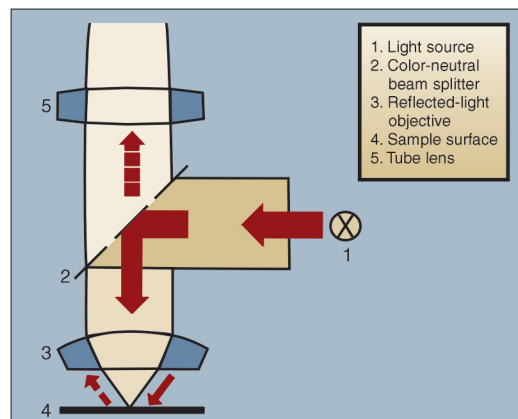
Figure 42. Schematic diagram showing the light path in bright field illumination.

this happens because only a portion of the lens is used. For this reason, there is a practical limit to the amount of contrast that can be achieved. Illumination also becomes uneven as the degree of "obliqueness" increases. Since differential interference contrast systems have been available, oblique illumination is rarely offered as an option on new microscopes.

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 43 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 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.
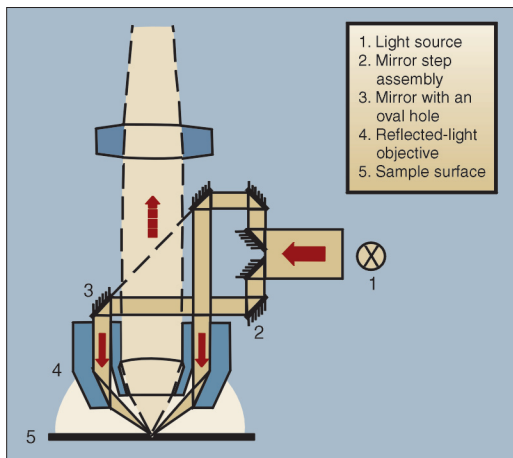
Figure 43. Schematic diagram showing the light path in dark field illumination.

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 44. 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.
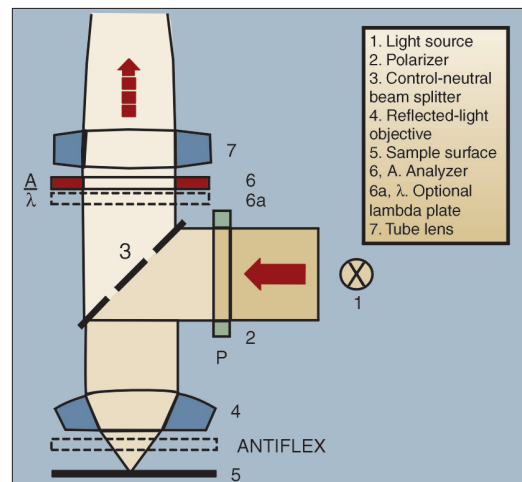

Figure 44. Schematic diagram showing the light path in polarized light with an optional lambda plate (sensitive tint plate).

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 45, two light beams are produced which exhibit coherent interference in the image plane. This leads to two slightly displaced (laterally) images differing in phase (λ/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 46 shows the micro- this happens because only a structure of an aluminum bronze
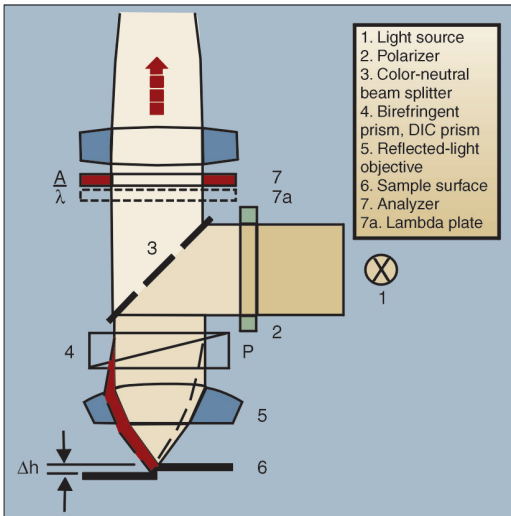
1. Light source
2. Polarizer
3. Color-neutral beam splitter
4. Birefringent prism, DIC prism
5. Reflected-light objective
6. Sample surface
7. Analyzer
7a. Lambda plate

Figure 45. Schematic diagram showing the light path for differential interference contrast (DIC) illumination.

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 46a shows the surface in bright field illumination. Due to the relief present, a faint image of the structure is visible. Dark field, Figure 46c, and differential interference contrast, Figure 46d, make use of this relief to reveal much more detail than in bright field.
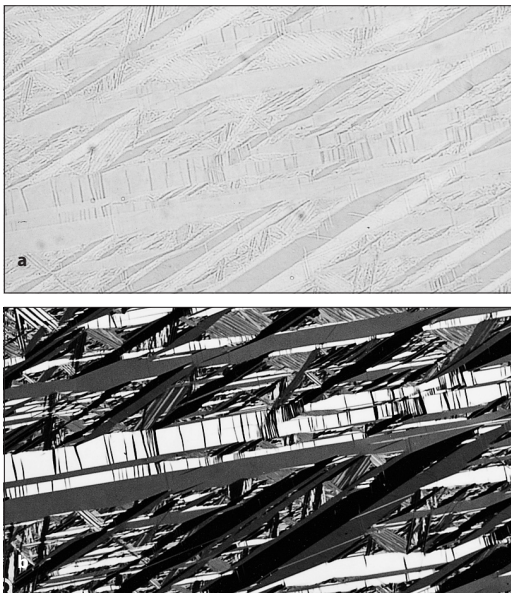
Figure 46. Martensitic microstructure of heat treated, eutectoid aluminum bronze (Cu–11.8% Al) in the unetched condition viewed using: a) bright field illumination, b) polarized light

However, as the martensite is noncubic in crystal structure, it responds well to crossed- polarized light, Figure 46b. 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.
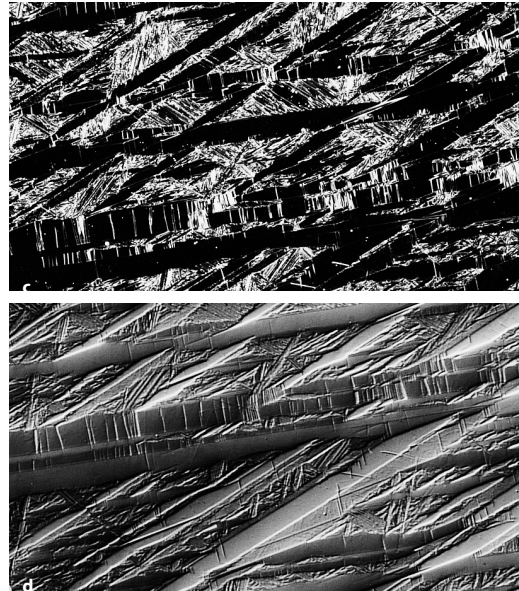
Figure 46. Martensitic microstructure of heat treated, eutectoid aluminum bronze (Cu–11.8% Al) in the unetched condition viewed using: c) dark field illumination; and, d) differential interference contrast illumination (200X).

HELPFUL HINTS FOR LIGHT 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.

MICROINDENTATION HARDNESS TESTING

Microindentation hardness testing, more commonly (but incorrectly) called microhardness testing, is widely used to study fine scale changes in hardness, either intentional or accidental. Heat treaters have utilized the technique for many years to evaluate the success of surface hardening treatments or to detect and assess decarburization. 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.

Metallurgists and metallographers tend to develop their own jargon, often as a matter of linguistic simplicity, which is not always as rigorously correct as it could be. 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. ASTM Standard E 384 fully describes the two most common microindentation tests - the Vickers and the Knoop tests.

