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Metallographic Handbook

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by Donald C. Zipperian, Ph.D.

METALLOGRAPHIC HANDBOOK

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CHAPTER 1
Introduction to Metallography

Metallography has been described as both a science and an art. Traditionally, metallography has been the study of the microscopic structure of metals and alloys using optical metallographs, electron microscopes or other surface analysis equipment. More recently, as materials have evolved, metallography has expanded to incorporate materials ranging from electronics to sporting good composites. By analyzing a material's microstructure, its performance and reliability can be better understood. Thus metallography is used in materials development, incoming inspection, production and manufacturing control, and for failure analysis; in other words, product reliability.

Metallography or microstructural analysis includes, but is not limited to, the following types of analysis:

- Grain size
- Porosity and voids
- Phase analysis
- Dendritic growth
- Cracks and other defects
- Corrosion analysis
- Intergranular attack (IGA)
- Coating thickness and integrity
- Inclusion size, shape and distribution
- Weld and heat-affected zones (HAZ)
- Distribution and orientation of composite fillers
- Graphite nodularity
- Recast
- Carburizing thickness
- Decarburization
- Nitriding thickness
- Intergranular fracturing
- HAZ Sensitization
- Flow-line Stress

Grain Size

For metals and ceramics, grain size is perhaps the most significant metallographic measurement because it can be directly related to the mechanical properties of the material. Although grain size is actually a 3-dimensional property, it is measured from a 2-dimensional cross section of the material. Common grain size measurements include grains per unit area/ volume, average diameter or grain size number. Determination of the grain size number can be calculated or compared to standardized grain size charts. Modern image analysis algorithms are very useful for determining grain size.

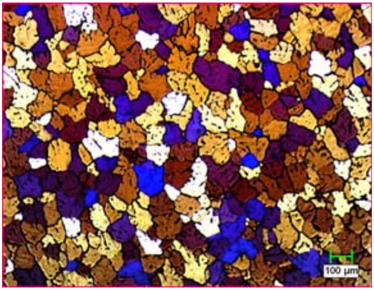


Figure 1-1 Grain size- anodized aluminum. (photo courtesy of Clemex Technologies)

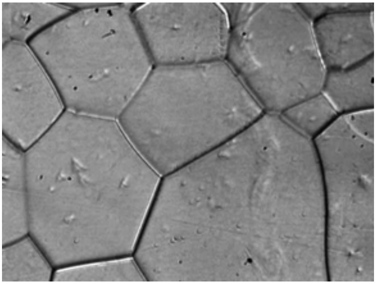


Figure 1-2 Rhenium grain size.

Twin Boundaries

Twin boundaries occur when two crystals mirror each other. For some materials, twinning occurs due to work hardening at low temperatures. To correctly determine the grain size in these types of materials, the twin boundaries need to be removed from the calculation.

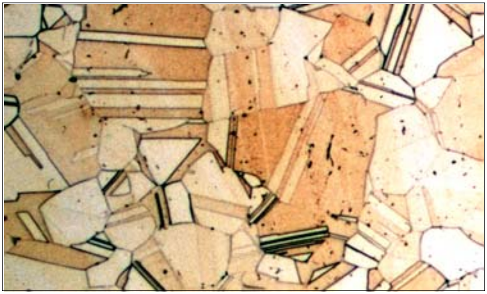


Figure 1-3 Twin boundaries in brass.

Porosity and Voids

Holes or gaps in a material can generally be classified as either porosity or voids. Porosity can also refer to holes resulting from the sintering of metal or ceramic powders or due to casting shrinkage issues. Voids are generally a result of entrapped air and are common in wrapped or injection molded materials such as polymer matrix composites (PMC's).

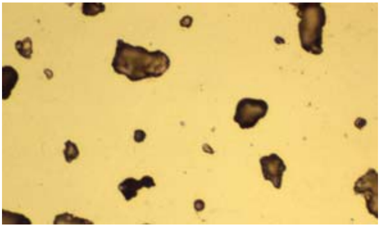


Figure 1-4 Porosity in a BaCl ceramic.

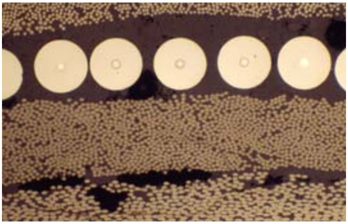


Figure 1-5 Voids due to entrapped air in a Boron-graphite composite.

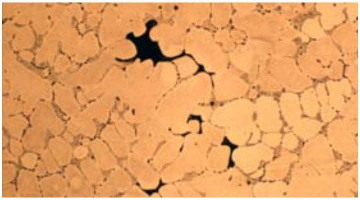


Figure 1-6 Casting porosity in copper.

Cracks

Defects such as cracking can lead to catastrophic failure of a material. Metallography is often used in failure analysis to determine why a material broke, however, cross sectional analysis is also a very useful technique to evaluate manufacturing issues which may cause these defects.

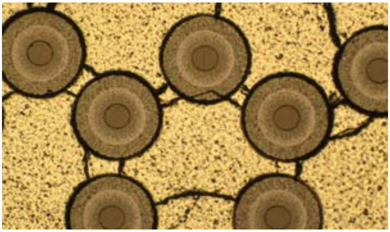


Figure 1-7 Stress cracks in a ceramic matrix composite.

Figure 1-8 Welding crack in a copper-stainless steel weld.

Phases

Metal alloys can exhibit different phase (homogenous) regions depending upon composition and cooling rates. Of interest to the metallographer might be the distribution, size and shape of these phases. For composite materials, identification and characteristics of the filler would also be of interest.

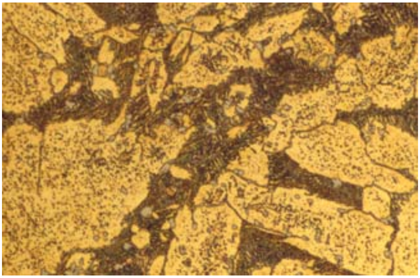


Figure 1-9 Ni-Fe-Al bronze phases.

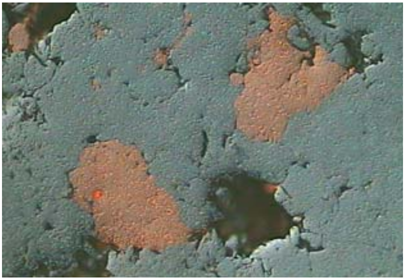


Figure 1-10 Copper and iron phases in a cold pressed metal.

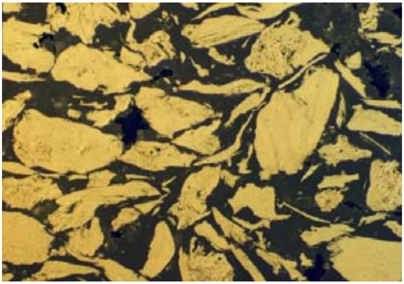


Figure 1-11 Graphite-polymer composite.

Dendrites

By slowly solidifying a molten alloy, it is possible to form a treelike dendritic structure. Dendrites initially grow as primary arms and depending upon the cooling rate, composition and agitation, secondary arms grow outward from the primary arms. Likewise, tertiary arms grow outward from the secondary arms. Metallographic analysis of this structure would consist of characterizing the dendrite spacing.

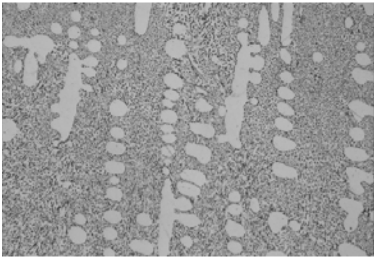


Figure 1-12 Dendrite in Al-Si alloy.

Figure 1-13 Dendrite treelike structure.

Corrosion

The effects of corrosion can be evaluated by metallographic analysis techniques in order to determine both the root cause as well as the potential remedies.

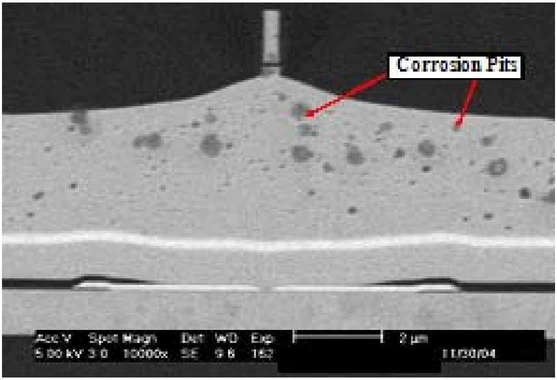


Figure 1-14 Corrosion analysis of a magnetic read-write hard-drive component.

Intergranular Attack

Intergranular corrosion (IGC), also termed intergranular attack (IGA), is a form of nonuniform corrosion. Corrosion is initiated by inhomogeneities in the metal and is more pronounced at the grain boundaries when the corrosion-inhibiting compound becomes depleted. For example, chromium is added to nickel alloys and austenitic stainless steels to provide corrosion resistance. If the chromium becomes depleted through the formation of chromium carbide at the grain boundaries (this process is called sensitization), intergranular corrosion can occur.

Figure 1-15 Intergranular alloy depletion in nickel.

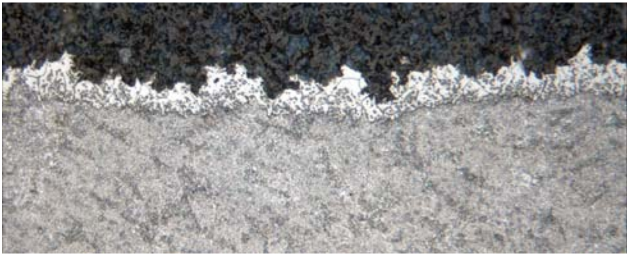


Figure 1-16 Intergranular attack in nickel.

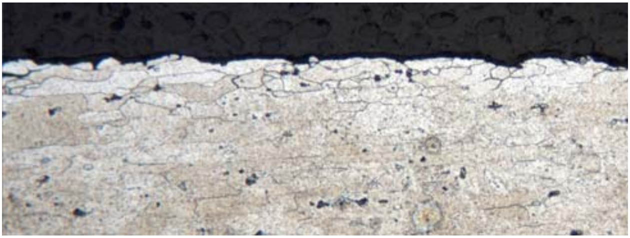


Figure 1-17 Intergranular cracking in aluminum.

Coating Thickness

Coatings are used to improve the surface properties of materials. Coatings can improve temperature resistance (plasma coating), increase hardness (anodizing), provide corrosion protection (galvanized coatings), increase wear resistance, and provide better thermal expansion adherence for dielectric/ metal interfaces. Metallographic analysis can provide useful information regarding coating thickness, density, uniformity and the presence of any defects.

Figure 1-18 Plasma spray coating.

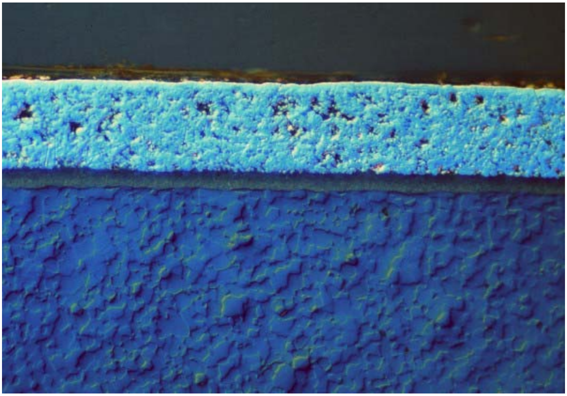


Figure 1-19 AlN dielectric with metallized coating.

Inclusions

Inclusions are foreign particles that contaminate the metal surface during rolling or other metal forming processes. Common inclusion particles include oxides, sulfides or silicates. Inclusions can be characterized by their shape, size and distribution.

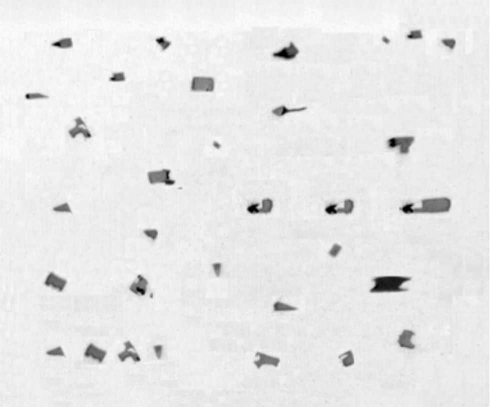


Figure 1-20a Oxide inclusions in steels (photo courtesy of Clemex Technologies).

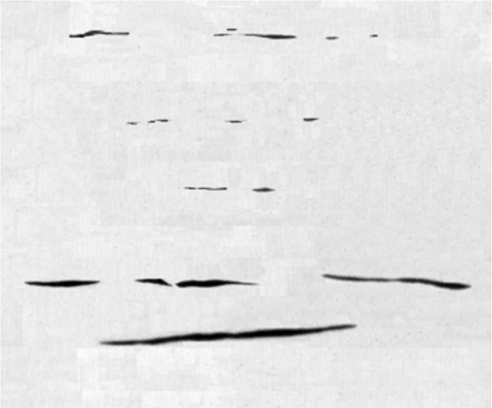


Figure 1-20b Sulfide inclusions in steels (photo courtesy of Clemex Technologies).

Weld Analysis

Welding is a process for joining two separate pieces of metal. The most common welding processes produce localized melting at the areas to be joined, this fused area is referred to as the bead and has a cast-like structure. The area or zone adjacent to the bead is also of interest and is known as the HAZ (heat affected zone). Typically the welded area will have a different microstructure and therefore different physical and mechanical properties as compared to the original metals. Analysis can also include evaluating cracks and interdiffusion of the base metals within the welded area.

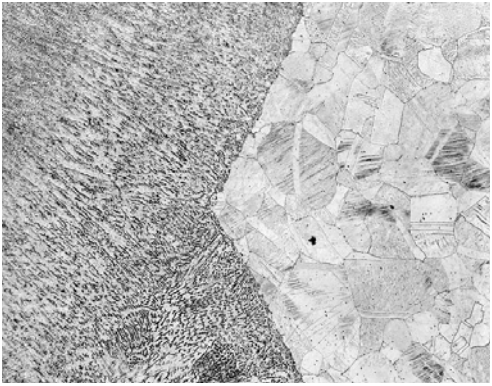


Figure 1-21a Perfect steel weld.

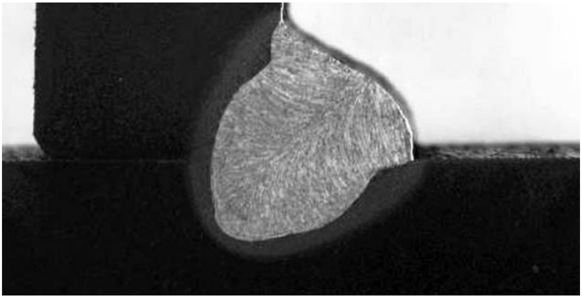


Figure 1-21b Filet steel weld
(photo courtesy of Clemex Technologies).

Figure 1-22 Copper-stainless steel weld diffusion of the stainless steel into the copper

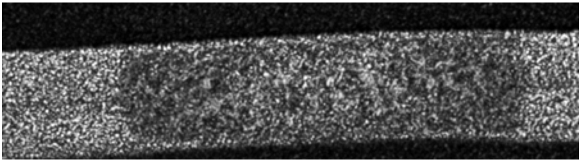


Figure 1-23a Seam weld with complete penetration.

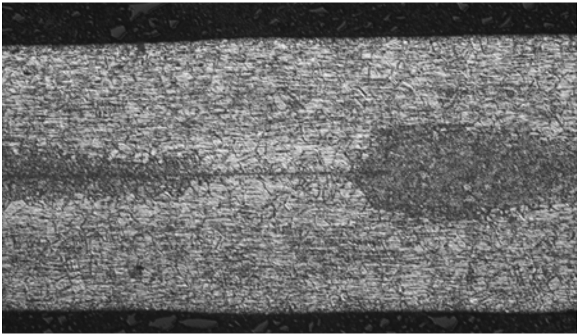


Figure 1-23b Discontinuous seam weld with poor penetration.

Solder Joint Integrity

For electronic components, the integrity of the solder joints is very important for characterizing the reliability of electronic components.

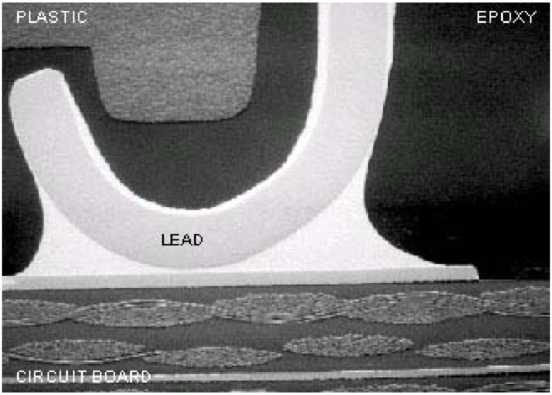


Figure 1-24 Electronic circuit board solder joint.

Composites

Composites are engineered materials which contain fillers in a matrix. Common fillers include ceramic or graphite particles and carbon or ceramic fibers. These fillers are encased, or cast, into a polymer, metal, or ceramic matrix. Metallographic analysis of composites includes analyzing the orientation and distribution of these fillers, voids and any other defects.

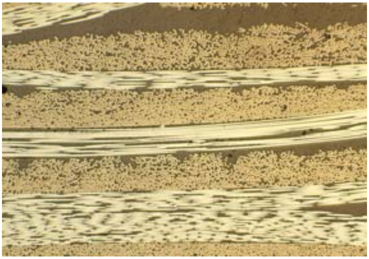


Figure 1-25 Carbon fiber composite.

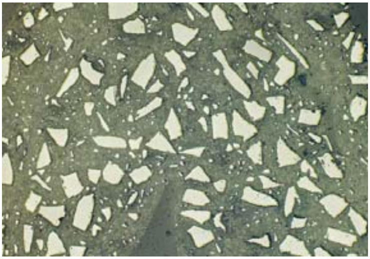


Figure 1-26 SiC particles in a metal matrix.

Graphite Nodularity

Cast irons are typically characterized by their nodularity (ductile cast iron) or by their graphite flakes (gray cast iron). Since gray cast irons can eventually fail due to brittle fracture, ductile nodular cast irons are the preferred structure. To produce ductile cast irons, magnesium or cerium are added to the iron melt prior to solidification. Cross-sectional analysis is used to characterize the melt prior to pouring the entire batch.

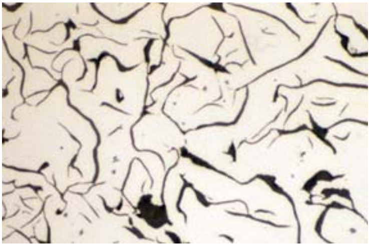


Figure 1-27a Gray cast iron (graphite flakes), as polished.

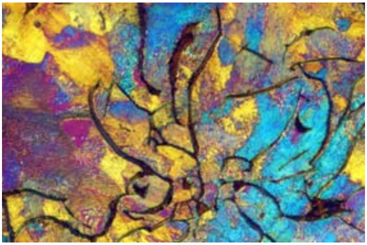


Figure 1-27b Gray cast iron (graphite flakes), etched

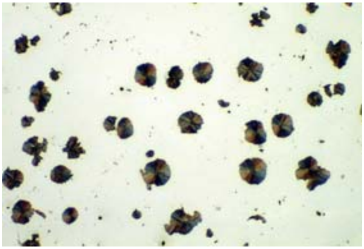


Figure 1-28a Nodular cast iron as polished.

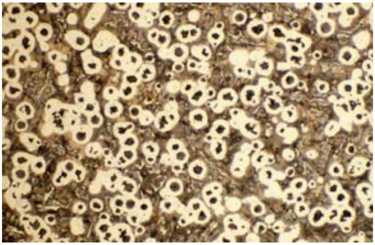


Figure 1-28b Nodular cast iron, etched.

Recast

The recast layer is made up of molten metal particles that have been redeposited onto the surface of the workpiece. Both the HAZ (heat affected zone) and recast layer can also contain microcracks which could cause stress failures in critical components.

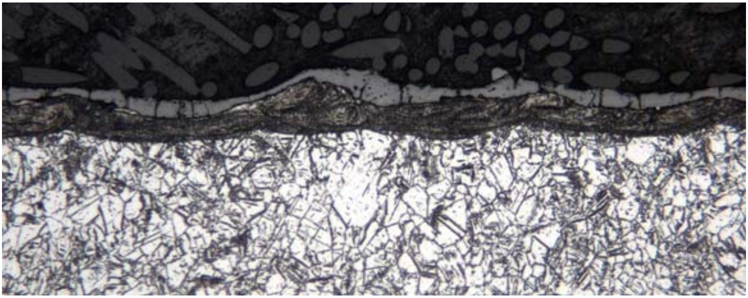


Figure 1-29 Continuous recast layer.

Figure 1-30 Localized recast layer.

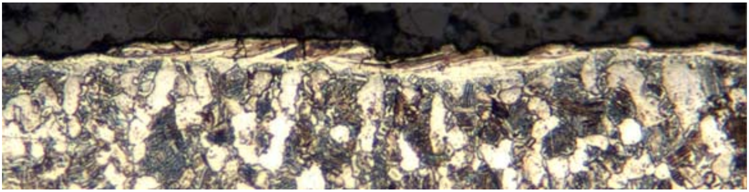


Figure 1-31 Cracks in recast layer.

Carburizing

The most common heat treating process for hardening ferrous alloys is known as carburizing. The carburizing process involves diffusing carbon into ferrous alloys at elevated temperatures. By quenching the metal immediately after carburizing, the surface layer can be hardened. Metallographic analysis, along with microhardness testing, can reveal details regarding the case hardness and its depth.

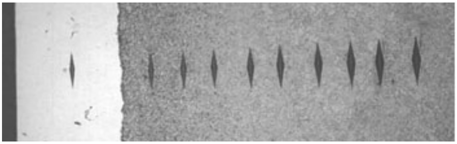


Figure 1-32 Knoop case depth hardness.

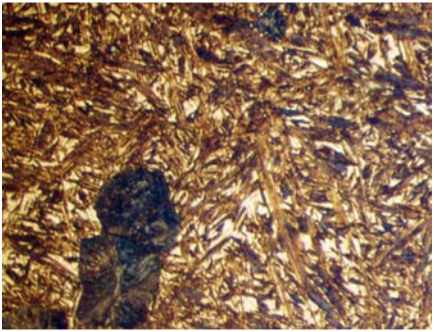


Figure 1-33 High carbon steel, quenched.

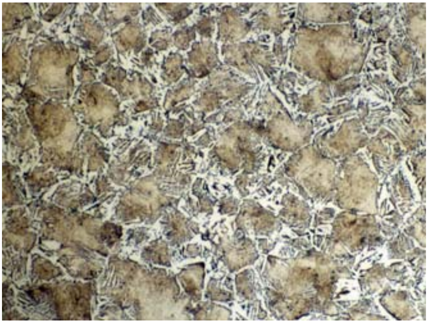


Figure 1-34 Low carbon steel, quenched.

Decarburization

Decarburization is a defect which can occur when carbon is lost at the surface of a steel when it is heated to high temperatures, especially in hydrogen atmospheres. This loss of carbon can reduce both the ductility and strength of the steel. It can also result in hydrogen embrittlement of the steel.

Figure 1-35 Gross decarburization in a steel fastener.

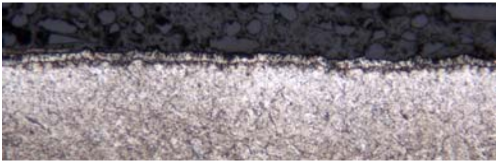


Figure 1-36 Steel decarburization.

Nitriding

Nitriding is a process for producing a very hard case on strong, tough steels. The process includes heating the steel at 500-540°C (930-1000°F) in an ammonia atmosphere for about 50 hours. No additional quenching or heat treating is required. The Vickers hardness is about 1100 and the case depth is about 0.4 mm. Nitriding can also improve the steel's corrosion resistance.

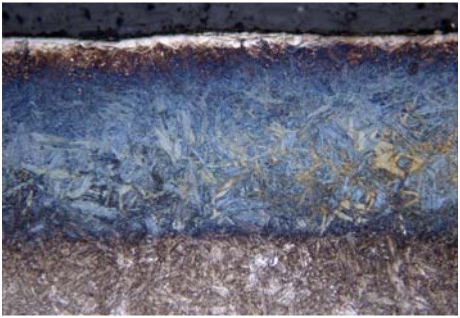


Figure 1-37 Nitrided steel.

Intergranular Fracture

Intergranular cracking or fracturing is a fracture that occurs along the grain boundaries of a material. An intergranular fracture can result from improper heat treating, inclusions or second-phase particles located at grain boundaries, and high cyclic loading.

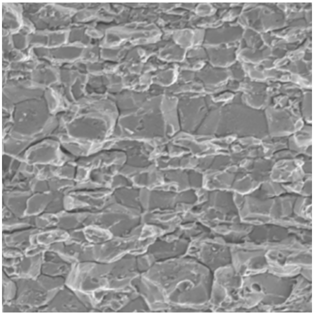


Figure 1-38 Intergranular fracturing for improperly heat treated 17-7PH, 1000X.

Weld Sensitization

Sensitization is a condition where the chromium as an alloy becomes depleted through the formation of chromium carbide at the grain boundaries. For welding, sensitization occurs due to slow heating and cooling through a temperature range specific to the alloy being welded. For example, 300 series stainless steels form chromium carbide precipitates at the grain boundaries in the range of 425-475°C.

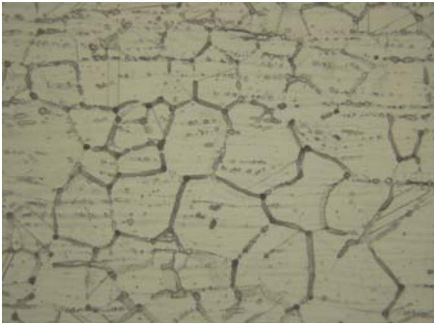


Figure 1-39 Sensitization of welded 304L Stainless Steel, Mag. 500X.

Flow Line Stress

Flow stress is the stress required to keep a metal flowing or deforming. the direction of the flow is important.

Figure 1-40 Improper flow line direction normal to maximum stress, Etchant $HCl+H_2O_2$.

CHAPTER 2
Abrasive Sectioning

2.0 ABRASIVE SECTIONING

The first step in preparing a specimen for metallographic or microstructural analysis is to locate the area of interest. Sectioning or cutting is the most common technique for revealing the area of interest. Proper sectioning has the following characteristics:

DESIRABLE EFFECTS:
- Flat and cut close to the area of interest
- Minimal microstructural damage

Figure 2-1 Abrasive Cut-off Blades and Coolants.

UNDESIRABLE EFFECTS:
- Smeared (plastically deformed) metal
- Heat affected zones (burning during cutting)
- Excessive subsurface damage (cracking in ceramics)
- Damage to secondary phases (e.g. graphite flakes, nodules or grain pull-out)

The goal of any cutting operation is to maximize the desirable effects, while minimizing the undesirable effects.

Sectioning can be categorized as either abrasive cutting or precision wafer cutting. Abrasive cutting is generally used for metal specimens and is accomplished with silicon carbide or alumina abrasives in either a resin or resin-rubber bond. Proper blade selection is required to minimize burning and heat generation during cutting, which degrades both the specimen surface as well as the abrasive blades cutting efficiency. Wafer cutting is achieved with very thin precision blades. The most common wafering blades are rim-pressed abrasive blades, in which the abrasive is located along the edge or rim of the blade. Precision wafering blades most commonly use diamond abrasives, however, cubic boron nitride (CBN) is also used for cutting samples that react to dull diamond (e.g. high carbon, heat treated steels cut more effectively with CBN as compared to diamond). Wafer cutting is especially useful for cutting electronic materials, ceramics and minerals, bone, composites and even some metallic materials.

2.1 ABRASIVE BLADE SELECTION GUIDELINES

Selecting the correct abrasive blade is dependent upon the design of the cut-off machine and, to a large extent, the operator preference. Abrasive blades are generally characterized by their abrasive type, bond type and hardness. Determining the correct blade is dependent upon the material or metal hardness and whether it is a ferrous or a nonferrous metal. In practice, it often comes down to odor and blade life. Resin/rubber blades smell more because the rubber will burn slightly during cutting, however resin/rubber blades do not wear as fast and therefore last longer. On the other hand, resin blades are more versatile and do not produce a burnt rubber odor, but they do break down faster. Resin blades also provide a modestly better cut because the cutting abrasive is continually renewed and thus produces a cleaner cut.

Also note that the traditional “older” technology for producing abrasive blades resulted in very specialized resin/rubber blades. Finding the proper resin/rubber hardness, abrasive size, and blade thickness to match the sample properties and the cutting machine parameter required a lot of testing and experimentation. Thus, in the past, resin/rubber blades had been more popular in the US market; however, in more recent years as resins have improved, there has been more of a trend towards resin bonded abrasives. Conversely, resin bonded blades have typically been more widely used in the European and Asian markets for quite some time.

MAXCUT-A Blade **MAXCUT-C Blade** **MAXCUT-D Blade**

MAXCUT-D-RT Blade **MAXCUT-E Blade** **MAXCUT-I Blade**

Figure 2-2 Cutting blades for specific cutting requirements.

TABLE I. Abrasive Blade Selection Guidelines

Material	**Composition**	**Recommended Blade**
Soft non-ferrous metals (aluminum, brass, zinc, etc.)	Alumina / resin bonded	MAX-E
Hard non-ferrous metals (titanium, zirconium, etc.)	Silicon carbide / resin-rubber bond	MAX-C
Soft steels	Alumina / resin bonded	MAX-E
Hard and case hardened steels	Alumina / resin bonded	MAX-D
General steel and ferrous metals	Alumina / resin bonded reinforced-thin blade	MAX-D-RT
Universal thin resin / rubber blade	Alumina / resin-rubber bonded	MAX-A
Industrial general purpose thin blade	Alumina / resin bonded	MAX-I

Summary:

- Resin bonded blades - less smell, higher wear, less sample burning, more versatile
- Resin-rubber bonded blades - longer life, burnt rubber smell, more likely to burn the sample, more difficult to find the correct blade

2.2 ABRASIVE CUTTING PROCESS DESCRIPTION

Abrasive sectioning has primarily been used for sectioning ductile materials. Examples include metals, plastics, polymer matrix composites, metal matrix composites, plastics and rubbers. The proper selection of an abrasive blade requires an understanding of the relationship between the abrasive particle, abrasive bonding and the specimen properties.

Abrasive Type - Today's high performance abrasive blades use alumina or silicon carbide abrasives. Alumina is a moderately hard and relatively tough abrasive which makes it ideal for cutting ferrous metals. Silicon carbide is a very hard abrasive which fractures and cleaves very easily. Thus, silicon carbide is a self-sharpening abrasive and is more commonly used for cutting nonferrous metals.

Bonding Material - The hardness and wear characteristics of the sample determine which resin system is best-suited for abrasive cutting. In general, the optimum bonding material is one that breaks down at the same rate as the abrasive dulls; thus, exposing new abrasives for the most efficient and effective cutting operation.

2.3 RECOMMENDED CUTTING PROCEDURES

- Select the appropriate abrasive blade.
- Secure specimen. Improper clamping may result in blade and/or specimen damage.
- Check coolant level and replace when low or excessively dirty. ***Note*** abrasive blades break down during cutting and thus produce a significant amount of debris.
- Allow the abrasive blade to reach its operating speed before beginning the cut.
- A steady force or light pulsing action will produce the best cuts and minimize blade wear characteristics, as well as maintain sample integrity (no burning).
- When sectioning materials with coatings, orient the specimen so that the blade is cutting into the coating and exiting out of the base material, thereby keeping the coating in compression.

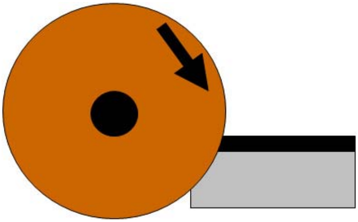


Figure 2-3 For coated samples, maintain the coating in compression when sectioning.

2.4 CUTTING FLUIDS

Lubrication and swarf removal during abrasive cutting and diamond wafer cutting are required in order to minimize damage to the specimen. For some older abrasive cutters, the proper cutting fluid can also have the added benefit of coating cast iron bases and the fixtures in order to reduce or eliminate corrosion.

TIP: Most metallographic abrasive cutters have a hood, which can produce a corrosive humidity chamber when not in use. In order to reduce these corrosive effects, keep the hood open when not in use.

Abrasive Cutting Fluid - The ideal cutting fluid for abrasive cutting is one that removes the cutting swarf and degraded abrasive blade material. It should have a relatively high flash point because of the sparks produced during abrasive sectioning.

Figure 2-4 Abrasive Cut-off Lubricants and Cleaning Agents.

2.5 ABRASIVE SECTIONING TROUBLESHOOTING

The most common problems with abrasive cutting include broken abrasive blades and cracked or burnt samples.

TABLE II. Troubleshooting Guidelines for Abrasive Cutting

Symptoms	Cause	Action
Chipped or broken blade	-Sample moved during cut -Cutting force too high	-Secure sample properly - Reduce cutting force
Bluish burnt color on specimen	-Incorrect cutting fluid -Improper blade or excessive force	-Use proper cutting fluid -Consult applications guideline or use a blade with a softer resin

Figure 2-5 MEGA-M250 Manual Abrasive Saw.

CHAPTER 3
Precision Wafer Sectioning

3.0 PRECISION WAFER SECTIONING

Precision wafer cutting is used for sectioning very delicate samples or for sectioning a sample to a very precise location. Precision wafering saws typically have micrometers for precise alignment and positioning of the sample, and have variable loading and cutting speed control (see Figure 3-1).

Figure 3-1 PICO 150 Precision Wafering Saw.

3.1 WAFERING BLADE CHARACTERISTICS

In order to minimize cutting damage, precision wafer cutting most frequently uses diamond wafering blades, however, for some materials the use of cubic boron nitride (CBN) is more efficient. In addition, optimal wafer cutting is accomplished by maximizing the abrasive concentration and abrasive size, as well as choosing the most appropriate cutting speed and load. Table III provides some general guidelines and parameters for precision sectioning a variety of materials.

The particle size of fine grit diamond blades is 10-20 microns, or approximately 600 grit. For medium grit diamond wafering blades, the particle size is 60-70 micron, or 220 grit. For these types of wafering blades, the abrasive is mixed with a metal binder and then pressed under high pressure (Figure 3-2). As will be discussed in the next section, periodic dressing/conditioning of the metal pressed blades is required for optimum cutting performance of the blade.

Figure 3-2 Metal pressed diamond and CBN wafering blades.

TABLE III. Precision Cutting Blade Specifications

Wafering Blade Description	Characteristic
Fine grit	10-20 micron (600 grit)
Medium grit	60-70 micron (220 grit)
Coarse grit	120 micron (120 grit)
High concentration	100%
Low concentration	50%

In some cases, precision cutting requires a coarser grit wafering blade. Usually the coarsest standard blade uses 120 grit abrasive particles. For metallographic applications, coarse abrasives are mostly associated with electroplated blades (Figure 3-3a). The main characteristic of coarse electroplated blades is that the abrasive has a much higher, or rougher, profile. The advantage of this higher profile is that the blade does not “gum up” when cutting softer materials such as bone, plastics and rubbery materials.

Although less common, thin resin-rubber abrasive blades can be used for cutting on precision wafering saws (Figure 3-3b). For cutting with abrasive blades on precision wafer saws, set the speed of the saw to at least 3500 rpm. Note that abrasive blades create significantly more debris which requires changing out of the cutting fluid more frequently.

Figure 3-3 (a) Electroplated diamond wafering blade for cutting soft materials (left) and (b) alumina resin-rubber blade (right).

Perhaps the most important parameter for precision sectioning is the abrasive size. Similar to grinding and polishing, finer abrasives produce less damage. For extremely brittle materials, finer abrasives are required to minimize and manage the damage produced during sectioning. Sectioning with a fine abrasive wafering blade is often the only way that a specimen can be cut so that the final polished specimen represents the true microstructure. Examples include: silicon computer chips, gallium arsenide, brittle glasses, ceramic composites, and boron-graphite composites. Figures 3-4a and 3-4b compare the effects of cutting with a fine grit blade vs. a standard medium grit blade for sectioning a boron graphite golf shaft. As can be seen, the fine grit blade produces significantly less damage to the boron fibers.