The Vickers Test

In 1925, Smith and Sandland of the UK developed a new indentation test for metals that were too hard to evaluate using the Brinell test. The hardened steel ball of the Brinell test limited the test to steels with hardnesses below ~450 HBS (~48 HRC). (The harder tungsten carbide ball was not available in 1925. The WC indenter extends the Brinell test to metals up to 615 HBW (~58 HRC). The WC ball has now replaced the steel ball for the Brinell test.) In designing the new indenter, a square-based diamond pyramid (see Figure 47), 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.
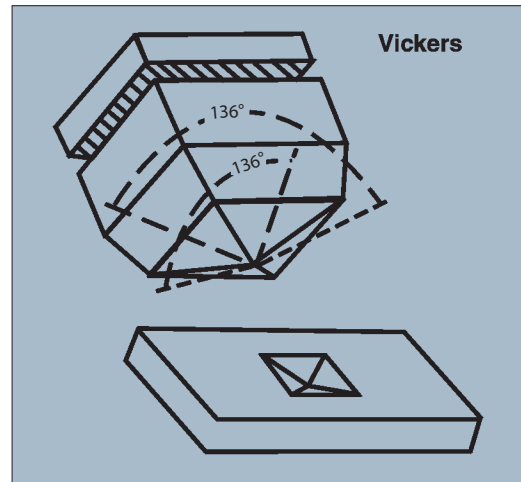

Figure 47. Schematic of the Vickers indenter and the shape of an impression.

Use of diamond allowed the Vickers test to be used to evaluate any material (except diamond) and, furthermore, had the very important advantage of placing the hardness of all materials on one continuous scale. This is a major disadvantage of Rockwell type tests where different scales (15 standard and 15 superficial) were developed to evaluate materials. Not one of these scales can cover the full hardness range. The HRA scale covers the broadest hardness range, but it is not commonly used.

In the Vickers test, the load is applied smoothly, without impact, forcing the indenter into the test piece. The indenter is held in place for 10 or 15 seconds. The physical quality of the indenter and the accuracy of the applied load (defined in E 384) must be controlled in order to get the correct results. After the load is removed, the two impression diagonals are measured, usually to the nearest 0.1-μm with a filar micrometer, 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/μm^2 although the equivalent units kgf/mm^2 are preferred; in practice the numbers are reported without indication of the units). The original Vickers testers were developed for test loads of 1 to 120kgf that produce 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.

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. Numerous studies of microindentation hardness test results conducted over a wide range of test loads have shown that test 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 [2] and an ASTM interlaboratory "round robin" of indents made by one laboratory but measured by twelve different people, reported all three possible ISE responses [24,25] for the same indents!

Since the 1960s, the standard symbol for Vickers hardness per ASTM E 92 and E 384, has been HV. This should be used in preference to the older, obsolete symbols DPN or VPN. The hardness is expressed in a standard format. For example, if a 300 gf load is used and the test reveals a hardness of 375 HV, the hardness is expressed as 375 HV$_{300}$. Rigorous application of the SI system results in hardness units expressed not in the standard, understandable kgf/mm^2 values but in GPa units that are meaningless to most engineers and technicians. ASTM recommends a 'soft' metric approach in this case.

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 48) appears to be square, and 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 49, 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 Knoop Test

As an alternative to the Vickers test, particularly to test very thin layers, Frederick Knoop and his associates at the former National Bureau of Standards developed a low-load test using a rhombohedral-shaped diamond indenter, Figure 50.
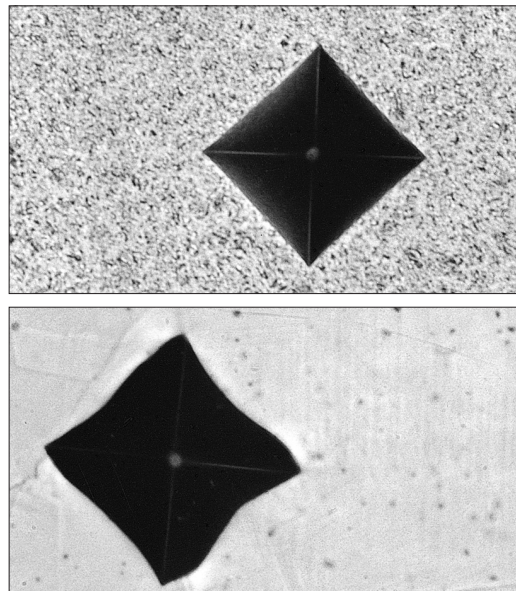

Figure 48. (top) Example of a well-formed Vickers indentation (400X). Figure 49. (bottom) Example of a distorted Vickers indentation (400X).

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. Some investigators claim there is no elastic recovery with the Knoop indent, but this cannot be true as measurements of the ratio of long to short diagonal often reveal results substantially different than the ideal 7.114 value.
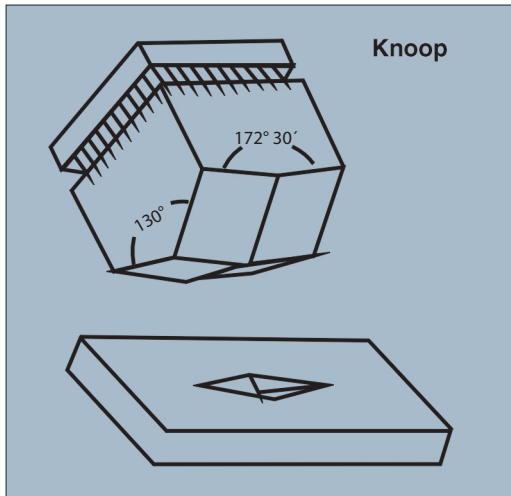
Figure 50. Schematic of the Knoop indenter and the shape of an impression

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 some time. The Knoop hardness is calculated from

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

where the load L is in gf and the long diagonal d is in μm. 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. The Knoop hardness is expressed in the same manner as the Vickers hardness; i.e., 375 HK_{300} means that a 300 gf load produced a Knoop hardness of 375. (The kgf/mm^2 unit information is no longer reported).

Aside from a minor savings of time, one chief merit of the Knoop test is the ability to test thin layers more easily. For surfaces with varying hardness, such as case hardened parts, Knoop indents can be spaced closer together than Vickers indents. Thus, a single Knoop traverse can define a hardness gradient more simply than a series of two or three parallel Vickers traverses where each indent is made at different depths. Furthermore, if the hardness varies strongly with the depth, the Vickers indent will be distorted by this change; that is, the diagonal parallel to the hardness change will be affected by the hardness gradient (i.e., there is a substantial difference in the lengths of the two halves of the diagonal), while the diagonal perpendicular to the hardness gradient will be

unaffected (both halves of this diagonal of the same approximate length).

The down side of the Knoop indent is that the three dimensional indent shape will change with test load and, consequently, HK varies with load. At high 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.

Factors Affecting Accuracy, Precision and Bias

Many factors (see Table 44) can influence the quality of microindentation test results [26]. In the early days of low-load (< 100gf) hardness testing, it was quickly recognized that improper specimen preparation may influence hardness test results. Most text books state that improper preparation will yield higher test results because the surface contains excessive preparation induced deformation. While this is certainly true, there are other cases where improper preparation can create excessive heat that will lower the hardness and strength of many metals and alloys. So either problem may be encountered due to faulty preparation. 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 [3], has led to development of excellent mechanical specimen preparation procedures, and electropolishing is no longer required.

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 measure the face angles and the sharpness of the tip. Specifications for Vickers and Knoop indenter geometries are given in E 384.

Table 44. 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	Numerical aperture of the objective	Strength of crystallographic texture, if present
Lateral movement of the indenter or specimen	Magnification	Quality of specimen preparation
Indentation time	Inadequate image quality	Low reflectivity or transparency
Indenter shap 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 indenter or specimen
Angle of indentation	Focusing of the image	

A prime source of error in the tests is the alignment of the specimen surface relative to the indenter. The indenter itself must be properly aligned perpendicular (±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 more than 1° from perpendicular results in nonsymmetrical impressions and can produce lateral movement between specimen and indenter. The occurrence of non- symmetrical indents is generally easily detected during measurement.