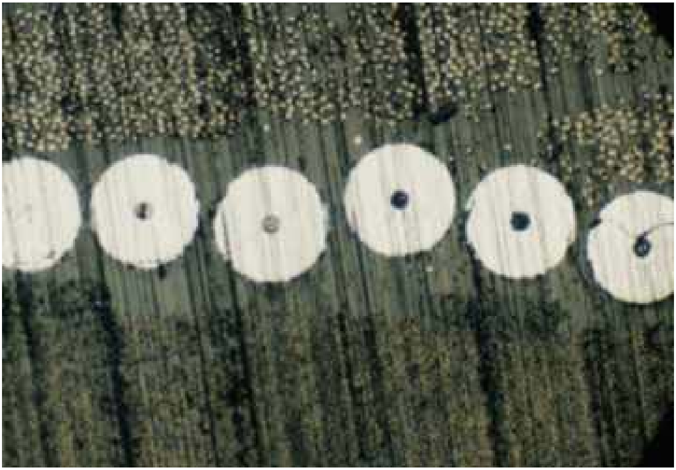


Figure 3-4a Fine grit diamond cut for boron graphite composite.

Figure 3-4b Medium grit diamond cut for boron graphite composite.

The second most important blade characteristic is the abrasive concentration because it directly affects the load which is applied during cutting. For example, brittle materials such as ceramics require higher effective loads to efficiently section; whereas, ductile materials such as metals require a higher abrasive concentration in order to have more cutting points. The result is that low concentration blades are recommended for sectioning hard brittle materials such as ceramics and high concentration blades are recommended for ductile materials containing a large fraction of metal or plastic.

TIP: Minimizing the amount of damage created during sectioning can significantly reduce the amount of time required for grinding and polishing.

The wafering blade bonding matrix can also significantly affect a blade's cutting performance. Metal pressed wafering blades require periodic dressing in order to maintain performance. A common misconception is that the cutting rates for these blades decrease because the diamond or abrasive is being "pulled out" of the blade. In reality, the metal bond is primarily smearing over the abrasive and "blinding" the cutting edge of the abrasive. With periodic dressing, using a ceramic abrasive encased in a relatively soft matrix (Figure 3-5), this smeared material is removed and the cutting rate restored. Figure 3-6 shows the effect of dressing a standard grit, low concentration diamond blade for cutting a very hard material such as silicon nitride. Without dressing the blade, the cut rate significantly decreases after each subsequent cut. After dressing the blade, the sample once again cuts like a new blade. Note it is highly recommended that a dressing fixture be used for conditioning or dressing the wafering blades in order to reduce the risk of breaking or chipping the wafering blades (Figure 3-7). Blade dressing is also accomplished at low speeds (<300 rpm) and at light loads (<200 grams).

Figure 3-5 Alumina wafer blade dressing sticks.

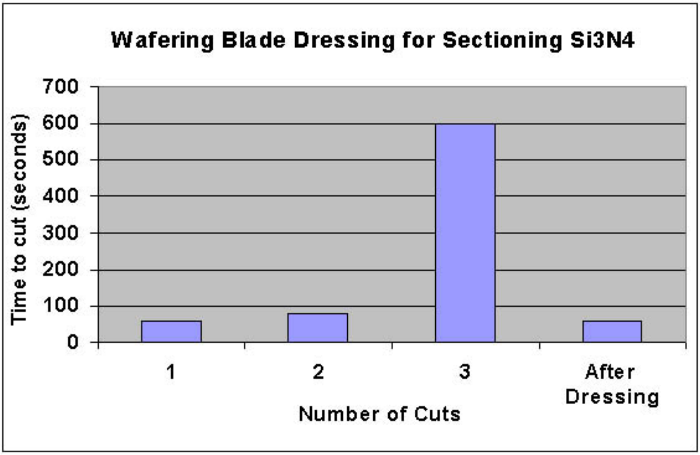


Figure 3-6 Cutting performance vs. wafering blade conditioning.

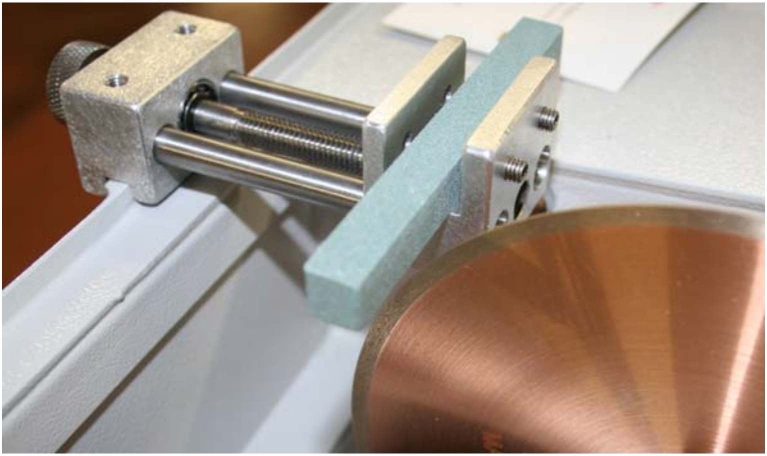


Figure 3-7 Proper dressing fixturing will minimize damage to the wafering blade.

Table IV provides some recommended guidelines for sectioning a variety of materials ranging from very brittle to very hard and tough.

TABLE IV. Guidelines for Wafering Cutting Various Materials

Material	**Characteristic**	**Speed (rpm)**	**Load (grams)**	**Blade (grit/conc.)**
Silicon substrate	soft / brittle	<300	<100	Fine / low
Gallium arsenide	soft / brittle	<200	<100	Fine / low
Boron composites	very brittle	500	250	Fine / low
Ceramic fiber composites	very brittle	1000	500	Fine / low
Glasses	brittle	1000	500	Fine / low
Minerals	friable / brittle	>1500	>500	Medium / low
Alumina ceramic	hard / tough	>1500	>500	Medium / low
Zirconia (PSZ)	hard / tough	>1500	>800	Medium / low
Silicon nitride	hard / tough	>3500	>800	Medium / low
Metal matrix composites		>3500	>500	Medium / high
General purpose		variable	variable	Medium / high

3.2 CUTTING PARAMETERS

Most wafer cutting is done at speeds between 50 rpm and 5000 rpm with loads varying from 10-1000 grams. Generally, harder specimens are cut at higher loads and speeds (e.g. ceramics and minerals) and more brittle specimens are cut at lower loads and speeds (e.g. electronic silicon substrates) (see Table IV). It is interesting to note that the cutting efficiency for sectioning hard/tough ceramics improves at higher speeds and higher loads. Figure 3.8 compares the resulting surface finish for sectioning partially stabilized zirconia at a low speed/low load (Figure 3-8a) vs. cutting at a higher load/higher speed (Figure 3-8b). As can be seen, partially stabilized zirconia has less fracturing and grain pull out after sectioning at higher speeds and loads. This observation may seem counter intuitive, however for sectioning hard/tough ceramics, high cutting speeds and loads result in producing a crack that propagates in the direction of the cut instead of laterally into the specimen.

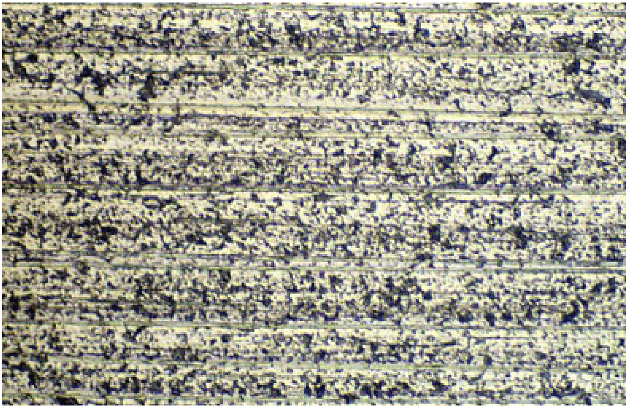


Figure 3-8a Partially stabilized zirconia sectioned at low speeds and low loads.

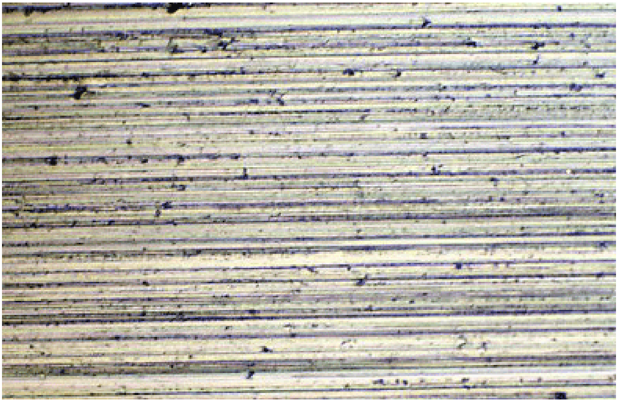


Figure 3-8b Partially stabilized zirconia sectioned at high speeds and high loads.

For wafer cutting it is recommended that a cutting fluid be used. The characteristics of a good cutting fluid include:

- Removes and suspends the cutting swarf
- Lubricates the blade and sample
- Reduces corrosion of the sample, blade and cutting machine parts

In general, cutting fluids are either water-based or oil-based (Figure 3-9). Water-based cutting fluids are the most common because they are easier to clean; whereas, oil-based cutting fluids typically provide more lubrication.

Figure 3-9 Oil and water-based cutting fluids.

3.3 RECOMMENDED WAFER CUTTING PROCEDURES

- Prior to cutting the sample, condition or dress the wafering blade with the appropriate dressing stick.
- Clamp the specimen sufficiently so that the sample does not shift during cutting. If appropriate, clamp both sides of the specimen in order to eliminate the cutting burr which can form at the end of the cut.
- For brittle materials clamp the specimen with a rubber pad to absorb vibration from the cutting operation.
- Begin the cut with a lower force in order to set the blade cutting kerf.
- Orient the specimen so that it is cut through the smallest cross section.
- For samples with coatings, keep the coatings in compression by sectioning through the coating and into the substrate material.
- Use largest appropriate blade flanges to prevent the blade from wobbling or flexing during cutting.
- Reduce the force toward the end of the cut for brittle specimens
- Use the appropriate cutting fluid.

3.4 TROUBLESHOOTING GUIDELINES

TABLE V. Troubleshooting Guidelines for Wafering Cutting

Symptoms	Cause	Action
Chipped or broken blade	-Improper blade dressing -Insufficient sample clamping -Cutting force initially too high	-Use mechanical dressing fixture -Secure specimen with rubber pad -Reduce initial force to set cutting kerf
Excessive blade wobble	-Cutting force to high	-Reduce applied force and/or use larger diameter support flanges
Low cutting rates	-Smeared material on the blade -Cutting speed and/or force is too low	-Redress blade at <200 grams and <300 rpm -Increase cutting speeds and applied force -Rotate specimen to minimize cutting area
Excessive specimen damage or chipping	-Too large an abrasive -Excessive vibration	-Use finer grit diamond blade -Secure specimen with rubber mounting pad
Burr formation on specimen at the end of the cut	-Cutting force or speed too high at the end of the cut -Excessive vibration	-Reduce speed and cutting force to reduce cutting rate -Secure sample with rubber mounting pads

CHAPTER 4
Specimen Mounting

4.0 SPECIMEN MOUNTING

The primary reasons for specimen mounting are to better hold the part to be ground and polished, and to provide protection to the edges of the specimen. Secondarily, mounted specimens are easier to fixture into automated machines or to hold manually. The orientation of the specimen can also be more easily controlled by fixturing it and then setting it in place via mounting. Metallographic mounting is accomplished by casting the specimen into a castable plastic material or by compression mounting the plastic under pressure and temperature.

4.1 CASTABLE MOUNTING

Castable resins are monomer resins which utilize a catalyst or hardener for polymerization. Polymerization results in cross-linking of the polymer to form a relatively hard mount. Castable resins also have the advantage of simultaneously mounting multiple samples at one time for increased throughput. A number of resin systems (Figure 4-1) are used for metallographic mounting and include:

- Epoxy resins
- Acrylic (castable) resins
- Polyester (clear) resins

Figure 4-1 (a) 2-part liquid epoxy resins (b) 1-part powder, 1-part liquid castable acrylic resins (c) 2-part liquid polyester resins.

Table VI lists the common properties of epoxy, acrylic and polyester resins.

TABLE VI. Castable Mounting Properties

PROPERTY	EPOXY	ACRYLIC	POLYESTER RESIN
Peak Temperature	100-375°F (38-190°C)	150°F (65-70°C)	100°F (35-40°C)
Shore D Hardness	82	80	76
Cure Time	30 minutes to 8 hours	5-8 minutes	6-8 hours
Comments	Moderate hardness, low shrinkage, transparent	Very fast cure, translucent, some shrinkage	Transparent, water clear

4.1.1 Epoxy Resins

The most common and best performing castable resins are epoxy based (Figure 4-2). Epoxy resins are typically two-part systems consisting of a resin and a catalyst (hardener). Mixing ratio's vary from ten-parts resin with one-part hardener to five-parts resin with one-part by weight of hardener. The advantages of mounting with epoxy resins include:

- Low shrinkage
- Relatively clear
- Relatively low exotherms
- Excellent adhesion
- Excellent chemical resistance
- Good hardness
- Relatively inexpensive

Figure 4-2 Epoxy resins are available in standard, low viscosity, or fast curing systems.

Epoxy curing times are dependent upon a number of variables including:
- Volume of mounting resin (larger mounts cure faster).
- Thermal mass of specimen (larger specimens absorb heat and therefore require longer curing time).
- Specimen material properties.
- Initial resin temperature (higher temperatures cure faster).
- Ambient temperature (higher temperatures cure faster).
- Relative humidity and shelf life (absorption of water degrades resin and shortens shelf life).
- Mounting molds (plastic, phenolic rings and rubber absorb heat differently).

As a general rule, curing times can vary from 30 minutes to 2 hours for fast curing epoxies up to 24 hours for slower curing epoxies. For metallographic epoxies to grind properly, the hardness needs to be at least a Shore D80. Note that epoxy resins typically will continue to harden over a longer period of time (maximum hardness, Shore D90).

In some cases, the curing time and temperature may need to be controlled to compensate for the above variables. For example, an 8-hour resin system can be cured in 30-45 minutes by preheating the resin to approximately 120°F (50°C) prior to mixing and then curing at room temperature. This procedure initiates the catalytic reaction sooner; however, this may also increase the maximum exotherm temperature.

TIP: Preheat the specimen to initiate the epoxy resin curing at the surface of the mount and thus have the epoxy shrink towards the sample for better edge retention.

Conversely, the resin curing cycle can be slowed or reduced by decreasing the curing temperature by forcing air over the curing mounts (fume hood or fan), placing the mounts into a water bath, or curing in a refrigerator. In these cases, care must be taken to not stop the reaction; however if this does occur or the resin is too soft after curing, heating it to 100-120°F for several hours should push the reaction to completion and the mount should be hard after cooling to room temperature.

Table VII lists the relative properties for several metallographic epoxy resin systems.

TABLE VII. Castable Epoxy Mounting Properties

EPOXY	Standard Epoxy	Quick Mounting Epoxy	Low Viscosity Epoxy
Characteristics	Lower cost	Fast cure	Lower viscosity, clearer, lower exotherm, low shrinkage
Cure Time	2-6 hours	30 minutes - 2-hours	2-6 hours
Mixing ratio (weight) (resin : hardener)	5:1	10:1	10:1
Peak Exotherm 20 grams 30 grams	 300°F (150°C) 325°F (165°C)	 350°F (180°C) 375°F (190°C)	 120°F (50°C) 150°F (65°C)
Color	Clear to slight yellow tint	Slight yellow tint	Clear
Viscosity	Medium	Medium	Low
Shrinkage	Moderate	High	Low

4.1.2 Acrylic Castable Resins

Castable acrylics are easy to use and are very robust (Figure 4-3). The main advantage of mounting with castable acrylics is the fast curing time. Depending upon the mixing ratio, castable acrylic mounts are typically ready to use within 8-15 minutes. Also unlike epoxy resins, the ratio of the various acrylic parts (powder to liquid) can be altered by up to 25% with no adverse effect to the final properties of the mount. This is because both the liquid and powder are acrylics with various additives and curing agents. By varying the ratio of the liquid to powder, the curing time and viscosity can be altered.

Note: the powder contains a catalyst that reacts with the liquid hardener to start the curing process. Fillers are added to increase hardness and to reduce shrinkage.

Figure 4-3 Castable mounting acrylics include powder and liquid components.

Characteristics of Castable Acrylics (see Table VIII) includes:
- Rapid mounting
- Very repeatable and consistent mounts
- Moderate shrinkage
- Good hardness
- Semi-transparent
- High odor

TIP: Acrylics can be submerged into a water bath during curing. This reduces the exotherm heat and thus reduces the shrinkage of the mount at the specimen interface. A secondary advantage is that the water absorbs the odor.

TABLE VIII. Castable Acrylic Mounting Properties

ACRYLICS	CASTAMOUNT	PREMIUM	SUPERMOUNT
Characteristics	Fast, semi-clear, low shrinkage	Fast, clear, low shrinkage	Fast, hard, opaque
Cure Time	10-15 minutes	8-12 minutes	10-15 minutes
Mixing ratio (volume) Powder: Liquid	3:2	3:2	3:2
Color	Semi-clear	Semi-clear	Gray to black

4.1.3 Polyester Castable Resins

Polyesters are typically used when a very clear mount is required. Polyester resins are also useful for mounting parts for display. In this case, the part appears suspended in the plastic. The procedure for molding samples for display is to first determine the mixing ratio of the resin to hardener (catalyst). This ratio is variable depending upon the mass of the casting (Table IX).