In most cases, errors in indenting with modern testers are not the major source of error, although this can occur [26]. It is important to check the performance of your tester regularly using a certified test block. It is safest to use a test block manufactured for microindentation testing and certified for the test (Vickers or Knoop) and the load that you intend to use. Strictly speaking, a block certified for Vickers testing at 300 or 500 gf (commonly chosen loads) should yield essentially the same hardness with loads from about 50 to 1000 gf. That is, if you take the average of about five indents and compare the average at your load to the average at the calibrated load (knowing the standard deviation of the test results), statistical tests can tell you (at any desired confidence level) if the difference between the mean values of the tests at the two loads is statistically significant or not.

Because of the method of defining HV and HK (equations given above) where we divide by d^2, measurement errors become more critical as d get smaller; that is, as L decreases and the material's hardness increases (discussed later). So departure from a constant hardness for the Vickers or Knoop tests as a function of load will be a greater problem as the hardness increases. For the Knoop test, HK increases as L decreases because the indent geometry changes with indent depth and width. The degree of the change in HK also varies with test load being greater as L decreases.

The greatest source of error is in measuring the indent as has been documented in an ASTM interlaboratory test [24,25]. 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 provide maximum contrast and resolution. The accuracy of the filar micrometer, or other measuring device, should be verified using a stage micrometer.

Specimen preparation quality becomes more important as the load decreases, and it must be at an acceptable level. Specimen thickness must be at least 2.5 times the Vickers diagonal length. Because the Knoop indent is shallower than the Vickers at the same load, somewhat thinner specimens can be tested. Spacing of indents is important because indenting produces plastic deformation and a strain field around the indent. If the spacing is too small, the new indent will be

affected by the strain field around the last indent. ASTM recommends a minimum spacing (center to edge of adjacent indent) of 2.5 times the Vickers diagonal. For the Knoop test where the long diagonals are parallel, the spacing is 2.5 times the short diagonal. The minimum recommended spacing between the edge of the specimen and the center of the indent should be 2.5 times. Again, Knoop indents can be placed closer to the surface than Vickers indents.

Several studies have stated that the resolution of the optical system is the major factor limiting measurement precision. They state that this causes indents to be undersized by a constant amount. However, as undersizing would increase the measured hardness, not decrease it, resolution limits *per se* cannot explain this problem. For the Knoop indent, the chief problem is image contrast at the indent tips that results in undersized indents. This problem, plus the variable indent shape, both result in increasing HK with decreasing test load. For the Vickers test, one would assume that undersizing or oversizing would be equally likely; but, experience suggests that oversizing is much more commonly encountered for low loads and small indents.

Automation

Microindentation hardness testing is tedious, 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 51 shows the Buehler OmniMet MHT automated microindentation hardness testing system. This system can be used in the fully automatic, semiautomatic or manual mode, depending upon the nature of the testing. OmniMet MHT features a tracing function which allows the operator to outline the exterior of a specimen,. rapidly set multiple indent paths, document the placement of hardness profiles, and automatically generate reports with a profile map, creating a visual record of the entire measurement.

Figure 51. OmniMet MHT

An automated system can be programmed to make any number of indents in either a defined pattern (x number of equally spaced indents, or x number between two chosen points) or a random pattern (locations selected at random with a mouse). Curved patterns may be used, not just straight-line patterns. The system then makes the indents at the requested load and locations, measures each indent, calculates the hardness (and desired conversions to other scales), and prints/plots the results. Statistical analysis can also be preformed.

In general, Vickers indents are easier to measure by image analysis than Knoop indents due to the lower contrast at the tips of the Knoop indents (leads to undersizing and higher HK values). Metal flow (plastic deformation) around the indent edges can interfere with measurement precision. Vickers indents, like the ones shown in Figure 48, 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. In this case, use a higher magnification, or use the semiautomatic measurement mode if this is not possible. The operator fits a box around the indent with a mouse to measure the indent.

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 20-µm in diameter, as recommended in ASTM standard E384. If you have not read E384, or it has been some time since you read it (all ASTM standards must be reviewed and revised, if necessary, every five years), get the latest copy and go over it. The Precision and Bias section (and appendix X1 of E384) contains a wealth of practical advice. Automation of indentation and measurement is possible and, for laboratories that do substantial testing, greatly reduces operator fatigue while reducing testing times.

HELPFUL HINTS FOR MICROINDENTATION 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.

IMAGE CAPTURE AND 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 45. 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 imagin products (Figure 52). 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 measure-

ments. 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 52. 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,

Table 45. 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 field or feature measurements
Data Analysis	Evaluate statistics and relevance of the measurements
Archive	Store images, annotations, and associated measurements in a database
Distribution	Printing or electronic distribution of images and results

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 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.
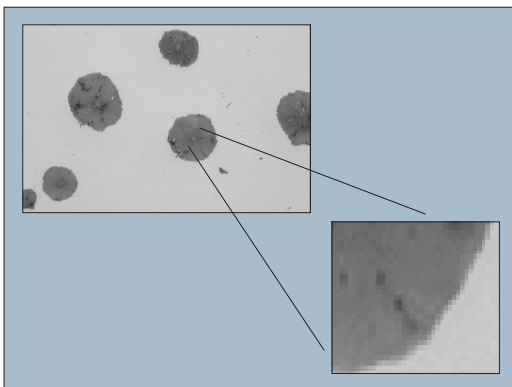
Figure 53. A small segment of an image zoomed to display the individual pixels.

An image on a monitor consists of hundreds of thousands of pixels, arranged in such a manner that they appear to be connected. Figure 53 displays a cast iron image with a small segment zoomed such that the pixels are observed.

Table 46. 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

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 46 lists some of the more common bit depths referred to in imaging software and camera literature.

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 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 54 represents pixels of nine different color values. These values are shown in Table 47 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

Figure 1   Figure 2   Figure 3
Figure 4   Figure 5   Figure 6
Figure 7   Figure 8   Figure 9
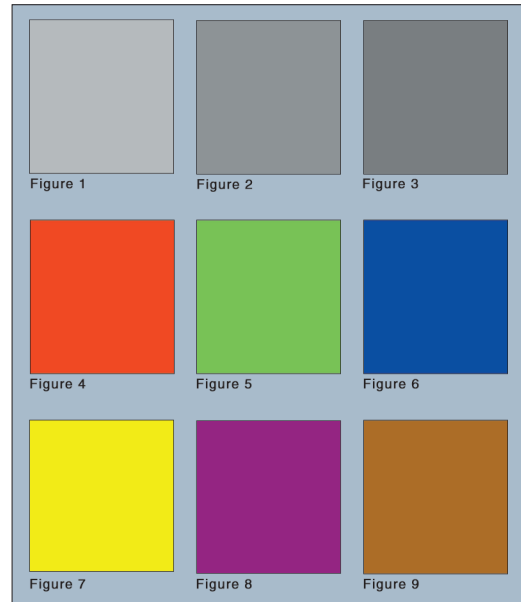
Figure 54. A representation of nine pixels with different color values.

Table 47. Values based on color models

Pixels	Greyscale	Hue	HLS Method Luminance	Saturation	Red	RGB Method 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

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.

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 55). 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 56 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 55. 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 56. 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 57 and is described below.
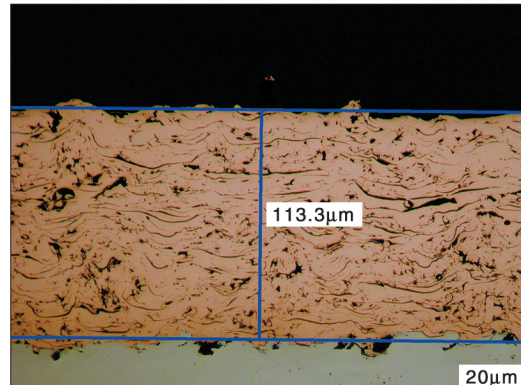
Figure 56. An example of an operator estimating coating thickness with the use of a parallel tool.