TABLE IX. Polyester Catalyst Mixing Ratios
(Proportions are based on ambient and resin temperature of 70°F (22°C))

Single Layer Casting (metallographic)		**Multiple Layer Casting (display)**	
Layer Thickness	Drops of catalyst per ounce of resin	Layer	Drops of catalyst per ounce of resin
1/8"	15	1st layer	5
1/4"	8	2nd layer	4
1/2"	6	3rd layer	3
3/4"	5	4th layer	2
1" - 1-1/2"	4	5th layer or more	2

For larger volumes, the amount of hardener needs to be reduced significantly. The procedure for suspending the sample in the mount is to pour an initial layer and allow it to pot or gel (do not let it fully cure). The object or specimen is then placed on the initial rubbery polyester layer and another layer of the liquid polyester is poured. Multiple layers can be poured in this fashion if required.

Characteristics of Polyester include:
- Very clear (water clear)
- High odor
- Best resin system for making large castings

Polyester resins are similar to acrylics and can be submerged into water during the curing cycle in order to reduce the exotherm temperature and shrinkage.

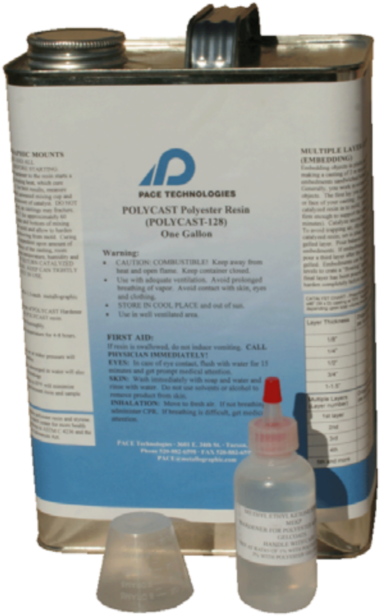


Figure 4-4 Clear castable mounting polyesters.

4.2 CASTABLE MOUNTING PROCEDURES

- Clean and thoroughly dry specimens to remove cutting and handling residues.
- Remove debris from molding cups.
- Apply thin coat of mold release compound to molding cup.
- Center specimen in molding cup.
- Accurately measure resin and hardener.
- Mix thoroughly (gentle mixing to avoid producing excessive air bubbles).
- To reduce air bubbles, pull a vacuum on the specimen before pouring the resin. After pouring the resin over the specimen, cure at room pressure or apply pressure in an autoclave chamber.

TIP: Before mixing, preheat resin, hardener and specimen to 85°F (30°C) to expedite curing cycle

Note: this will also increase maximum exotherm temperature.

4.2.1 Vacuum/Pressure Mounting

Vacuum impregnation is a very useful technique used to fill in pores or voids prior to specimen preparation. It is highly useful for thermal spray coatings and other porous samples.

The most effective technique is to pour the resin under vacuum and/or apply pressure during the curing cycle (advantages - better infiltration of pores and cracks, more transparent mounts, and fewer air bubbles) (see Figure 4-5).

Figure 4-5 Vacuum impregnation device.

For porous or cracked specimens, the resin can aid in supporting these features. Filling these voids can be difficult depending upon their size, with the smaller voids being much more difficult to impregnate than larger voids. This arises mainly because of the compressibility and volume of air within the void. By applying a vacuum to the specimen and pouring while under vacuum the total pressure of this air can be reduced significantly. Subsequent curing at increased pressures will force (or push) the resin into the voids. Note that the vacuum time on both the resin and specimen should be kept to a minimum in order to minimize degassing of the resin.

$PV = nRT$ (gas law)
P - Pressure
V - Volume
T - Temperature

$$V(\text{bubble size}) = \frac{nRT}{P}$$

Thus in order to decrease the air bubble size, impregnate at low pressures and cure at higher pressures.

Recommended Procedure:

1. Place mold and sample into impregnation chamber
2. Mix castable mounting resin
3. Place cover on chamber and pull vacuum
4. Pour resin into mount
5. Slowly increase the pressure
6. Allow the mount to cure at room pressure or apply an external pressure.

TIP: Do not pull vacuum for more than 60 seconds. Extended vacuum causes the dissolved gases in the liquid resin to degass and bubble (similar to opening up a carbonated beverage bottle).

TIP: To reduce the curing time, preheat resin, hardener and specimen to 85°F (30°C).

Note: this will also increase maximum exotherm.

TIP: Slight preheating of the epoxy will also reduce the viscosity of the resin and allow it to flow better.

4.3 CASTABLE MOUNTING MISCELLANEOUS

Figures 4-6 to 4-8 show a variety of accessories used with castable mounting, ranging from mounting molds and mounting clips to mixing cups and storage containers. Table X provides a description of each..

Figure 4-6 Castable mounting molds
(clockwise: silicon rubber, 2-piece plastic, ring forms, disposable).

Figure 4-7 Castable mounting clips (plastic, metal).

Figure 4-8 Plastic mixing cups, measuring cups and storage containers.

TABLE X. Castable Mounting Accessories

ACCESSORY	**APPLICATION**
Silicon rubber molds	Reusable molds
2-piece Reusable molds	Reusable molds
Mounting cups	Disposable mounting molds
Silicon mold release	To aid in releasing the mount from the mold
Plastic clips	Used for holding or orienting thin specimens perpendicular to the examination plane
Metal clips	Used for holding or orienting thin specimens perpendicular to the examination plane
Plastic mixing cups	For mixing acrylic resins which absorb into paper cups
Measuring cups	For measuring the volume of the castable mounting material
Storage cups	To protect and archive the specimens

4.4 CASTABLE MOUNTING TROUBLESHOOTING

In general, acrylics are the easiest and most robust castable mounting materials to use. Epoxies are very useful; however, complete mixing and the proper resin-to-hardener ratio is very important. Polyesters, especially for larger casting, may require some trial and error testing prior to mounting one-of-a-kind samples.

TABLE XI. Castable Mounting Troubleshooting

SYMPTOM	CAUSE	ACTION
Lack of or partial curing of resin	Improper or insufficient mixing	-Remount taking care to sufficiently mix epoxy resin and hardener -Check expiration date on catalyst (typically 1-year life)
Soft or gummy resins (grinding produces a matted finish)	Insufficient curing of resin – primarily by too low an exotherm	-Heat mount in an oven at 90-100°F (30-40°C) for 1-2 hours and let cool. -Resin should harden upon cooling
Bubbling, cracking, or yellowing of resin	Exotherm too high	-Mount at room temperatures below 85°F (30°C) -Decrease volume or volume percentage of hardener
Curing time takes too long	Improper resin mixture, old hardener, or mounting temperature to cold	-Replace old hardener -Mount at room temperature 70-80°F (30°C) -Preheat resin, but cure at room temperature
Bubbles in resin	Improper mixing or degassing of specimen	-Mix with a slow folding motion -Pour resin under vacuum and/or cure under higher pressures -Clean specimen prior to mounting

4.5 COMPRESSION MOUNTING

Compression mounting is a very useful mounting technique which can provide better specimen edge retention compared to castable mounting resins. Compression mounting resins are available in different colors and with various fillers to improve hardness or conductivity (Figure 4-9).

Several compression mounting characteristics include:

- Convenient means to hold the specimen
- Provides a standard format to mount multiple specimens
- Protects edges
- Provides proper specimen orientation
- Provides the ability to label and store the specimens

Figure 4-9 Compression Mounting Compounds.

Compression mounts are quick and easy to produce, requiring several minutes to cure at the appropriate mounting temperature. Most of the time required occurs during the heating and cooling cycles. When choosing a compression mounting machine, the most important features include its maximum heating temperature and how intimately the heater and water cooler are connected to the mold assembly. The better compression mounting machines have heaters which can reach temperatures of at least 250-300°C (480-575°F). For faster turn around time, water cooling is essential (see Figure 4-10).

Figure 4-10 Compression Mounting Press.

The primary compression mounting resins include:

- Phenolic Resins (standard colors are black, red and green) (see Figure 4-11)
- Acrylic Resins (clear)
- Diallyl Phthalate Resins (blue and black) (Figure 4-12)
- Epoxy Resins (glass-filled) (Figure 4-13)
- Conductive Resins (phenolics with copper or graphite filler) (see Figure 4-14)

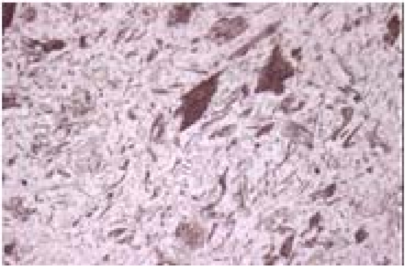


Figure 4-11 Red phenolic resin, 100X.

Figure 4-12 Glass-filled diallyl phthalate resin, 100X.

Figure 4-13 Glass-filled epoxy resin, 100X.

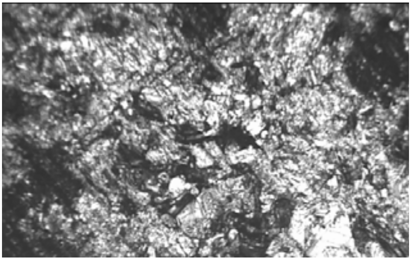


Figure 4-14 Conductive graphite mount, 100X.

4.6 COMPRESSION MOUNTING RESIN PROPERTIES

There are a variety of compression mounting materials. The two main classes of compression mounting materials are thermoset and thermoplastics. Thermoset resins require heat and pressure to cross-link the polymer and the reaction is irreversible. Thermoplastic, on the other hand, can theoretically be remelted. Table XIIa provides a relative comparison of the most common compression mounting resins, and Table XIIb provides more specific information for the various compression mounting resins.

TIP: Compression mounting at higher then the recommended minimum temperature generally improves the properties of the mount.

TIP: A useful tip for marking or identifying a specimen is to mold the label inside of the mount (Figure 4-15). If the entire mount is an acrylic, just place the label on top the mount and cover it with a little acrylic powder. To label other compression mounting resins, add a thin layer of acrylic over the base mounting material and then position the label on this layer. Finish off the mount with another layer of acrylic.

Figure 4-15. Example of labeling mounts using acrylic resin on top of a phenolic base.

TABLE XIIa. Compression Mounting Resin Characteristics

	PHENOLICS	**ACRYLICS**	**EPOXY (Glass-filled)**	**DIALLYL PHTHALATES**
Type	Thermoset	Thermoplastic	Thermoset	Thermoset
Cost	Low	Moderate	Moderate	Moderate
Ease of use	Excellent	Moderate	Good	Good
Availability of Colors	Yes	No	No	Yes
Cycle times	Excellent	Moderate	Good	Good
Edge retention	Fair	Good	Excellent	Excellent
Clarity	None	Excellent	None	None
Hardness	Low	Medium	High	High

TABLE XIIb. Compression Mounting Resin Characteristics

	PHENOLICS	**ACRYLICS**	**EPOXY (Glass-filled)**	**DIALLYL PHTHALATES**
Form	Granular	Powder	Granular	Granular
Specific gravity (gm/cm^3)	1.4	0.95	1.75-2.05	1.7-1.9
Colors	Black, Red, Green	Clear	Black	Blue, Black
Shrinkage (compression) (in/in)	0.006	N/A	0.001-0.003	0.001-0.003
Coefficient of Linear Thermal Expansion (in/in/°C x 10^{-6})	50	N/A	28	19
Chemical resistance	Glycol, petrochemicals, solvents, some acids and bases	Alcohol, dilute acids & alkalies, and oxidizers	Solvents, acids, alkalies	Solvents, acids, alkalies
Molding Temperature	150°-200°C (300°-420°F)	185°-200°C (350-420°F)	143°-200°C (290°4250°F)	160°-200°C (320°-420°F)
Hardness	N/A	Rockwell M63	Barcol 72	N/A
Min. curing time (1/2" mount @ temperature and pressure)	3-5 minutes	5-7 minutes	5 minutes	5 minutes

4.6.1 Phenolics

In general, phenolics are used because of their relatively low cost. In addition, phenolics are available in a variety of colors (Figure 4-16).

Figure 4-16 Phenolic resins are available in a variety of colors.

TIP: Use different color phenolics to color code jobs, specimen types, or for different testing dates. For example, changing the phenolic color each month will show which samples or jobs are getting old.

TIP: If the color dye in the mount bleeds out when rinsing with an alcohol, this is an indication that the mount was not cured either at a high enough temperature or for the proper length of time (see Figure 4-17).

Figure 4-17 Insufficient curing of phenolic resin causes alcohol to dissolve the dye.

4.6.2 Acrylics

The main application for compression mounting acrylics is for their excellent clarity. This is particularly important for locating a specific feature within the specimen mount (Figure 4-18).

Figure 4-18 Acrylic resins produce very clear mounts.

TIP: A common problem, know as the "cotton ball" effect, can occur with thermoplastic resins if they are not heated and held for a sufficiently long enough time to completely melt the plastic. For acrylic resins, the unmelted resin takes the appearance of a cotton ball in the middle of the mount. To correct this problem, simply put the mount back into the mounting press and either increase the time or temperature of the press. Eventually this will eliminate the "cotton ball" (see Figure 4-19).

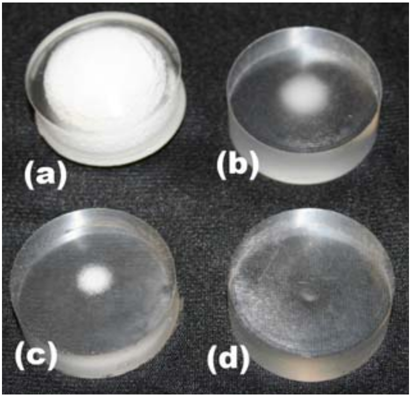


Figure 4-19 Acrylic "cotton ball" defects from insufficient temperature, pressure and cure time.
(a) 150°C, 3 minutes, water cooled 2 minutes
(b) 150°C, 3 minutes, water cooled 3 minutes
(c) 170°C, 4 minutes, water cooled 3 minutes
(d) 200°C, 5 minutes, water cooled 3 minutes

4.6.3 Epoxies / Diallyl Phthalates

Glass-filled epoxies and diallyl phthalates are compression mounting resins used to provide a harder mounting support edge next to the specimen (see Figure 4-20). These resins are commonly used to support the edges of coatings, heat treated samples and other specimens requiring better flatness. Figure 4-21 shows the polished interface between a glass-filled epoxy and tungsten carbide specimen. Note that there is no noticeable gap between the specimen and the mounting material, therefore showing that glass-filled epoxies provide excellent support to the specimen edge even for extremely hard specimens.

Figure 4-20 Glass-filled diallyl phthalate and epoxies have a glass filler to provide better edge retention during grinding and polishing.

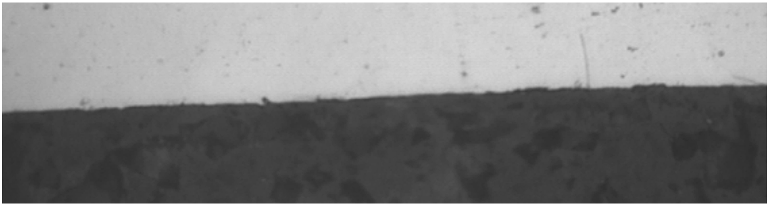


Figure 4-21 Polished edge for tungsten carbide mounted in glass-filled epoxy.

TIP: Epoxies (glass-filled) and diallyl phthalates are significantly more expensive then phenolic and acrylics. In order to reduce the cost of these mounts, they can be layered with a lower cost mounting compound such as a phenolic. The technique is to place a sufficiently thick enough layer of the glass-filled epoxy or diallyl phthalate around the specimen in order to compensate for any grinding loss. The rest of the mount can then be supported with a lower cost compression mounting compound such as a phenolic. Red phenolics are used frequently for this technique (Figure 4-22).

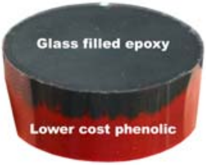


Figure 4-22 Glass-filled epoxy laminated with phenolic to reduce the cost of the mount.

4.6.4 Specialized Compression Mounting Resins

With the addition of fillers such as graphite or copper, the compression mounting compounds can be made conductive (Figure 4-23). Conductive mounts are used in scanning electron microscopes (SEM) to prevent the specimen from building up a charge. Conductive mounts are also used for specimens requiring electrolytic etching or polishing.

Figure 4-23 Graphite and copper are common fillers used to increase the conductivity of compression mounting resins.

4.7 COMPRESSION MOUNTING PROCEDURES

- Clean specimens to remove cutting and handling residues
- Remove debris from mold assembly
- Apply thin coat of mold release compound to mold assembly

- Raise mold ram to up position
- Center specimen on ram
- Lower ram assembly
- Pour predetermined amount of resin into mold
- Clean and remove any excess resin from around the mold assembly threads
- Lock mold assembly cover
- Slowly raise ram into up position
- Apply recommended heat and maintain pressure for specified period of time
- Cool to near room temperature
- Remove mounted specimen
- Clean mold and ram assembly

TIP: Preheat resin and sample to 95°F (35°C) to expedite the initial heating process and for increasing throughput.

4.8 COMPRESSION MOUNTING TROUBLESHOOTING

The most common problems associated with compression mounting typically relate to rushing the curing or cooling cycle (Table XIII).

TABLE XIII. Compression Mounting Troubleshooting

Symptoms	Cause	Action
Large bubbles in acrylic resins	Insufficient mounting pressure	-Increase mounting pressure or reduce temperature
Soft surfaces on mounts	Mount did not completely polymerize because of polymer incompatibility with mold release or oil on the specimen surface	-Clean specimen and mounting machine to remove incompatible contamination. -Use a compatible mold release
Voids or cracks	High internal stress due to overly rapid cooling.	-Allow mounts to cool slower and longer
Haze around specimen (acrylic mounts)	Specimen contains moisture Specimens contain copper or some other polymerization retrading alloy	-Use a desicaator or low temperature oven to dry specimens -Coat specimens with an appropriate lacquer before mounting
Phenolic dye leaching out with alcohol rinsing	Insufficient mounting temperature	-Increase mounting temperature or service check the heating element
Distortion or cracking of specimen	Autoclave pressure is too great for the specimen	-Reduce mounting pressure or use a castable epoxy resin

CHAPTER 5
Abrasive Grinding

5.0 ABRASIVE GRINDING

In most cases, the specimen surface and subsurface are damaged after cutting and sectioning. The depth or degree of damage is very dependent on how the material was cut. The purpose of abrasive grinding is to remove this damage and to restore the microstructural integrity of the specimen for accurate analysis. It is also important to realize that it is possible to create more damage in grinding than in sectioning. In other words, it is better to properly cut the sample as close as possible to the area of interest using the correct abrasive or wafering blades as opposed to grinding with very coarse abrasives. For metallographic specimen preparation, silicon carbide, zirconia, alumina and diamond are the most commonly used abrasives (Figure 5-1).