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
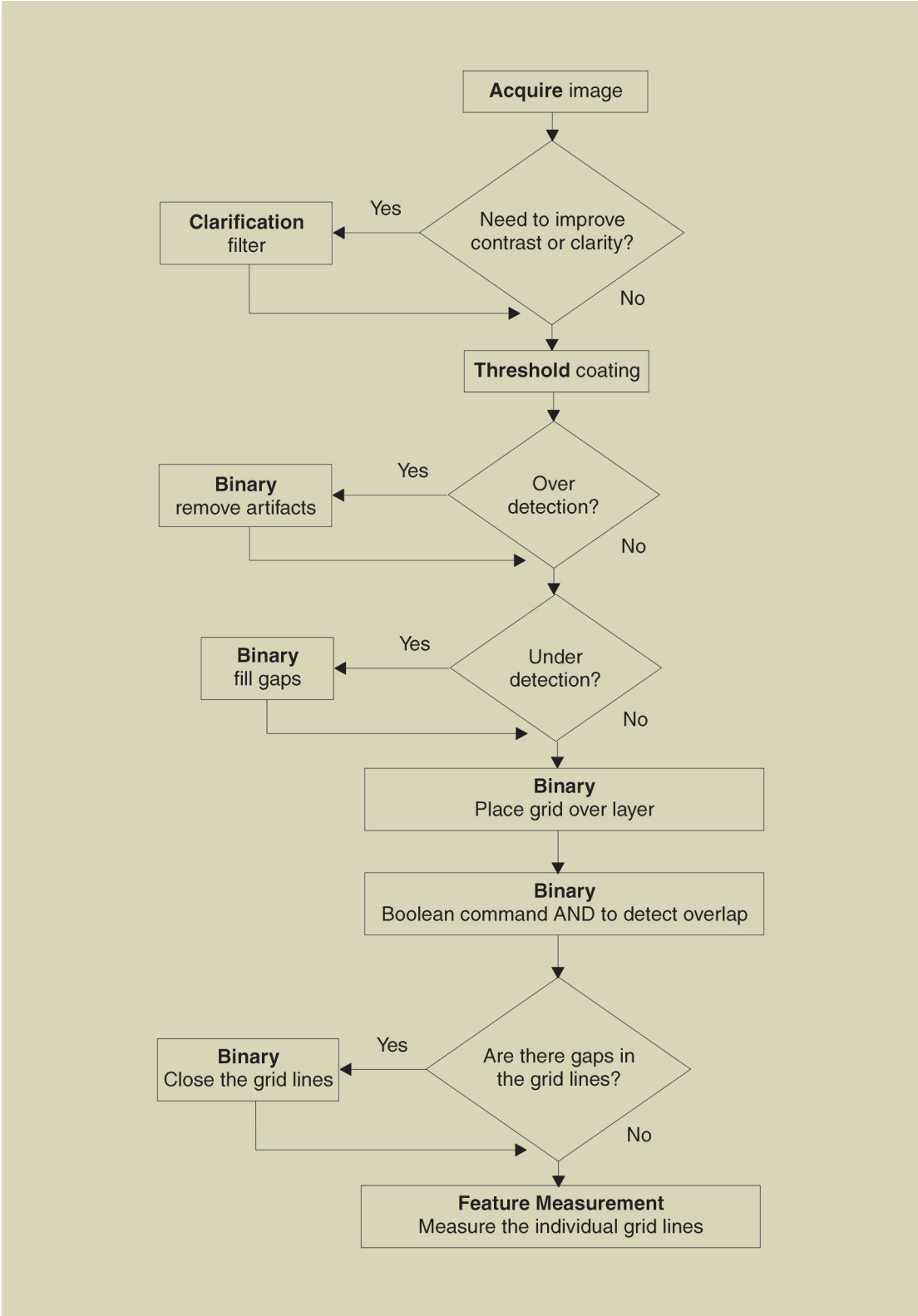
Figure 57. A flowchart of the logic used when creating an automated measurement routine for coating thickness.

to luminance. Figure 59 demonstrates a yellow bitplane being assigned to the coating (Figure 58) based on the HLS values of the pixels as discussed previously.
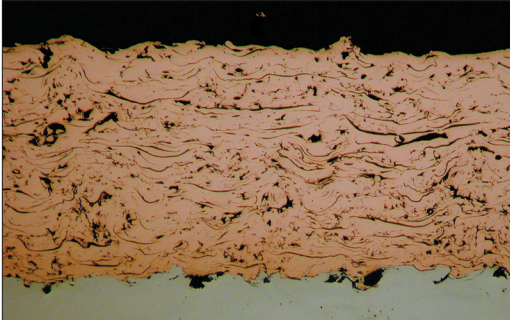

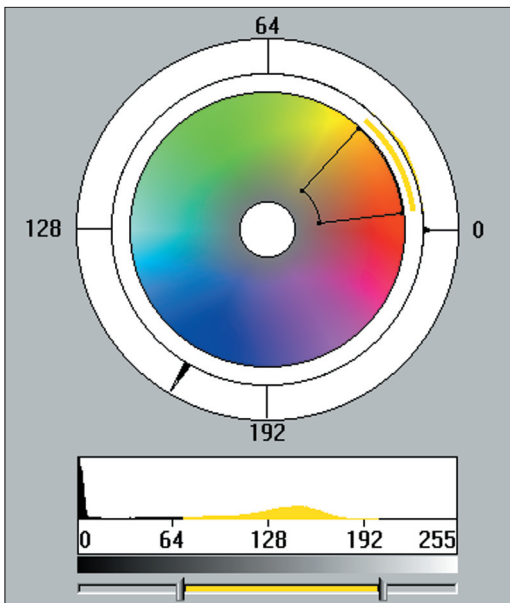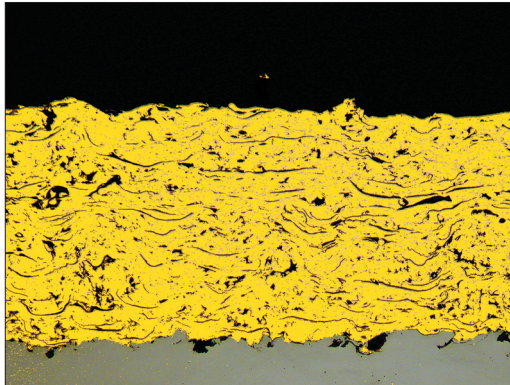Figure 58. A TSC color image captured at 1200 x 1600 pixels.

Figure 59. Thresholding the coating with a yellow bitplane overlay using the HLS color model.

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 morphology 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 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 averag-
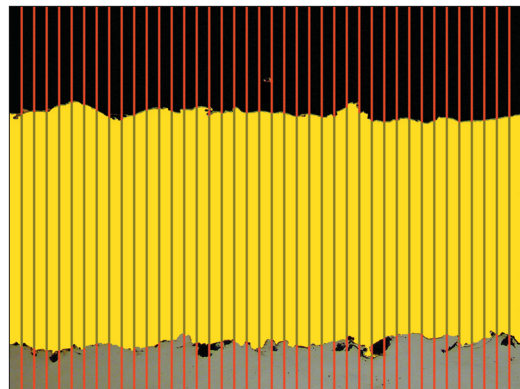
Figure 60. The effect of the Binary commands, Close, Fill, and Grid on the detected coating.

ing 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 60 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
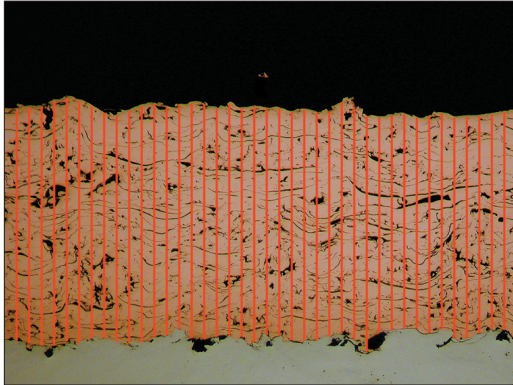
Figure 61. The chords that result from the overlapping coating and grid lines.

segments of the red grid bitplane that overlap the yellow coating bitplane. Figure 61 shows the red chords which result from the AND command. OR combines two source bitplanes together to form a single 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 (>5-µm) 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. For this coating, the pores were separated from the elongated oxides to insure that the porosity level was not overestimated.