Figure 5-1 Common abrasive grinding papers.

Proper abrasive grinding is dependent to various degrees upon the following parameters:
- Abrasive type
- Abrasive bond
- Grinding speeds
- Grinding loads
- Lubrication

5.1 ABRASIVES USED FOR GRINDING

The following description offers a more detailed explanation of these abrasive grinding variables. Perhaps the most significant variable is the abrasive and how it interacts with the specimen. The properties of the more commonly used abrasives for metallographic cutting, grinding and polishing are shown in Table XIV.

TABLE XIV. Common Metallographic Abrasives

Abrasive	Hardness (Knoop-HK)	Hardness (Mohs)	Crystal Structure
Silica	820	6-7	Hexagonal -triagonal
Alumina	2150	8-9	Hexagonal-rhombohedral (alpha or gamma phases)
Silicon Carbide	2480	9.1-9.5	Hexagonal - rhombohedral
Boron Carbide	2750	9-10	Rhombohedral
Zircon	1500	7.5-8	Tetragonal
Diamond	8000	10	Cubic - hexagonal

5.1.1 Silicon Carbide

Silicon carbide (SiC) is a manufactured abrasive produced by a high temperature reaction between silica and carbon. It has a hexagonal-rhombohedral crystal structure and has a hardness of approximately 2500 HK. It is an ideal abrasive for cutting and grinding because of its high hardness and sharp edges. It is also somewhat brittle, and therefore it cleaves easily to produce sharp new edges (self-sharpening). SiC is an excellent abrasive for maximizing cutting rates while minimizing surface and subsurface damage. For metallographic preparation, SiC abrasives are used in abrasive blades and in coated abrasive grinding papers ranging from very coarse 60 grit to very fine 1200 (P4000) grit abrasive sizes.

Bonded or coated abrasive papers of SiC (Figure 5-2) are designed so that the abrasive will have a large number of cutting points (negative abrasive rank angle). This is achieved by aligning the abrasive particles approximately Normal to the backing. **Note:** coated abrasives are not quite coplanar, however SiC papers, produce excellent cut rates (stock removal) and produce minimal damage.

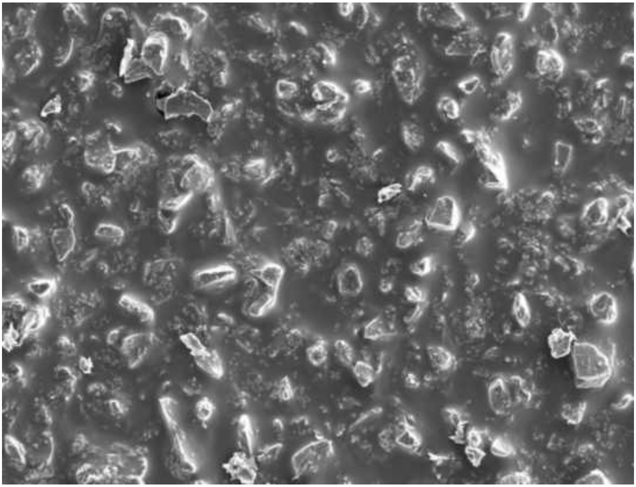


Figure 5-2 Coated SiC abrasive grinding paper.

Grinding with SiC grinding papers is the most common and repeatable process for obtaining consistent stock removal for rough grinding of metals. SiC abrasives are sized or classified by grit size, where the smaller grit number represents coarser abrasive sizes.

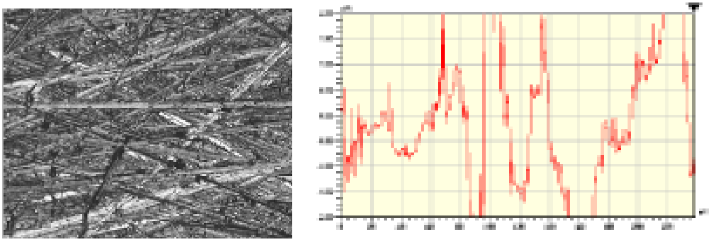
Also note that the European grading system is slightly different than the U.S. grading system. Simply put, both systems are related to the number of openings in a metal mesh screen. The primary difference is when the size of the openings approaches the size of the metal wire. For the European grading system, the size of the wire is not taken into account, whereas, the ANSI or U.S. grit size compensates for the wire size. Thus for the finer grit sizes, the European numbers can be significantly larger. Proper classification or identification of the European grading system should include the letter “P” in front of the grit number.

TABLE XV. Standard Abrasive Grading Comparisons

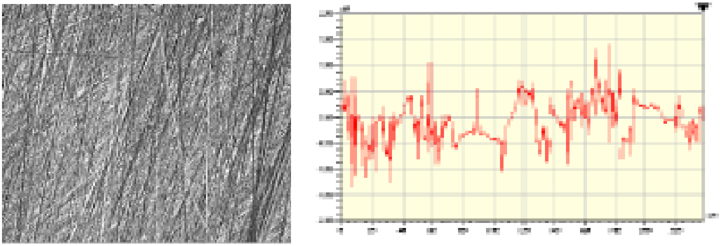
Standard grit size (ANSI) U.S. grading system	European P-grading convention	Medium Particle Diameter (microns)
60	60	250
120	120	106
180	180	75
240	P220	63
320	P360	40.5
360	P500	30.2
400	P800	21.8
600	P1200	15.3
800	P2400	6.5
1200	P4000	2.5

Figure 5-3 shows both the micrographs and 2D surface profiles for the surface roughness produced by grinding a medium hard steel with SiC grinding papers.

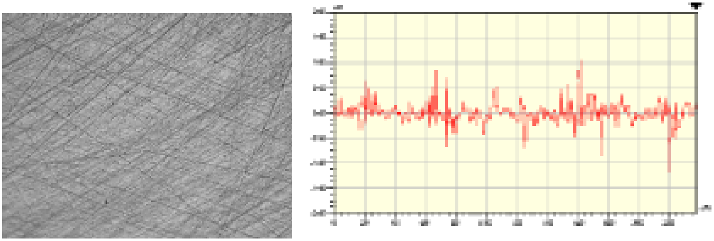
Figure 5.3 Surface roughness produced by SiC abrasive grinding of Rc 30 steel.



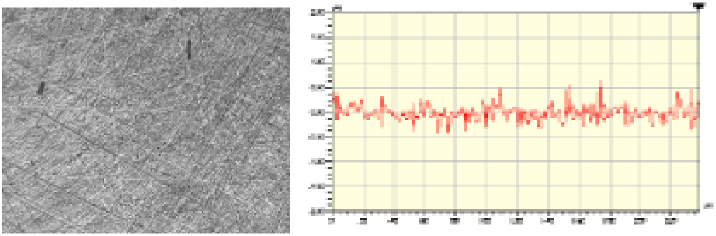
60 grit surface roughness micrograph and 2D line profile, 100X.



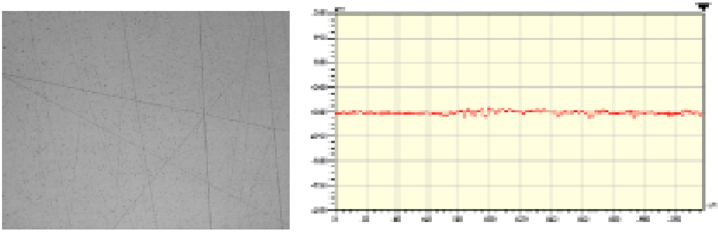
240 (P220) grit surface roughness micrograph and 2D line profile, 100X.



400 (P800) grit surface roughness micrograph and 2D line profile, 100X.



600 (P1200) grit surface roughness micrograph and 2D line profile, 100X.



1200 (P4000) grit surface roughness micrograph and 2D line profile, 100X.

TABLE XVI. Surface Roughness vs. SiC abrasive size

Silicon carbide (U.S. grit size)	80	240	400	600	1200
Rc-30 steel surface roughness (Ra-um)	1140	300	120	75	20

As can be seen in Table XVI, the surface roughness significantly decreases when grinding with finer silicon carbide papers. In particular, there is a large improvement in surface roughness using finer than 600 (P1200) grit grinding papers. It should be noted that the process used to manufacture metallographic papers ranging from 60 grit to 600 (P1200) grit is done by coating the abrasive on the grinding paper by a process known as electrostatic discharge. For electrostatic discharge, the abrasive is charged by passing it over a high-voltage wire. This process charges the abrasive particles and orients them so that the sharp edge of the abrasive is facing up. These charged abrasives are then coated onto a paper backed adhesive and cured in an oven.

For the finer abrasives ranging from 800 (P2400) to 1200 (P4000) grit, metallographic SiC abrasive papers are produced by a completely different manufacturing process. For these finer abrasive sizes, the manufacturing process is accomplished with a slurry coat process. For slurry coating, the abrasive is mixed into an epoxy binder to form a slurry. This slurry is then uniformly spread onto the paper backing using a knife blade. The resulting abrasive exposure is much lower for slurry coating than by electrostatic deposition. The result is that metallographic fine grit papers produce a much finer surface finish as compared to industrial or other commercially manufactured fine grit abrasive papers.

Grinding characteristics of silicon carbide abrasives

Grinding with SiC abrasives produces very repeatable and consistent results. In general, grinding papers are typically used once and thrown away, thus they do not change with time as is the case for alternative abrasive grinding surfaces such as diamond impregnated grinding surfaces.

The following figures show the effects of grinding with SiC and the effects that abrasive size, applied load, and grinding times have on the performance of SiC grinding papers.

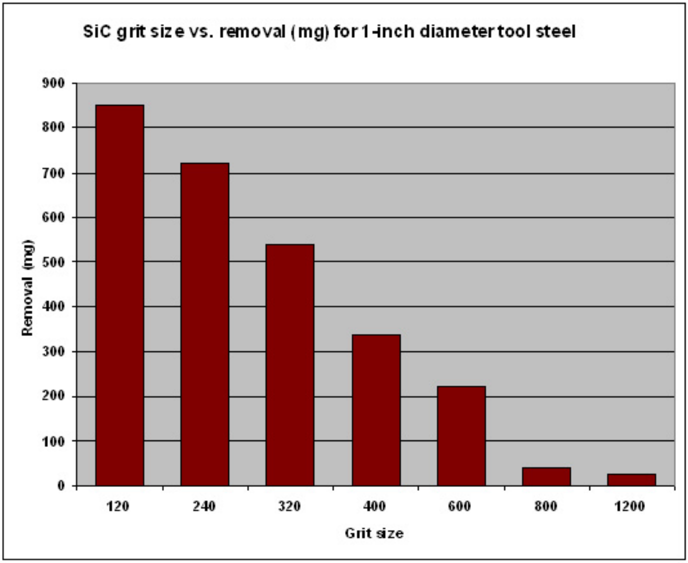


Figure 5-4 Abrasive removal vs. grit size for tool steel.

Figure 5-4 shows the effect that abrasive size has on stock removal for a 1-inch diameter tool steel specimen. It is not surprising that coarser silicon carbide abrasives remove more material; however, as previously noted, there is a significant drop in removal rates between 600 (P1200) grit and 800 (P2400) grit grinding papers. Charts such as these can also be used to determine how large a step can be made between grit sizes and still remove sufficient material in order to eliminate the damage from the previous step.

Figure 5-5 shows how the silicon carbide paper breaks down and loses its cut rate over time. For the tool steel specimen, removal rates drop to half within a couple of minutes. This chart also compares the results of grinding at 10 lbs per specimen vs. 5 lbs per specimen. Interestingly, at higher loads, the initial grinding rate is greater; however, after the initial minute of grinding, there is no advantage to grinding rates at higher forces.

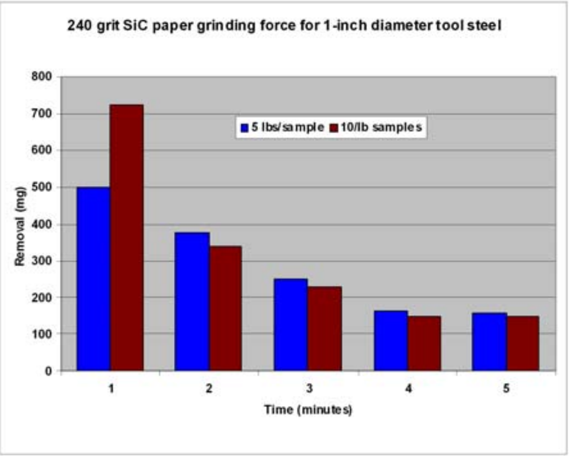


Figure 5-5 SiC paper wear vs. time for 240 (P220) grit SiC paper (Rc 30 steel).

Figure 5-6 illustrates the optimum removal for silicon carbide papers for a 1-inch diameter tool steel specimen at approximately 15 lbs force. Note that most procedures are written for forces of 5 lbs per sample. There are two reasons why lower forces are suggested: (a) the optimum load has not been previously studied and (b) many automated machines can only apply a maximum of 60-90 lbs force due to air compressor limitations.

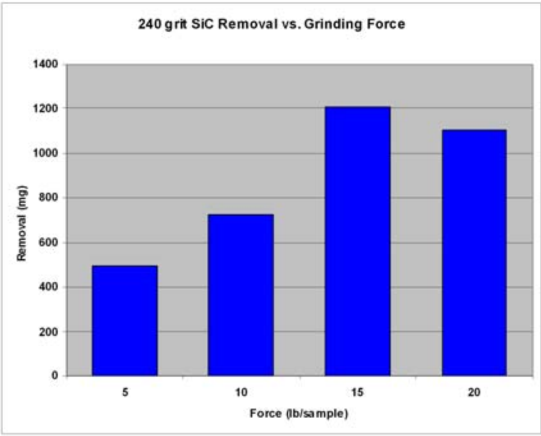


Figure 5-6 SiC abrasive removal vs. grinding force (tool steel).

5.1.2 Alumina

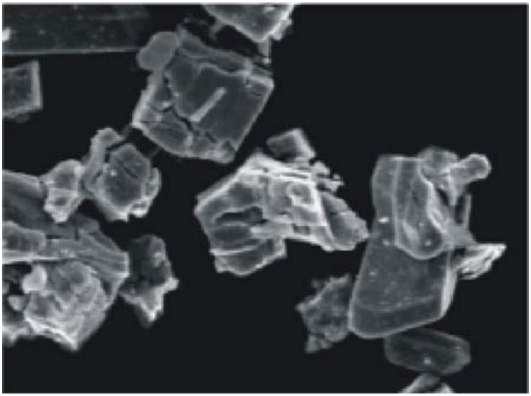


Figure 5-7 Calcined alumina abrasive.

Alumina is a naturally occurring mineral (Bauxite) (see Figure 5-7). It exists in either the softer gamma (Mohs 8) or harder alpha (Mohs 9) phase. Alumina abrasives are used primarily as final polishing abrasives because of their high hardness and durability. Unlike SiC abrasives, alumina is readily classified or sized to submicron or colloidal particles (< 1 micron).

Note that larger coated or bonded grit size papers of alumina are also commercially available. Alumina grinding papers are an excellent alternative to grinding with SiC abrasives, primarily because initial grinding can be obtained with a much finer abrasive. Therefore reducing the number of grinding steps. For example, for planarization of most metals one 600 (P1200) grit alumina paper can replace grinding with 240 (P220), 320 (P360), 400 (P800) and 600 (P1200) grit SiC papers. Alumina abrasives also do not fracture as easily as SiC so they produce less embedded abrasives in soft materials.

5.1.3 Diamond

Diamond is the hardest material known to man (Mohs 10, 8000 HV). It has a cubic crystal structure, and is available either as a natural or an artificial product. Although diamond would be ideal for coarse grinding, its price makes it a very cost-prohibitive coarse grinding material for anything except hard ceramics and glass (see Figure 5-8).

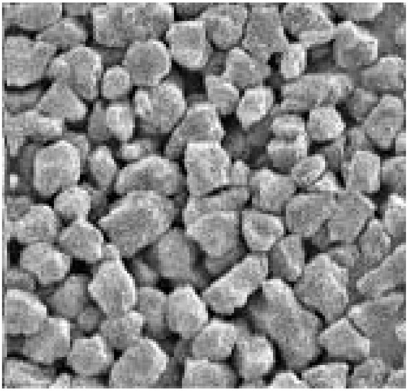


Figure 5-8 Blocky monocrystalline diamond.

For metallographic applications, both monocrystalline and polycrystalline diamond are used, however polycrystalline diamond has a number of advantages over monocrystalline diamond, especially for the finer micron sizes. These advantages include:

- Higher cutting rates
- Very uniform surface finish
- More uniform particle size distribution
- Higher removal rates (self-sharpening abrasives)
- Harder/tougher particles
- Blocky shaped
- Hexagonal microcrystallites (equally hard in all directions)
- Extremely rough surface (more cutting points)
- Surface area 300% greater than monocrystalline diamond
- No abrasion-resistant directionality (abrasion independent of particle orientation)

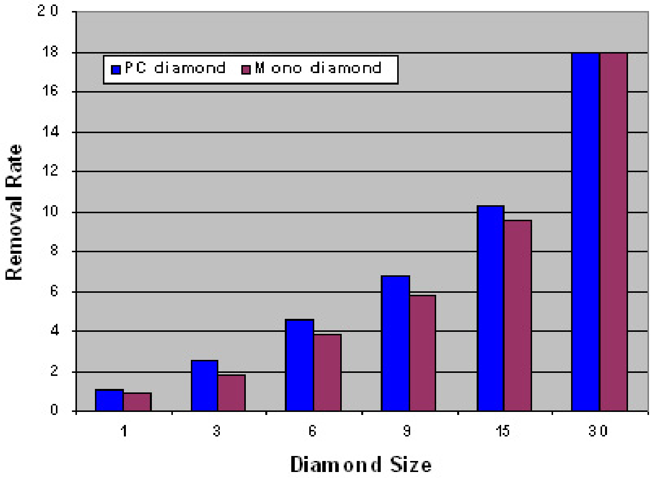
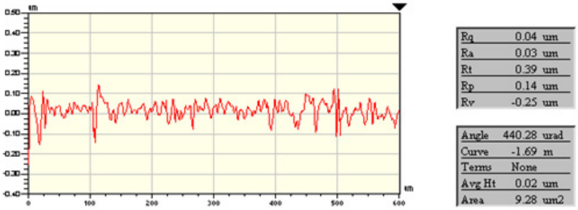


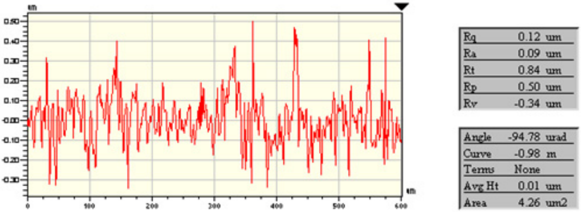
Figure 5-9 Polycrystalline diamond vs. monocrystalline diamond removal rate.

Figure 5-9 shows polycrystalline diamond has a higher cut rate as compared to monocrystalline diamond for sizes up to 15 micron. For coarser diamond the cut rates do not differ significantly between polycrystalline and monocrystalline diamond.

In addition to higher cut rates, polycrystalline diamond also produces a finer surface finish. From Figure 5-10, the surface roughness, Ra, for rough polishing a low carbon steel with a 3 micron diamond was 0.03 micron for polycrystalline diamond and 0.09 micron for monocrystalline diamond. As demonstrated by the Rq value (0.012 micron for monocrystalline diamond, 0.04 micron polycrystalline diamond), the average depth of the scratches is also much deeper for monocrystalline diamond as compared to the PC diamond.



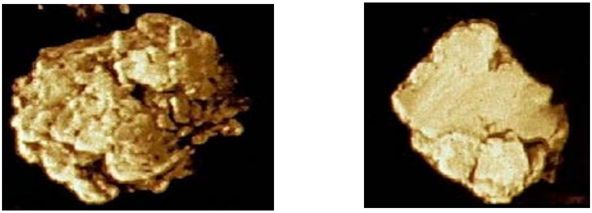
Surface roughness of a low carbon steel polished with 3 um polycrystalline diamond



Surface roughness of a low carbon steel polished with 3 um monocrystalline diamond

Figure 5-10 Surface roughness comparison for grinding with polycrystalline diamond vs. monocrystalline diamond.

Higher magnification characterization of polycrystalline and monocrystalline diamond shows that polycrystalline diamond has a rougher surface with a larger number of smaller cutting points (Figure 5-11). Polycrystalline diamond also has higher friability due to its ability to cleave along these microcrystalline planes. In general, higher-friability diamonds produce better surface finishes.



Polycrystalline diamond (high friability)

Monocrystalline diamond (low friability)

Figure 5-11 Surface roughness comparison between polycrystalline diamond and monocrystalline diamond.

5.1.4 Zircon

Zircon, or zirconium silicate, is another less common abrasive used for coarse grinding (Figure 5-12). It is a very tough abrasive, so it lasts longer, however it is generally not as hard or sharp, and thus requires higher pressures to be effective. Typically 60 or 120 grit sizes have been found to be the most useful grain sizes for metallographic grinding with zircon.

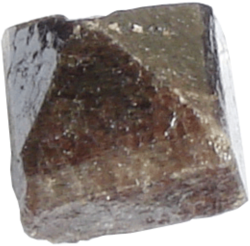


Figure 5-12 Zircon abrasive particle.

5.2 ABRASIVE BONDING

5.2.1 Fixed Abrasive Grinding

For fixed abrasive (two-body) grinding disks or surfaces, the abrasive is rigidly held in place (Figure 5-13). Common bonding materials include:

- Nickel plating
- Polymer / epoxy resins
- Soft lapping plates (tin, zinc or lead alloys)

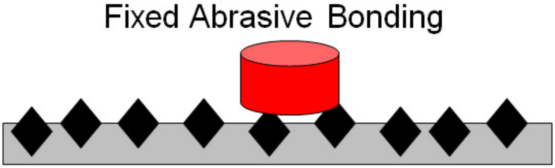


Figure 5-13 Rigidly-held abrasive grinding for fixed abrasive bonding.

The characteristic features for grinding with fixed abrasives are high, or aggressive, removal rates with the potential for significant surface and subsurface damage. Common fixed abrasive grinding surfaces are bonded diamond disks, silicon carbide / alumina papers and lapping films.

Application (Fixed-abrasive Grinding)
- Start with the finest abrasive possible (typically 240 (P220) or 320 (P360) grit abrasive paper for SiC papers, 600 (P1200) grit alumina paper, or 30-45 micron diamond).

Note: Only use coarser grits or larger abrasives for very heavy stock removal and be careful about the additional damage produced.

- Apply lubricant to abrasive surface. Water is the most common lubricant; however, light oils can be used for water-sensitive samples.
- Clean specimens and holder thoroughly before proceeding to the next finer abrasive step.

5.2.2 Free Abrasive Grinding

For free abrasive (three-body) grinding, the abrasive is not rigidly held in place and is allowed to freely move between the specimen and the working plate (Figure 5-14). This abrasive action leads to very non-aggressive removal with the flatness of the specimen matching that of the base lapping plate surface. Free abrasive grinding is commonly used for lapping hard materials on hard lapping surfaces such as cast iron. This is not a very common metallographic specimen preparation technique.

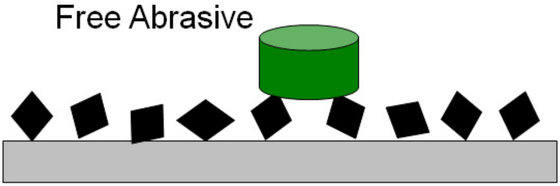


Figure 5-14 Abrasive rolls between workpiece and base plate for free abrasive grinding.

5.2.3 Semi-fixed Abrasive Grinding

Semi-fixed abrasive grinding is a hybrid process which uses a rough, or an interrupted, grinding surface (Figure 5-15). The abrasive is applied in the same fashion as free abrasive grinding, however the abrasive can become temporarily fixed in the interrupted surface, thereby providing a more aggressive grinding action.

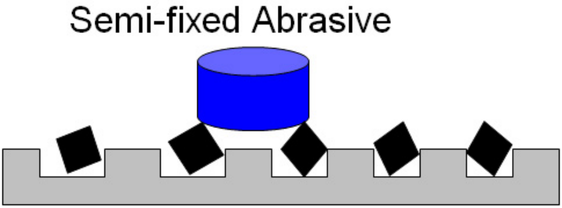


Figure 5-15 Abrasive is temporarily held in place for semi-fixed abrasive grinding.

The characteristic grinding features of semi-fixed abrasive grinding includes:
- Good removal rates
- Medium abrasive exposure (less damaging)
- Excellent for grinding/polishing brittle materials
- Rechargeable abrasive

Application (CERMESH metal mesh cloth) (see Figure 5-16)
- Apply CERMESH metal mesh cloth to flat base surface
- Pre-charge CERMESH metal mesh cloth with DIAMAT polycrystalline diamond
- To avoid tearing the cloth, begin initial grinding at 50% of the force to planarize the specimen(s) with the metal mesh cloth
- Ramp-up force gradually
- Add abrasive as required
- Rinse CERMESH metal mesh cloth with water at the end of the grinding cycle to remove grinding swarf debris

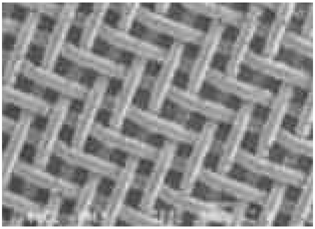


Figure 5-16 CERMESH interrupted metal mesh disk for semi-fixed abrasive grinding.

TIP: To apply adhesive-backed abrasives, peel back protective paper at one corner and align it with the working wheel surface. Continue to pull the backing liner with one hand while applying the paper/film with the other hand.

5.3 ROUGH GRINDING PARAMETERS

Successful grinding is also a function of the following parameters:

1. Grinding pressure
2. Relative velocities and grinding direction
3. Machine considerations

The machining parameters which affect the preparation of metallographic specimens include grinding/polishing pressure, relative velocity distribution between the specimen and grinding surface, and the direction of grinding/polishing action relative to the specimen. In general, grinding removal rates are described by Preston's Law. This relationship states that removal rates are proportional to the grinding velocity and applied pressure.

PRESTON'S LAW

Removal Rate = kPV
k - Preston's constant
P - Polishing pressure
V - Polishing velocity

5.3.1 Grinding Pressure

Grinding/polishing pressure is dependent upon the applied force (pounds or Newtons) and the area of the specimen and mounting material. Pressure is defined as the Force/Area (psi, N/m^2 or Pa). For specimens significantly harder than the mounting compound, the pressure is better defined as the force divided by the specimen surface area. Thus, for large hard specimens, a higher grinding/polishing pressure increases stock removal rates, however, higher pressure can also increase the amount of surface and subsurface damage.

Note: Increasing the grinding force can extend the life of the SiC grinding papers as the abrasive grains dull and cut rates decrease.

Higher grinding/polishing pressures can also generate additional frictional heat which may actually be beneficial for the chemical mechanical polishing (CMP) of ceramics, minerals and composites. Likewise for extremely friable specimens such as nodular cast iron, higher pressures and lower relative velocity distributions can aid in retaining inclusions and secondary phases.

5.3.2 Relative Velocity

Current grinding/polishing machines are designed so that the specimens are mounted in a disk holder and machined on an abrasive grinding disk surface. This disk-on-disk rotation allows for a variable velocity distribution depending upon the specimen head speed relative to the abrasive wheel base speed (see Figure 5-17).

Figure 5-17 Automated polishers using disk on disk rotation.

For disk-on-disk rotation, the relative direction of the specimen disk and the grinding disk are defined as operating in either the complementary direction (same rotation) or contra direction (opposite rotation) (see Figure 5-18).

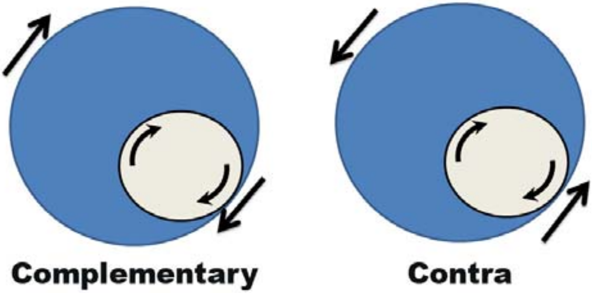


Figure 5-18 Complementary and contra disk on disk rotation.

For high stock removal, a slower head speed relative to a higher base speed produces the most aggressive grinding/polishing operation (Figure 5-19a). As can be seen from Figure 5-19, the relative velocity is very high at the outside edge of the working wheel when the specimen and abrasive are traveling in the opposite, or contra, direction. Conversely, at the inside diameter of the abrasive working wheel, where the specimen is traveling in the same direction as the abrasive, the relative velocities cancel each other and are at a minimum.

This "hammering" action of contra rotation produces very aggressive grinding rates and can possibly damage the brittle components, inclusions or the more sensitive features of the specimen. Another drawback to high velocity distributions is that the abrasive (especially SiC papers) may not breakdown uniformly. This effect can result in nonuniform removal across the specimen surface.

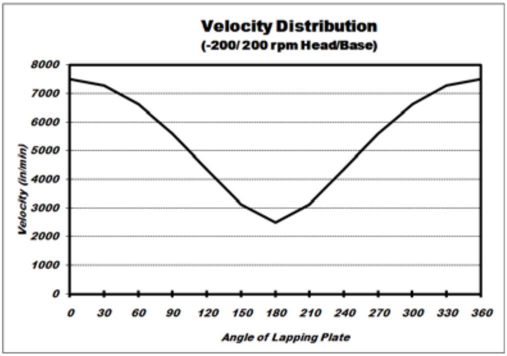


Figure 5-19a High velocity differential for disk-on-disk rotation operating in the contra direction.

Operating the specimen power head in the same direction and at the same rpm as the abrasive working wheel produces a condition having a minimal velocity distribution (Figure 5-19b). This condition is known as grinding / polishing in the complementary direction, and provides the best condition for retaining inclusions and brittle phases, as well as obtaining a uniform finish and flatness across the entire specimen. The main disadvantage of operating at the same speeds in the complementary direction is that stock removal rates are relatively low.

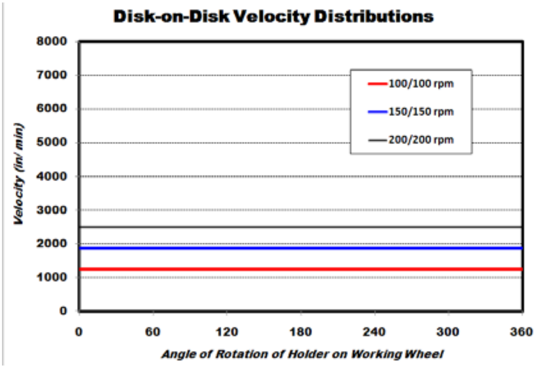


Figure 5-19b Low velocity differential for disk-on-disk rotation operating in the complementary direction.

In practice for most common materials, matching the head and base speed at as high a speed as possible is the best condition for obtaining a uniform and flat surface which also minimizes damage to the critical features of the microstructure. Matching the head and base speed is also more critical when coarse grinding is accomplished with individual specimen loading using semi-automated machines. For example, grinding with 180 grit or coarser paper in the contra direction (-200 rpm head/ 200 rpm base) will result in a wedge being ground across the sample when using individually loaded pistons (see illustration in Figure 5-20). Conversely, if the head is run at 200 rpm and the base is run at 200 rpm in the same direction, the sample remains squarer.

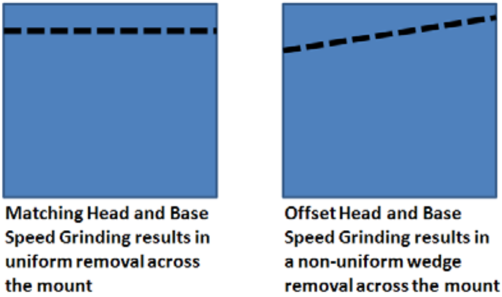


Figure 5-20 Grinding with individual force machines flatness consideration.

Note: for certain materials where chemical mechanical polishing (CMP) is recommended, high velocity distributions can provide some frictional heat which can enhance the chemical polishing action. For CMP polishing, high speeds and high relative velocity distributions can be useful as long as brittle phases are not present (e.g. monolithic ceramics such as silicon nitride and alumina). Figure 5-21 shows the relative guideline charts for planar grinding various classes of materials.

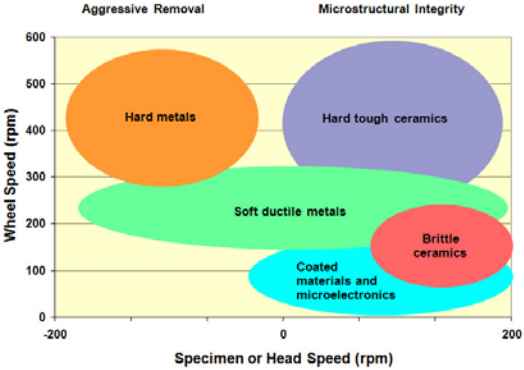


Figure 5-21a Relative velocity distribution guidelines for planar grinding of various materials using Central Pressure Machines.

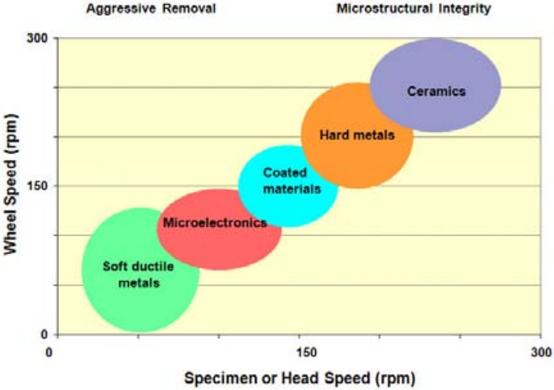


Figure 5-21b Relative velocity distribution guidelines for planar grinding of various materials using Individual Pressure Machines.

The orientation of the specimen can also have a significant impact on the preparation results, especially for specimens with coatings. In general, when grinding and polishing materials with coatings, the coating component should be kept in compression. In other words, the direction of the abrasive should be through the coating and into the substrate.

TABLE XVI. Disk-on-Disk Velocity Distributions

Head Speed (rpm)	**Base Speed (rpm)**	**Relative Velocity Distribution**	**Characteristic**	**Application**
100	300 to 600	High	-Aggressive stock removal -Differential grinding across the specimen surface	-Useful for gross removal on hard specimens when using central pressure machines
200	200	Moderate	-Matching head and base speed in the same direction eliminates the relative velocity differences -Reasonable removal and minimal damage	-RECOMMEDED for most applications -Standard grinding with individual pressure machines
100	100	Low	-Matching head and base speed in the same direction eliminates the relative velocity differences -Uniform stock removal -Low stock removal -Produces minimal damage	-For very brittle and/or soft materials

5.3.3 Machine Considerations

There are a number of considerations that need to be considered when determining the type of machine to be used for metallographic specimen preparation. A few of the more significant include:

1. Size of the sample
2. Material properties
3. How the sample was cut or sectioned
4. Flatness required
5. Stock removal requirements or limitations
6. Number of samples

Sample Size Limitation: As a general rule, the largest specimen size that can be effectively ground or polished is approximately 1/3 the diameter of the working wheel. This limitation is due to the changing velocities which occur as the sample crosses the center of the working wheel (change in the direction of grinding).

Note: Grinding samples too large for the working wheel can create significant safety issues which can result in personal injury. *DO NOT grind samples larger then 1/3 the diameter of the working wheel!*

Material Properties: The specimen preparation procedure and thus the equipment design depends upon the properties of the material. The two basic material properties which dictate the grinding/ polishing procedure are the hardness and ductility (brittleness) of the specimen. In general, machines with variable speed working wheels and variable speed polishing heads utilizing variable force are recommended for metallographic specimen preparation.