Returning to Figure 61, where grid lines are placed across the coating, the length of all the individual
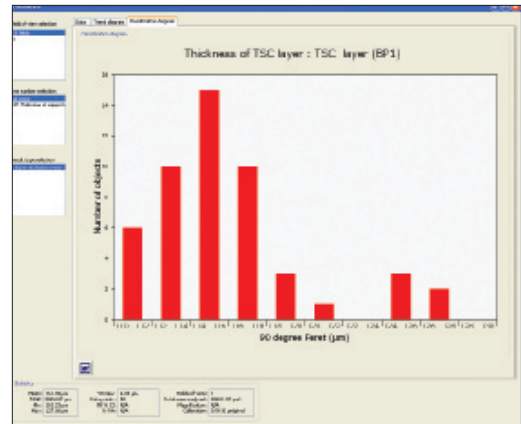
Figure 62. The histogram of results representing the chords shown in Figure 61.

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 (Figure 62).

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 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 the past 20 years of Buehler's 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 "nodular-ity" 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 and 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.

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 Material Safety Data Sheets (MSDS) 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 48 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 63). 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.

Figure 63. Carelessness in the laboratory can result in dangerous chemical spills and a clean-up nightmare.

Most reagents, chemical or electrolytic polishing electrolytes, and solvents should be used under a ventilation hood designed for use with chemicals. Many of these chemicals used in metallo- graphy 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 shatterproof 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

Table 48. 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. MSDS 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

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.

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 cutoff saws 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)

Metallographer 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 polish ing, pickling, etc. The gloves should al-

ways 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 MSDS for most laboratory chemicals and products.

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 1000 mL 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.

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 64. 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 65. If sulfuric acid is added to

Figure 64. 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.

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 65. 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 metallographer'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 49 lists examples from the literature (27-29) of chemical polishing solutions, electrolytic polishing solutions and etchants that have been involved in accidents. Table 50 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 mintlike 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.

Table 49. 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 autocatalytically. 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 autocatalytically. 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 40-10 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 slowing 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 for 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 HNO_3	Etchant for Ni	A closed bottled exploded about 4 hours after it was mixed. Authors state that solutions without the acetic acid also cannot be stored.
10mL HNO_3 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 950mL Isopropyl Alcohol	Etchant	Mixtures have exploded violently after mixing or about 20 minutes after mixing.

Table 50. Some Incompatible Chemicals

Chemical	Use in Metallography	Do Not Mix With the Following:
Acetic 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	Acetic 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 perchlorate and potassium permanganate

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, 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 litre 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 litre 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 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 com- positions of perchloric acid, acetic anhydride, and water, Figure 66. 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.
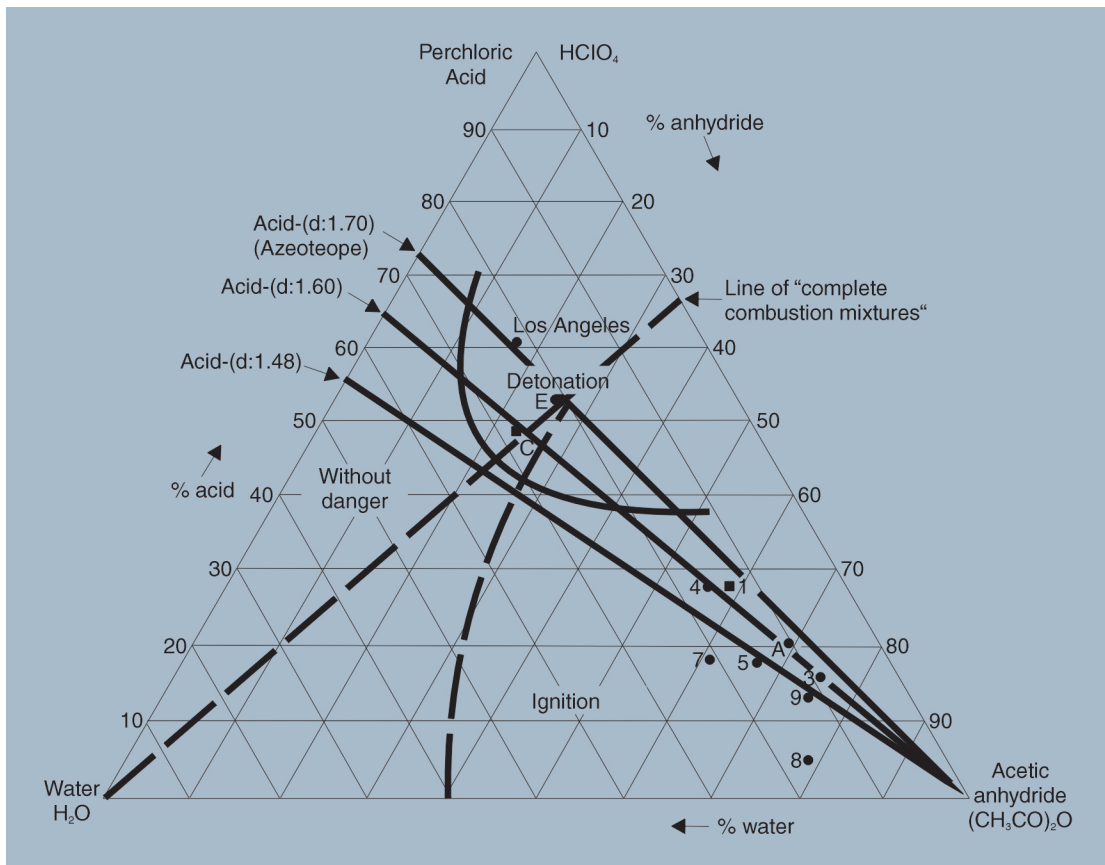
Figure 66. Diagram developed by Médard, Jacquet and Sartorius showing safe (A and 1 to 9) nonexplosive electropolishing solutions and explosive (C, E and Los Angeles) compositions of perchloric acid, acetic anhydride and water (brought in by the perchloric acid, but not corrected for the effect of acetic anhydride).

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 (CH₃COOH) 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 [(CH₃CO)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 49).

Citric acid [C₃H₄(OH)(COOH)₃·H₂O] 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 8 g per 100 mL, 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 [$Fe(NO_3)_3 \cdot 6H_2O$], 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 commonsense 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.

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.

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Useful Web sites on laboratory safety:

http://ull.chemistry.uakron.edu

http://www.hhmi.org/research/labsafe/lcss/lcss.html

http://www.cdc.gov/niosh/ipcsneng/nengsyn.html

http://www.state.nj.us/health/eoh/rtkweb/ rtkhsfs.htm

APPENDICES

Classification of Metals Using Common Characteristics and the Periodic Table

Group	Element	Symbol	Atomic Number	Atomic Weight	Density g/cc@20°C	Melting Point,°C	Crystal Structure
Alkaline	Beryllium	Be	4	9.015	1.848	1280	cph
Earth, IIA	Magnesium	Mg	12	24.312	1.74	650	cph
Coinage	Copper	Cu	29	63.545	8.94	1083	fcc
Metals, IB	Silver	Ag	47	107.87	10.49	961.9	fcc
	Gold	Au	79	197	19.32	1064.4	fcc
Group IIB	Zinc	Zn	30	65.38	7.1	419.57	cph
	Cadmium	Cd	48	112.4	8.65	320.9	cph
Group III	Aluminum	Al	13	26.98	2.7	660	fcc
Group IVA	Tin	Sn	50	118.69	7.29	232	bct
	Lead	Pb	82	207.19	11.35	327.5	fcc
Group IVB	Titanium	Ti	22	47.9	4.507	1670	cph
	Zirconium	Zr	40	91.22	6.44	1853	cph
	Hafnium	Hf	72	178.49	13.21	2225	cph
Group V	Antimony	Sb	51	121.75	6.79	630.5	rhombohedral
	Bismuth	Bi	83	208.98	9.78	271.3	rhombohedral
Group VB	Vanadium	V	23	50.94	6.1	1895	bcc
	Niobium/ Columbium	Nb/ Cb	41	92.91	8.66	2468	bcc
	Tantalum	Ta	73	180.95	16.63	2996	bcc
Group VIB	Chromium	Cr	24	52	7.14	1867	bcc
	Molybdenum	Mo	42	95.94	9.01	2615	bcc
	Tungsten	W	74	183.85	19.3	3410	bcc
Group VIIB	Manganese	Mn	25	54.94	7.3	1244	complex cubic
	Rhenium	Re	75	186.22	21.04	3175	cph
Group VIII	Iron	Fe	26	55.85	7.874	1536	bcc
	Cobalt	Co	27	58.93	8.832	1495	cph
	Nickel	Ni	28	58.71	8.8	1452	fcc
Platinum	Ruthenium	Ru	44	101.1	12.1	2310	cph
Metals,	Rhodium	Rh	45	102.91	12.44	1963	fcc
Group VIII	Palladium	Pd	46	106.4	12.16	1551	fcc
	Osmium	Os	76	190.2	22.48	3015-3075	cph
	Iridium	Ir	77	192.2	22.42	2410	fcc
	Platinum	Pt	78	195.09	21.37	1772	fcc

Transformation Temperatures for Allotropic Metals Listed Above

Element	Transformation	Temperature (C°/F°)
Beryllium (Be)	$\alpha \leftrightarrow \beta$	1256/2293
Cobalt (Co)	$\varepsilon \leftrightarrow \alpha$	427/801
Hafnium (Hf)	$\alpha \leftrightarrow \beta$	1742/3168
Iron (Fe)	Curie	770/1418
	$\alpha \leftrightarrow \gamma$	912/1674
	$\gamma \leftrightarrow \delta$	1394/2541
Manganese (Mn)	$\alpha \leftrightarrow \beta$	707/1305
	$\beta \leftrightarrow \gamma$	1088/1990
	$\gamma \leftrightarrow \delta$	1139/2082
Nickel (Ni)	Curie	358/676
Tin (Sn)	$\alpha \leftrightarrow \beta$	13.0/55.4
Titanium (Ti)	$\alpha \leftrightarrow \beta$	883/1621
Zirconium (Zr)	$\alpha \leftrightarrow \beta$	872/1602

Note: The Curie temperature is the temperature where a magnetic material losses its magnetism upon heating above and regains it upon cooling below.

Appendices

Abrasive Cutting Troubleshooting Guides

Problem	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; loose specimen fixturing; abrupt contact with specimen	Adjust coolant flow to be even on both sides of the wheel; clamp the specimen more tightly; start cut contact carefully
Resistance to cutting	Slow wheel bond breakdown	Select a wheel with softer bonding; use a "pulse" cutting mode; use cutter with oscillating motion or with minimal area of contact cutting ability
Stalled wheel	Inadequate cutter capacity; pinched blade due to movement of specimen	Use cutter with greater horsepower; tighten the clamp on one side less than on the other side; reduce pressure or feed rate; use a cutter with oscillating motion or with minimal area of contact cutting ability

Abrasive Wheel Troubleshooting Guides

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
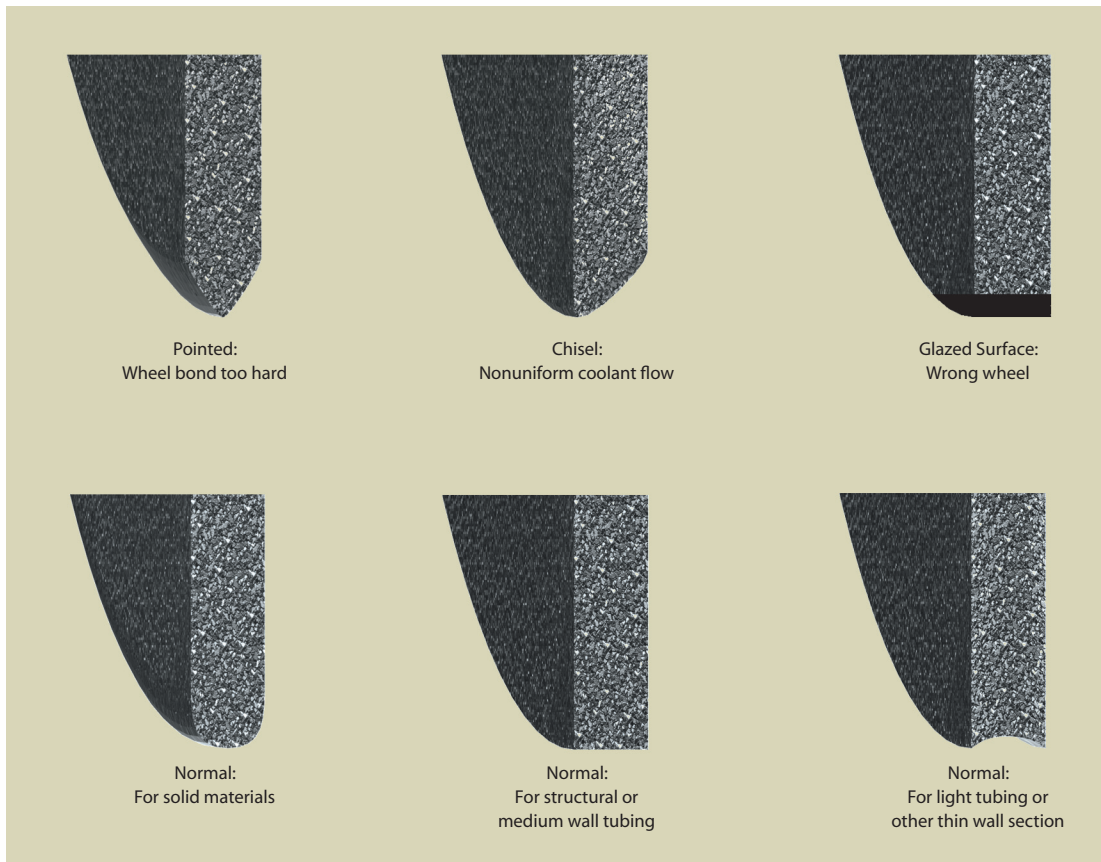
Compression (Hot) Mounting Troubleshooting Guide

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 resins
	Circumferential cracks	Resin contained moisture	Keep resins dry during storage; keep containers closed when not using; dry resin by baking at 38-49°C (100-120°F)
	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

Thermoplastic Resins (Acrylics)

	Defect	Probable Cause	Suggested Remedy
	Cottonball	Incomplete polymerization of resin; not enough time at temperaure	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

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
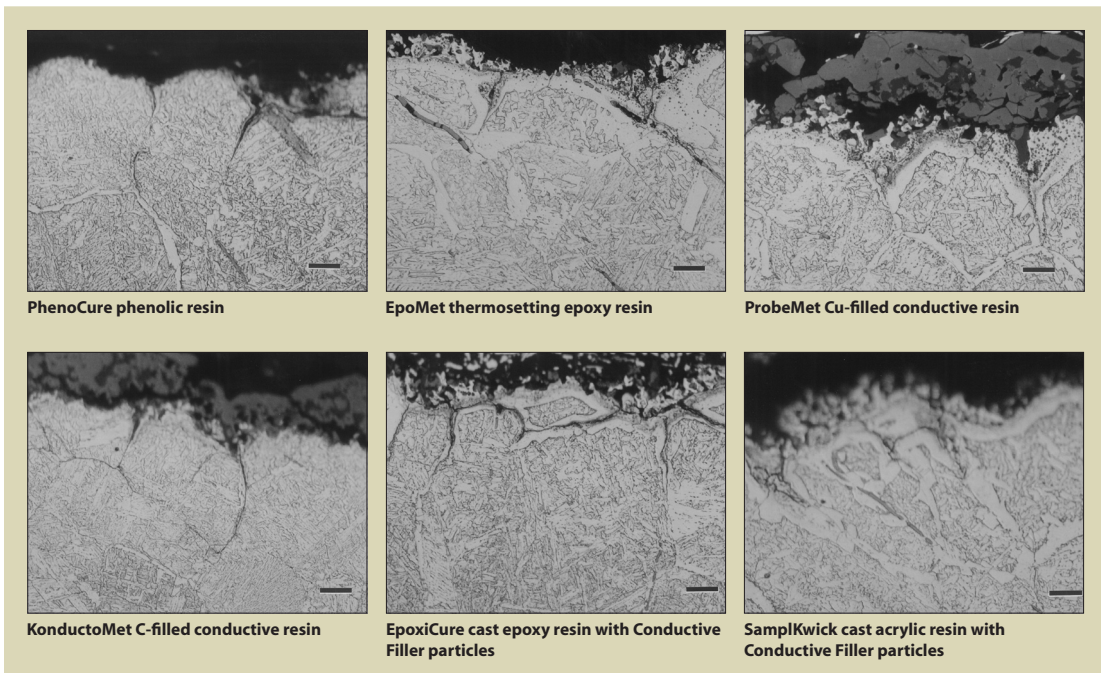
Micrographs showing the as-forged surface of a hardened modified 5130 alloy steel part mounted using a variety of resins showing different degrees of edge retention. The specimens were polished simultaneously in the same holder and were etched with 2% nital. The magnification bars are 20 μm long. Best results were obtained with EpoMet, ProbeMet and EpoxiCure resin with the Conductive Filler particles.

Castable Mounting Resin Selection Guide

Name	Typical Cure Time	Uses and Restrictions
SamplKwick Acrylic Resin	5-8 minutes	Least expensive resin; used for mounting printed circuit boards and polymers; good for x-ray diffraction work (no peak interferences); high shrinkage (not recommended for edge retention)
VariKleer	10-15 minutes	Designed for use with a pressure vessel, this general use acrylic hardens crystal clear with minimal shrinkage. To achieve crystal clear samples, reusable silicon or EPDM molds should bot be used.
VariDur 3000	15-30 minutes	Minimal shrinkage, good edge retention
VariDur Acrylic Resin	10 minutes	Inexpensive; (good edge rentention for acrylic); used with many metals when the edge detail is not important
EpoKwick Epoxy Resin	90 minutes	Fast curing epoxy; more shrinkage than other epoxies; viscosity 250-400 cps at 25 °C; not recommended for vacuum infiltration of voids due to fast curing time; physically adheres to specimen; gives acceptable edge retention; good for most specimens
EpoColor Epoxy Resin	90 minutes	Provides color contrast between mount and specimen that can be helpful when studying edges; viscosity 400-700 cps at 25 °C; good for most specimens; but not recommended for vacuum impregnation
EpoxiCure Epoxy Resin	6 hours	General purpose epoxy; viscosity 400-600 cps at 25 °C, good adherence to specimen; can be used to vacuum impregnate voids (viscosity can be reduced by warming to 50 °C); good for heat-sensitive specimens (very low exotherm during polymerization)
EpoxiCure NC	5 hours	General purpose epoxy - No hazard shipping fees.
EpoHeat Low Viscosity Epoxy	90 minutes	Fast, heat cure epoxy system with low shrinkage and low viscosity (32 cps at 176 °F (80 °C)). Excellent for impregnation. Requires oven cure. Golden color.
EpoThin Epoxy Resin	9 hours	Lowest viscosity (can be further reduced by warming resin to 50 °C) and best penetration of voids during vacuum impregnation; good adherence to specimen; good for heat-sensitive specimens (lowest exotherm during polymerization)

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.

Greek Alphabet and its English Equivalents

Greek Letter	Name	English Equivalent	Greek Letter	Name	English Equivalent
Α, α	Alpha	A, a	Ν, ν	Nu	N, n
Β, β	Beta	B, b	Ξ, ξ	Xi	X, x
Γ, γ	Gamma	G, g	Ο, ο	Omicron	O, o
Δ, δ	Delta	D, d	Π, π	Pi	P, p
Ε, ε	Epsilon	E, e	Ρ, ρ	Rho	R, r
Ζ, ζ	Zeta	Z, z	Σ, σ	Sigma	S, s
Η, η	Eta	E, e	Τ, τ	Tau	T, t
Θ, θ	Theta	Th	Υ, υ	Upsilon	U, u
Ι, ι	Iota	I, i	Φ, φ	Phi	Ph
Κ, κ	Kappa	K, k	Χ, χ	Chi	Ch
Λ, λ	Lambda	L, l	Ψ, ψ	Psi	Ps
Μ, μ	Mu	M, m	Ω, ω	Omega	O, o

Cast Resin ("Cold" Mounting) Troubleshooting Guide

Defect	Probable Cause	Suggested Remedy
Non-Curing	Incomplete mixing; Wrong resin – to – hardener ratio	Measure weights of resin and hardener; after prescribed time, and it has not hardened, heat the mold, liquid and specimen to 50 °C (120 °F) to force curing; check age before using resin and hardener
Slow Curing	Incomplete mixing; too little hardener used; curing at too low a temperature	Mix thoroughly; measure weights of resin and hardener; place in warm area*
Rapid Curing	Too much hardener; curing at too high a temperature; mold size too large (excessive resin volume)	Measure weights of resin and hardener; mix smaller quantities; increase air circulation; place in a cooler place
Gas Tunnels	Excessive hardener used; mold size too large (excessive resin volume); use of a low thermal conductivity mold molds	Measure weights of resin and hardener; increase air circulation; use higher thermal conductivity
Mount Stuck in Mold to interior surfaces of mold	Did not use mold release agent	Apply Silicon Mold Release or Release Agent
Solvent Softening	Poor resistance to solvents; mount may not be fully cured	Heat the mold, liquid and specimen to 50 °C (120 °F) to force curing
Excessive Shrinkage	Polymerization is too fast; too much hardener used; wrong resin selected	Use an epoxy rather than an arcylic; use a slow curing epoxy; add Flat Edge Filler particles; use the correct resin – to – hardener ratio

* Placing in an oven at 120°F (50°C) for up to two hours may accelerate the curing of epoxies

Guide to Fine Polishing Abrasives

Name	Characteristics	Applications
MicroPolish alumina powder	1-, 0.3- and 0.05-µm alumina powder	Inexpensive general-purpose abrasive for polishing most minerals and metals
MicroPolish II deagglomerated alumina powder and suspensions	1-, 0.3- and 0.05-µm deagglomerated alumina powders and 5-, 1-, 0.3- and 0.05-µm deagglomerated alumina suspensions	Higher quality alumina suitable for polishing most minerals and metals; produces a better surface finish than standard alumina abrasives of the same size
MasterPrep agglomerate-free seeded-gel alumina suspension	0.05-µm sol-gel alumina suspension	Unique seeded-gel alumina produces better finishes than calcined alumina abrasives; excellent with minerals, metals, carbides, PCBs. Good for percious metals.
MasterMet colloidal silica suspension	0.06-µm amorphous silica colloidal suspension with a pH of ~10 producing a chemo-mechanical polishing action	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	0.02-µm non-crystallizing amorphous silica colloidal 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 0.05-µm alumina and colloidal silica with a slightly basic pH (can be diluted with water)	Proprietary blend has little water and can be used with materials that are sensitive to water, such as Mg and most of its alloys; good for most Fe-, Ni- or Co-based alloys and MMCs
MasterPolish 2 proprietary chemo-mechanical suspension	Proprietary 0.06-µm 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