Cutting or Sectioning Damage: As a guideline, it is better to reduce the initial cutting/ sectioning damage by using the recommended abrasive blade, abrasive size and cutting conditions. By reducing sectioning damage, finer initial grinding abrasives can be used. This is very important for manual and individual specimen automated polishing machines where if not carefully controlled can result in a non-square surface/mounts.

Note: For individual specimen preparation, variable speed power heads are recommended in order to match the speed of the polishing base with the polishing head to reduce damage and to produce square mounts.

Flatness Required: For specimens requiring equal stock removal or a high degree of flatness the grinding machine must be able to hold the specimen flat. The best way to maintain flatness is to mount multiple samples into a fixed or central specimen holder. The advantage of central pressure polishing is that the sample is held in a larger diameter fixed plane. The disadvantages include (1) a minimum of three samples is required to establish the plane, (2) samples cannot be removed and remounted without having to re-planarize the specimens and (3) controlling the removal rate across multiple samples can be very difficult. It is also possible to use individual specimen preparation and obtain flat specimens; however, in order to achieve flat specimens with this polishing mode the speed of the specimen head needs to match the speed of the polishing wheel rotating in the same direction.

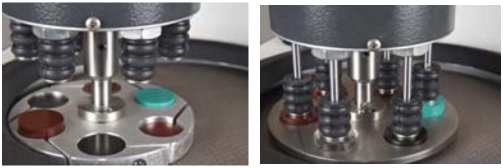


Figure 5-22 Application of specimen force (a) left Central pressure (b) individual pressure.

Stock Removal Limitations: In some cases, a materials removal needs to be monitored and perhaps controlled. One such example is the specimen preparation of a heat treated part for testing surface hardness. For this requirement the surface needs to be flat and relatively smooth; however, the amount of stock removal is limited. Specimen preparation for these types of samples requires an individual specimen preparation machine which has the ability to produce flat or uniform stock removal. This requires that the head and base speed be run at the same speed and in the same direction. A machine with both a variable speed head and a variable speed working wheel provides for the most efficient specimen preparation techniques.

Material removal: It is also important to be able to measure the stock removal in order to ensure that the sample is not overground. A very simple way to do this is to mount one or more steel ball bearings in the mount with the sample. Using either a low powered microscope or the filars on a microhardness tester the measured segment diameter of the ball bearing can be used to calculate the amount of material removed (Figure 5-23). 1/8 or 1/4-inch diameter steel ball bearings are commonly used for this application.

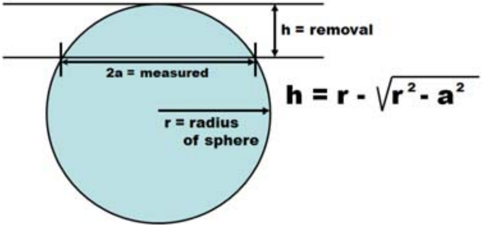


Figure 5-23 Ball bearing method for measuring material removal.

5.4 PLANAR GRINDING (ROUGH GRINDING)

The best time-tested methods for rough grinding metals, plastics, rubber and softer composite materials are to use alumina or SiC abrasive grinding papers. Other techniques have been used; however, they typically are very expensive and require too much maintenance for these types of materials. Alumina and SiC abrasive papers fit into a class of grinding known as fixed abrasive grinding.

Number of Samples: The required number of samples that are polished also determines the size of the polishing machines and power head, as well as, whether central or individual specimen preparation is more efficient. Unless, there are other overriding requirements, central polishing pressure on 12-inch diameter polishing machines are recommended for high volume samples. For low volume samples, 8-inch diameter machines using individual specimen preparation heads is acceptable as long as the head and base speeds are set properly as previously described.

5.4.1 Soft Nonferrous Metals

It is recommended that the initial grinding of soft nonferrous metals be done with 600 (P1200) grit alumina abrasive paper followed by 800 (P2400) and 1200 (P4000) grit SiC papers. Since these materials are relatively soft and can embed fractured abrasives, initial grinding with alumina is generally sufficient for minimizing initial deformation while maintaining good removal rates.

5.4.2 Soft Ferrous Metals

Soft ferrous metals are relatively easy to grind with the depth of deformation being a major consideration. Using 240 (P220) grit SiC abrasives provides a good initial start, with the subsequent use of 320 (P360) , 400 (P800), 600 (P1200), 800 (P2400) and 1200 (P4000) grit SiC papers. Planar grinding starting with 360 (P500) or 600 (P1200) grit alumina grinding papers can also be effective and reduce the number of grinding steps.

5.4.3 Hard Ferrous Metals

Harder ferrous metals require more aggressive abrasives to achieve adequate material removal. Thus, coarse SiC abrasives (120 or 180 grit) are recommended for stock removal requirements. Once planarity and the area of interest are obtained, a standard 240 (P220) , 320 (P360), 400 (P800) and 600 (P1200) grit series is recommended.

5.4.4 Super Alloys and Hard Nonferrous Alloys

Hard nonferrous metals such as titanium are relatively easy to grind with SiC papers. Depending upon the initial condition of the specimen, grinding with 240 (P220), 320 (P360), 400 (P800), 600 (P1200), 800 (P2400) and 1200 (P4000) grit papers will produce excellent results.

5.4.5 Ceramics

Engineered ceramics are extremely hard, corrosion-resistant and brittle materials. They fracture easily, producing both surface and subsurface damage. Proper grinding minimizes both of these forms of damage. This requires the application of a semi-fixed abrasive. The use of a metal mesh cloth (CERMESH cloth) with an applied abrasive accomplishes these goals. The abrasive size is also important because very coarse abrasives will quickly remove material but can seriously damage the specimen. For ceramics, consideration of the damage produced at each preparation step is critical to minimizing the preparation time.

5.4.6 Composites

Composite materials are perhaps the most difficult specimens to prepare because of their wide range of mechanical and chemical properties. For example, a metal matrix composite (MMC) such as silicon carbide ceramic particles in an aluminum metal matrix is a difficult specimen to prepare. This composite contains extremely hard/brittle ceramic particles dispersed in a relatively soft/ductile metal matrix. As a general rule, initial grinding should focus on planarization of the metal and grinding to the area of interest. The secondary grinding steps require focusing on the ceramic particles and typically require the use of diamond and CMP polishing.

Figure 5-24 PENTA-5000 Five station hand grinder.

5.5 PLANAR GRINDING TROUBLESHOOTING

The most common problems associated with planar grinding result from using the incorrect abrasive type or size (see Table XVII).

TABLE XVII. Troubleshooting Guidelines for Planar Grinding

Symptoms	Cause	Action
Uneven grinding across the specimen	-Improper tracking of specimen over the entire abrasive paper -Mount not square with individual force grinding	Orient specimen holder so that hardest portion of specimen / mount tracks over entire abrasive paper (uniform degradation of paper) -Match head speed with base speed in the same direction
Excessive vibration in machine	-Load too high or speed to low -Inadequate machine design for application -Improper lubricant	-Reduce initial grinding force or increase grinding speed -Check with equipment vendor for equipment upgrades -Increase lubricant flow and / or use a water soluble lubricant
Embedding of fractured abrasive grains	-Common in the grinding of very soft materials	-Switch from SiC papers to a more durable grinding abrasive such as alumina

5.6 PRECISION GRINDING WITH LAPPING FILMS

The use of lapping films range from polishing semiconductor dies, fiber optics, optical components, ceramic capacitors, computer hard drive read-write heads, ceramic seals, etc. The main characteristic of lapping films is that they produce very flat surfaces, especially across materials having a wide range of hardness.

Figure 5-25 Polyester backed lapping films.

Lapping films consist of a polyester backing (typically 3 mils) on which the abrasive is rigidly fixed with an epoxy binder (Figures 5-25 & 5-26). The most commonly used abrasives for lapping films include diamond, silicon carbide, alumina, and to a lesser extent ceria and colloidal silica.

5.6.1 Diamond Lapping Films

Diamond lapping films are uniformly coated abrasives using precisely graded diamond. A very flexible adhesive bonding agent is used which tenaciously holds the abrasive particles to the backing. This adhesive is designed to resist cracking or peeling. It is applied to a tough, durable, tear-resistant polyester plastic film which is waterproof and resistant to many solvents.

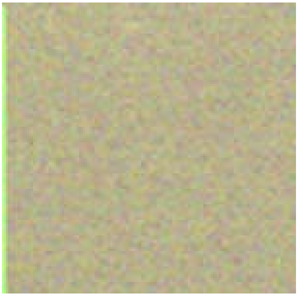
Diamond lapping films provide less polishing relief than intermediate rough polishing cloths and remove material in a fixed diamond grinding mode. Diamond lapping films are exceptional abrasives for preparing microelectronic materials for SEM and TEM analysis.

Diamond lapping films range in particle sizes from 0.1 micron up to 60 micron, with rough grinding lapping films typically ranging from 15 micron to 60 micron (Figure 5-26).

Diamond Lapping Films Applications

For plain backed diamond lapping films:

- Wet the flat lap plate with water or a water/ surfactant solution (or use a receiver disk)
- Place the plain backed lapping film on surface
- Roll or press out any entrapped air bubbles
- Apply necessary lubricant
- Begin polishing with lower force to avoid tearing film
- Increase grinding force gradually
- Clean specimen and film for final 10-15 seconds of polishing cycle
- Clean and dry specimens



45 micron Diamond

30 micron Diamond

15 micron Diamond

Figure 5-26 Diamond lapping films.

For PSA backed diamond lapping films:

- Place the PSA backed lapping film on the surface
- Apply necessary lubricant
- Begin polishing with lower force to avoid tearing film
- Increase grinding force gradually
- Clean specimen and film for final 10-15 seconds of polishing cycle
- Clean and dry specimens

5.6.2 Silicon Carbide Lapping Films

Silicon carbide lapping films are similar to SiC grinding papers; however, the abrasive is applied to a polyester backing instead of paper. The advantages of a polyester backing are that it produces flatter surface finishes and less polishing round-off compared to paper backed abrasives.

Silicon Carbide Lapping Films Applications

For plain backed silicon carbide lapping films:

- Wet the flat lap plate with water or a water/ surfactant solution (or use a receiver disk)
- Place the plain backed lapping film on the surface
- Roll out or press out any entrapped air bubbles
- Apply necessary lubricant
- Begin polishing with lower force to avoid tearing film
- Clean specimen and film for final 10-15 seconds of polishing cycle
- Clean and dry specimens

For PSA backed silicon carbide lapping films

- Place the PSA backed lapping film on the surface
- Apply necessary lubricant
- Begin polishing with lower force to avoid tearing film
- Clean specimen and film for final 10-15 seconds of polishing cycle
- Clean and dry specimens

5.6.3 Alumina Lapping Films

Alumina lapping films consist of an aluminum oxide, which is a naturally occurring material (Bauxite). It exists in either the softer gamma (mohs 8) or harder alpha (mohs 9) phase. Alumina abrasives are used primarily as final polishing abrasives because of their high hardness and durability. Alumina abrasives are also available in a wide range of particles sizes for lapping films, ranging from 0.05 micron up to 60 grit. The typical range for rough lapping is 12 micron and coarser. Lapping films are also color coded in order to better distinguish the abrasive size (Figure 5-27).

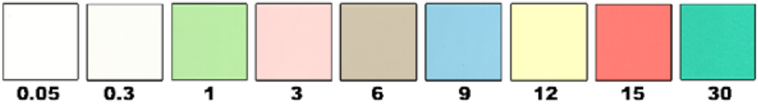


Figure 5-27 Color coding for alumina lapping films.

Alumina Lapping Films Applications

For plain backed alumina lapping films:

- Wet the flat lap plate with water or a water/surfactant solution (or use a receiver disk)
- Place the plain backed lapping film on the surface
- Roll out or press out any entrapped air bubbles
- Apply necessary lubricant
- Begin polishing with lower force to avoid tearing film
- Increase grinding force gradually
- Clean specimen and film for final 10-15 seconds of polishing cycle
- Clean and dry specimens.

For PSA backed alumina lapping films:

- Place the PSA backed lapping film on the surface
- Apply necessary lubricant
- Begin polishing with lower force to avoid tearing film
- Increase grinding force gradually
- Clean specimen and film for final 10-15 seconds of polishing cycle
- Clean and dry specimens.

5.7 LAPPING FILM TROUBLESHOOTING

The common problems associated with using abrasive lapping films are listed in Table XVIII.

TABLE XVIII. Troubleshooting Guidelines for Lapping Films

Symptoms	Cause	Action
Uneven grinding across the specimen	-Improper tracking of specimen over the entire abrasive paper	Orient specimen holder so that hardest portion of specimen / mount tracks over entire abrasive paper (uniform degradation of paper)
Excessive vibration in machine	-Load too high or speed to low -Inadequate machine design for application -Improper lubricant	-Reduce initial grinding force or increase grinding speed -Check with equipment vendor for equipment upgrades -Increase lubricant flow and / or use a water soluble lubricant
Embedding of fractured abrasive grains	-Common in the grinding of very soft materials	-Switch from SiC papers to a more durable grinding abrasive such as alumina

5.8 ROUGH POLISHING

The most critical metallographic preparation step is rough polishing. At this step, the remaining surface and subsurface damage needs to be removed. After this stage, the true microstructure of the material should be restored (inclusions, brittle phases, voids, porosity, etc.) with exception of a few surface imperfections, which can be subsequently removed at the final polishing stage.

Rough polishing is most commonly accomplished with woven, low napped (napless) polishing pads paired with abrasive slurries such as diamond or alumina (Figure 5-25). The primary objective for rough polishing with woven polishing pads is to maintain the flatness across the specimen surface, especially if the specimen has both hard and soft phases, coatings or other critical features.

Note: For cases where flatness is absolutely critical, lapping films may be a better alternative.

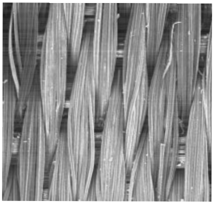


Figure 5-28 Woven polishing pads provide both a support for the abrasive, as well as a flat surface for the specimen.

5.8.1 Rough Polishing Abrasives

Rough polishing abrasives typically range from 15 micron down to 1 micron, with alumina and diamond suspensions or fixed lapping films representing the majority of the abrasive applications. For relatively soft materials, alumina powders, suspensions or slurries are widely used. **Note:** Alumina is also a relatively inexpensive abrasive, compared to diamond.

On the other hand, diamond has either a blocky (monocrystalline) structure or a spherical nodular (polycrystalline) structure. For rough polishing, polycrystalline diamond typically produces higher cut rates compared to monocrystalline diamond and, in general, produces a better surface finish (see section 5.1.3).

5.8.2 Rough Polishing Pads

For rough polishing operations that use alumina or diamond slurries, the correct choice for the polishing pad surface is very critical. As already indicated, low napped polishing pads are recommended for rough polishing. Low napped polishing pads include woven, urethane coated fibers and porous urethane pads. Although, to a certain extent, determining the correct polishing pad is based on empirical trial and error experimentation, a number of properties which affect the polishing pad characteristics include:

1. Resin fibers (hardness, density, size, count, chemistry)
2. Type of weave
3. Compressibility of the pad
4. Porosity or polishing pad surface area
5. Wetability of the abrasive suspension with the pad

Following are examples of common polishing pads:

POLYPAD polishing pad - a very tightly woven, large weave polishing pad for coarse and intermediate polishing. This polishing pad is very similar to the material used for sailboat sails. POLYPAD polishing pads are typically used with coarser diamond polishing compounds and suspensions (6, 9 and 15 micron). Due to the durability of the POLYPAD polishing pad material, it has been used for both a metallographic and industrial polishing pads.

Figure 5-29 POLYPAD polishing pad and SEM image of fibers.

TEXPAN polishing pad - a fibrous material that has been coated or impregnated with a urethane polymer. This is a very commonly used polishing pad for intermediate polishing of metal with diamond abrasives (1,3,6 and 9 micron). It has also been successfully used for intermediate polishing of ceramics when diamond and colloidal silica are combined to produce a chemical mechanical polishing (CMP) action (e.g. 1 micron diamond and 0.06 micron SIAMAT colloidal silica).

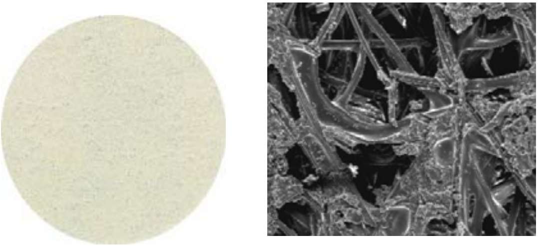


Figure 5-30 TEXPAN polishing pad and SEM image of porous matrix.

The next set of polishing pads are used with the finer diamond and alumina polishing abrasives. These polishing pads are woven polishing pads with a relatively small weave. Examples of these polishing pads include: **DACRON (Dupont DACRON fibers), NYPAD (silk pad)** and a hybrid **GOLDPAD polishing pad**. These can also be referred to as low nap or woven polishing pads. Typical diamond abrasive sizes used for these polishing pads range from 0.05 micron up to 9 micron.

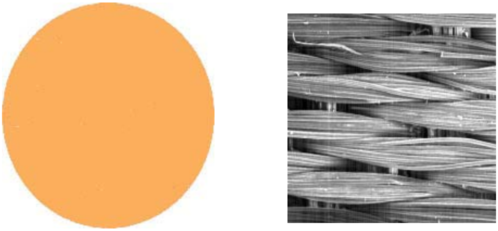
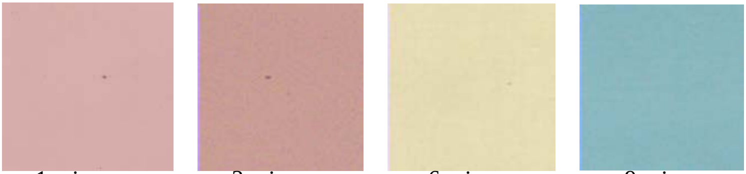


Figure 5-31 GOLDPAD polishing pad and SEM image of fiber weave.