A Guide to Equivalent Loads for Different Mount Sizes
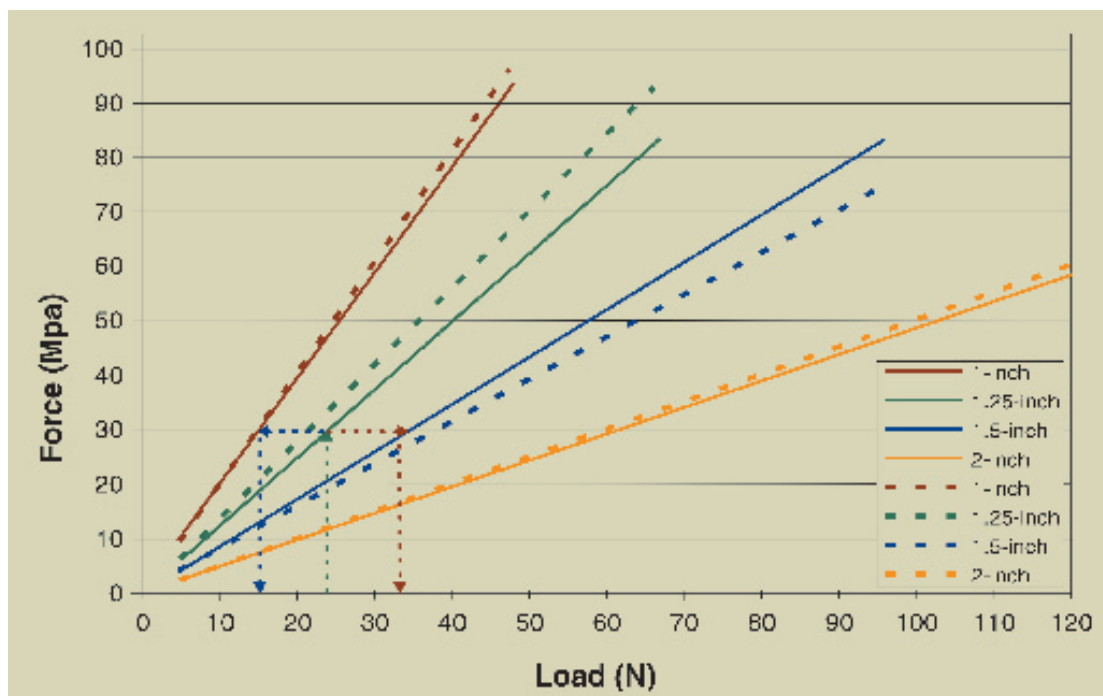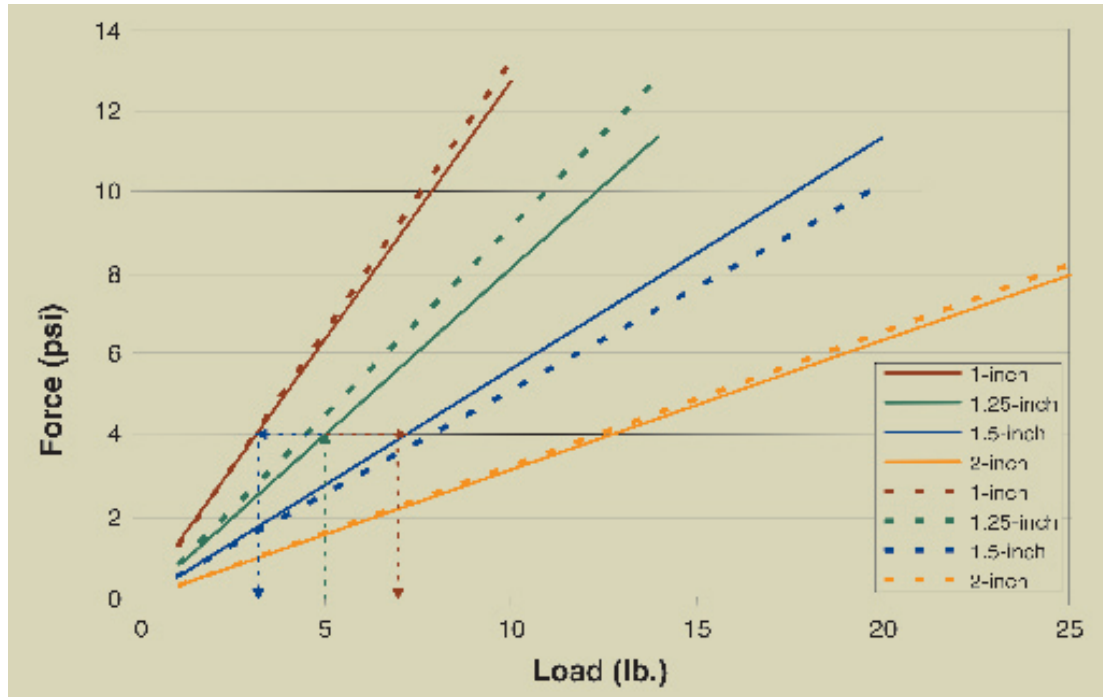
The two graphs plot the load applied per specimen, and the resulting pressure, as a function of mold diameter for both Imperial and metric units. An example of the use of the graphs is shown. Assume that we have a practice developed using 1.25-inch diameter mounts that calls for 5 lbs. load per specimen. We want to use this method with mounts that are 1-inch 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 1.25-inch diameter mount. Then draw a horizontal line (shown in blue) from the intersection point to the line for 1-inch 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 1-inch mounts. In similar manner, if we want to use 1.5-inch diameter specimens, the graph would suggest that we use about 7 lbs. per specimen (red lines).

Preparation Troubleshooting Guide

Troubleshooting Definitions

Comet Tails: 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. The hard particles may be nonmetallic inclusions or nitrides. This appears to be a material-specific phenomenon as repeated efforts to produce this defect in randomly chosen specimens fail. Historically, with manually-prepared specimens, it was claimed to be caused by unidirectional grinding; however, repeated efforts to create comet tails with only unidirectional grinding fail. Comet tails have been observed on susceptible specimens prepared with automated machines using only complementary grinding and polishing with specific combinations of head and platen speeds. In a specimen prone to comet tailing, using contra rotation in the final step will enhance the problem compared to complementary rotation.

Edge Rounding: 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 with the light microscope (this condition is more critical as the magnification is raised due to the higher numerical aperture of the objective which reduces the depth of focus).

Embedding: Hard abrasive particles that become fixed in the surface of the softer specimen. This is a common problem with the low melting point alloys (e.g., Pb, Sn, Bi, Cd, Zn), aluminum (mainly fine diamond), and precious metals, but has been observed with refractory metals such as titanium.

Pull Out: Removal of second phase particles (either softer or harder than the matrix) during preparation. Pull out may be enhanced if the interface between the particle and matrix is weak or if the particle is particularly brittle.

Relief: Excessive height differences between second-phase particles and the matrix due to differences in grinding and polishing rates between the matrix and the particles. Soft particles abrade faster than the matrix and are recessed below the matrix while the reverse is observed for hard particles.

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. The width and depth of the cut is a function of the size of the abrasive particle, its angle to the specimen surface, the applied pressure, and other factors. Scratches become a problem when they are excessively coarse for the abrasive size used, indicating a problem with the preparation method. Very fine scratches may not be a problem in routine examination for grain size or inclusions but could be a problem in failure analysis if critical fine detail is obscured.

Smear: Matrix flow over voids, cracks, or second phase particles that make detection of these features and measurement of their extent difficult or impossible.

Stain: A contamination residue on the surface of a specimen, that may form around second-phase particles due to interactions between the specimen and abrasives and/or lubricants, or may form on the matrix due to inadequate cleaning or drying or may form due to interactions between the specimen and solvents after preparation or etching.

Preparation Troubleshooting Guide Cont.

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 or 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 ApexHercules rigid grinding discs; use napless cloths.
Embedding	Hard abrasive tends to embed in soft metals; finer particles are more likely to embed than larger particles.	Coat SiC abrasive paper with paraffin 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 $\leq$ 3-μm 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 last 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 and pressures can leave a heavy magnetic disc system, store the discs in a clean scratch pattern. Drawer or cabinet and scrub surfaces if contamination is believed to have occurred.
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, such as with the TorraMet dryer. Compressed air systems can contain impurities, such as oils, that can stain the surface.

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 HARDNESS TESTING 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 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

Sweden (continued)

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

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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