5.8.3 Rough Polish Lapping Films

As an alternative to polishing pads, alumina or diamond lapping films can be used for the rough polishing step. The primary advantage to lapping films over polishing pads is better flatness. Recall that the abrasive is rigidly fixed to a polyester backing and thus has a much lower compressibility. The result is a flatter surface with less relief between hard and soft constituents. The primary drawback to lapping films is that they are not as compliant to the specimen surface compared to polishing pads. One way to improve this disadvantage is to stack the lapping film on top of a polishing pad or, in some cases, a rubber pad. This is commonly done for polishing ceramic fiber optic ferrules.



1 micron 3 micron 6 micron 9 micron

Figure 5-32 Fine diamond lapping films.

5.8.4 Automated Rough Polishing

For automated disk-on-disk polishing, the relative velocity speed of rough polishing is dependent upon the type of material being prepared (Figure 5-33). For metals and materials highly susceptible to damage (brittle ceramics and microelectronic materials), the relative velocity differences should be small.

Thus, running the specimen holder in the same direction and at approximately the same head and base speed (e.g. 100 rpm base speed / 100 rpm head speed) will create the least amount of damage to the specimen. However, keep in mind, removal rates will also be relatively low under these conditions.

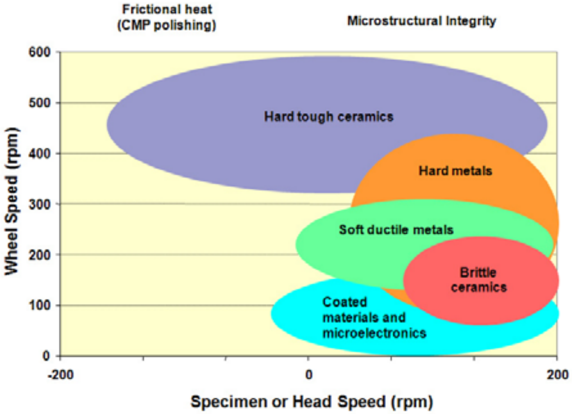


Figure 5-33 Relative velocity distribution guidelines for rough polishing various materials.

For harder and tougher engineered ceramics such as silicon nitride, zirconia and boron nitride, it is recommended that the relative velocity difference be much higher, thus producing a much more aggressive rough polishing condition. This coupled with high polishing pressures, works very well for chemical mechanical polishing (CMP) of these types of materials.

5.8.5 CMP (Chemical Mechanical Polishing)

Chemical Mechanical Polishing (CMP) is a technique which utilizes both mechanical and chemical abrasive polishing. CMP polishing is very useful for minimizing both surface and subsurface damage in ceramics, semiconductor materials, and soft metals. The most common CMP abrasives are colloidal silica and alumina at either low or high pH values. In addition to pH, the solution chemistry or oxidation / reduction potential of the solution is very important for CMP polishing. This change in oxidation potential can significantly enhance the chemical polishing attack on the specimen surface.

A useful tool for initially evaluating a particular specimen's ability to be polished by CMP processes is with the use of Pourbaix diagrams. Pourbaix diagrams are thermodynamically derived stability diagrams for various pH and oxidation potentials. Figure 5-34 shows the Pourbaix diagram for the alumina or aluminum-water system. From the Pourbaix diagram, the stable species at pH values ranging from 4 to 10 is alumina. However, at pH values below 4, Al^{+3} is more thermodynamically stable and, at pH values above 10, AlO^{2-} is the most stable species. This would indicate that CMP polishing of an alumina ceramic would be more feasible at either low or high pH values. **Note:** although Pourbaix diagrams are useful for determining the most thermodynamically stable species, they do not provide any kinetic or dissolution rate information. The actual dissolution rates can be determined with more sophisticated electrochemical methods, although in practice, the effectiveness of CMP polishing is determined by experimental techniques.

Colloidal Silica Abrasives for Ceramics

Colloidal silica polishing suspensions are unique because they provide both a dispersing action as well as a chemical mechanical polishing (CMP) action when used in combination with other mechanical abrasives, such as diamond. The increased polishing effect can be very significant. In fact, the combination of diamond and colloidal silica provides the best polishing abrasive action for eliminating subsurface and surface damage in ceramics.

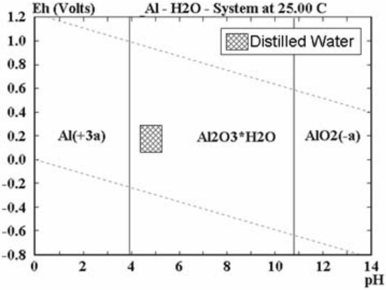


Figure 5-34 Electrochemical Pourbaix diagram for determining the regions of chemical stability.

Colloidal silica is a very unique polishing suspension because it provides a chemical mechanical polishing (CMP) action for materials such as ceramics, composites and soft metals. The most common colloidal silica's have particle size distributions between 0.05 micron and 0.07 micron and are stabilized at alkaline pH values (typically >9.5). At higher pH values, colloidal silica is held in a nearly perfect suspension by the electrochemical repulsive forces of the fine particles themselves. This chemical balance electrochemically attacks the surface of a ceramic or mineral surface to form a thin reacted layer on the specimen surface. This reacted layer can then be removed via the mechanical action of the polishing cloth or another abrasive (e.g. diamond). CMP polishing rates and surface finishes are significantly better for ceramics and minerals as compared with diamond polishing. Thus, the highest removal rates and the most reliable action for removing both surface and subsurface damage on ceramics and minerals is via chemical mechanical polishing with colloidal silica.

As with other exothermic chemical reactions, the chemical contribution can be enhanced by increasing the temperature of the polishing action. This can be accomplished by increasing the polishing pressure and polishing velocity to increase the friction between the polishing cloth, colloidal silica and the specimen.

Cleaning of the surface is best accomplished by rinsing the polishing pad surface with distilled water for the final 10-15 seconds of the polishing cycle and then immediately rinsing the specimen surface with water. This allows the cloth to mechanically remove the reacted layer and any residual colloidal silica.

CMP polishing example (silicon nitride)

An example of CMP polishing for ceramics can be illustrated for the specimen preparation or polishing of silicon nitride. Silicon nitride is a very hard and tough ceramic and has found many applications for advanced materials engineering. Figures 5-35a and 5-35b compare the effects of stepwise polishing with more traditional finer diamond polishing to that with colloidal silica. For Figure 5-35a, diamond polishing included the following diamond abrasive polishing steps: 30,15, 9, 6, 3, 1, 0.5, 0.25 and 0.10 micron diamond steps for 5 minutes each.

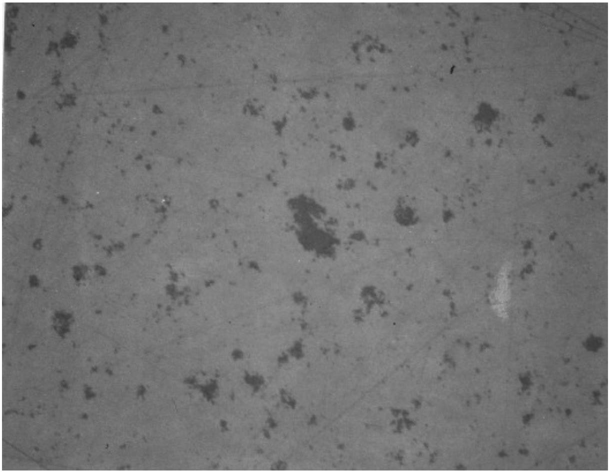


Figure 5-35a Diamond polishing of silicon nitride.

Figure 5-35b was prepared with the same initial 30 micron surface grind; however, this step was followed by a 6 micron diamond / colloidal silica mixture and then finished with colloidal silica only. Each of these polishing steps was for 5 minutes.

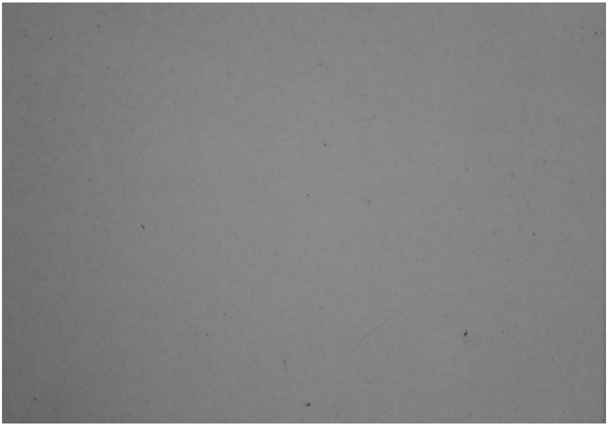


Figure 5-35b CMP polishing of silicon nitride with colloidal silica.

As can be clearly observed, the use of colloidal silica significantly reduced the subsurface and surface damage to the silicon nitride surface, whereas the diamond steps, clearly did not remove the existing damage and only appears to have propagated the initial damage.

Figure 5-36 NANO double wheel without and with FEMTO 1000 variable speed polishing head.

CHAPTER 6
Final Polishing

6.0 FINAL POLISHING

If the sample has been properly prepared up to this point, the true microstructure of the specimen should be intact. This includes retention of the inclusions, brittle phases/structures, sharp edges with no rounding, distinct porosity edges (no rounding), no smeared metal and no embedded abrasive particles. **Note:** the only purposes for final polishing should be to clean up the surface and to prepare the surface for etching (if required). If the true microstructure has not been resolved by this point in the preparation process, it is highly probable that the resulting surface after final polishing will still contain microstructural artifacts.

Final polishing is most commonly accomplished with flocked or napped polishing pads using an abrasive slurry, such as alumina. However, with the proper backing, woven pads can also be used successfully.

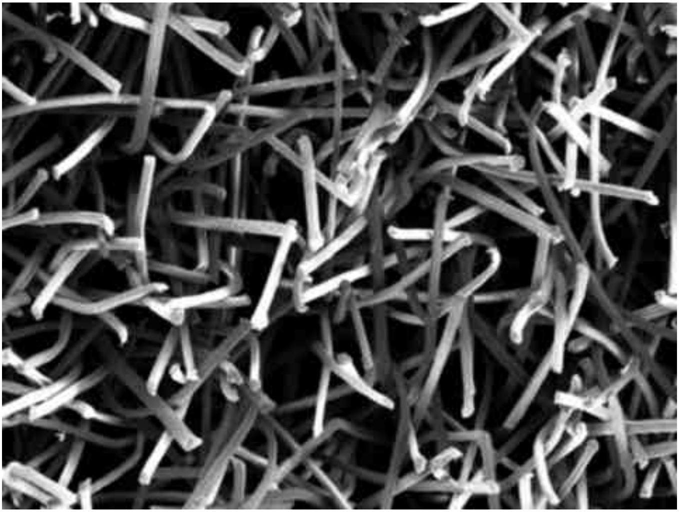


Figure 6-1 Flocked or napped polishing pad.

6.1 FINAL POLISHING ABRASIVES

Final polishing is accomplished with alumina, diamond, colloidal silica, ceria (cerium oxide) and rouge (iron oxide). However, the most commonly used abrasive for final polishing metals is alumina. Alumina abrasives can be classified as either calcined, levigated, or polycrystalline, based on their manufacturing process, crystal structure (hardness) and their sizing process (Table XIX).

TABLE XIX. Alumina Abrasive Properties

Alumina Property	Polycrystalline	Calcined	Levigated
Crystal structure	Polycrystalline alpha alumina	Monocrystalline alpha alumina	Monocrystalline gamma or alpha alumina
Shape	Rough spherical particles	Hexagonal platelets	Hexagonal platelets
Particle size	0.05 - 0.25 micron	0.5 - 15 micron	<1 micron
Specific gravity	3.95 gm/cc	3.95 gm/cc	3.95 gm/cc
Hardness	Knoop 2000 (Mohs 9)	Knoop 2000 (Mohs 9)	Mohs 8 or 9
Applications	Available in a slurry at either a pH 4 or pH 10, primarily for metal polishing	Available in powders, slurries or suspensions, rough polishing	More commonly known as Linde A (0.30 micron), Linde B (0.05 micron), Linde C (1 micron)

6.1.1 Polycrystalline Alumina

Polycrystalline, or nanometer alumina is a colloidal alumina manufactured by a proprietary seeded gell process. Polycrystalline abrasives are also milled at either a low or a higher pH in order to avoid agglomeration. The abrasive is also maintained in solution to avoid aggregation. This processing offers two significant improvements over conventional calcined alumina processes:

- Tighter, more controlled particle size distributions
- Harder alpha alumina crystal

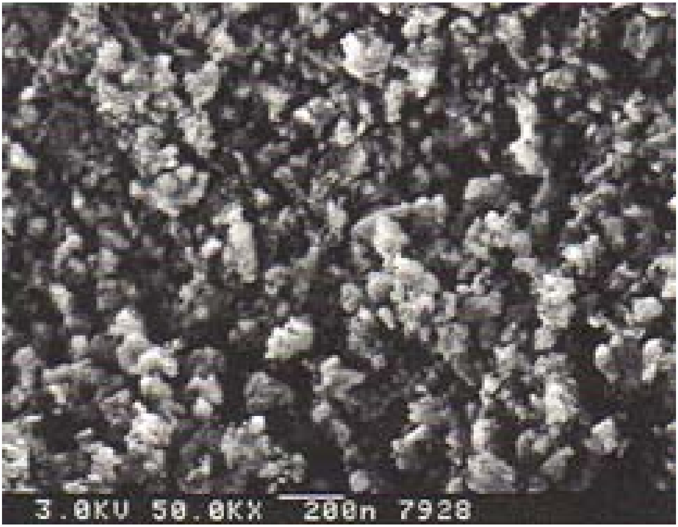


Figure 6-2 0.05 micron polycrystalline alumina.

A tighter, more controlled particle size distribution is the result of less particle aggregation. For example, standard calcined 0.05 micron gamma alumina products form aggregate sizes as large as 5 micron (Figure 6-3). In some cases, these aggregates can be broken down during polishing with harder specimens; however, they are a problem when polishing softer metals (such as aluminum, tin, lead, copper and soft steels).

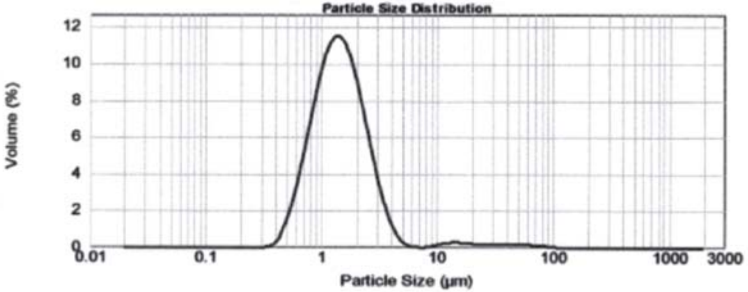


Figure 6-3 Particle size distribution for 0.05 micron deagglomerated alumina.

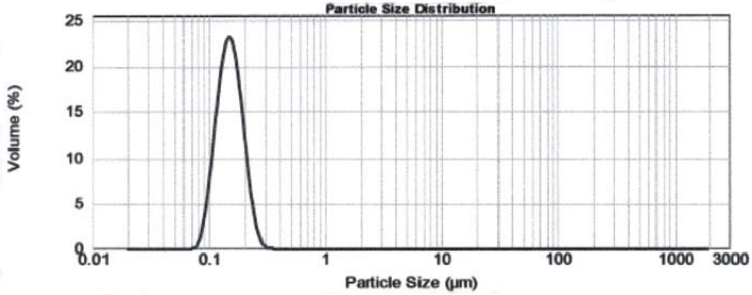


Figure 6-4 Particle size distribution for 0.05 micron polycrystalline alumina.

Nanometer polycrystalline alumina is specifically milled to produce a much smaller aggregate particle size distribution (<0.5 micron) (Figure 6-4). Polycrystalline alumina is also a harder alpha alumina particle, therefore making it a more efficient cutting abrasive. Thus, polycrystalline alumina is a much more controlled polishing abrasive compared to calcined gamma alumina abrasives.

In general, the more modern polycrystalline alumina abrasives outperform traditional calcined and levigated alumina abrasives for final polishing because they produce a much more consistent and better surface finish.

Advantages and Features of Polycrystalline Alumina

- Improved surface finishes over gamma alumina
- Less random scratching

- Higher cutting rates than gamma alumina
- More uniform surface finish
- Tighter particle size distributions than gamma alumina
- Low viscosity for easy dispensing
- Semi-permanent suspensions

Application of Polycrystalline Alumina Polishing

The most efficient polishing techniques are to initially wet the polishing cloth with the polycrystalline alumina slurry and then to slowly drip or spray the suspension onto the polishing cloth. The most common polishing cloths for alumina polishing are **MICROPAD (MICROPAD 2) pad**, **ATLANTIS pad**, **TRICOTE pad, NAPPAD pad** and **FELT PAD pad**.

MICROPAD /MICROPAD 2 polishing pads - are the most commonly used final polishing pads for alumina abrasives. These polishing pads have a high-nap, or flocked, fiber and work well for polishing metals if the polishing time is kept to a minimum. Overpolishing with high-napped pads can lead to edge rounding, inclusion pull-out, and excessive relief in the specimen. **Note: MICROPAD** and **MICROPAD 2** are the same polishing fiber; the difference between these two pads is that the **MICROPAD 2** is laminated with a stiffer backing which eliminates stretching of the cloth for easier application and removal.

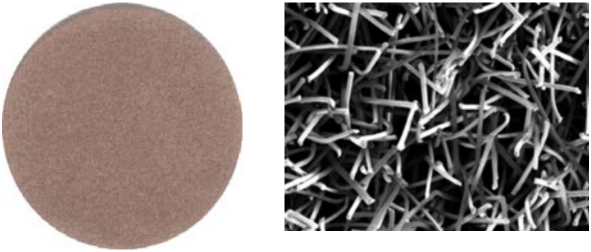


Figure 6-5 MICROPAD polishing pad and SEM image of fibers.

ATLANTIS polishing pad - is a unique woven polishing pad that has had tremendous success in final polishing. This polishing pad is a laminated polishing cloth having a resilient foam backing. The foam backing allows the polishing pad to conform better to the specimen surface and thus has similar polishing characteristics to high-napped polishing pads. The most significant difference is that this polishing pad produces flatter surfaces with less edge rounding and polishing relief. Thus the **ATLANTIS polishing pad** is very useful for final polishing of materials with coatings, composites and other materials having a wide range of hardness and ductility.

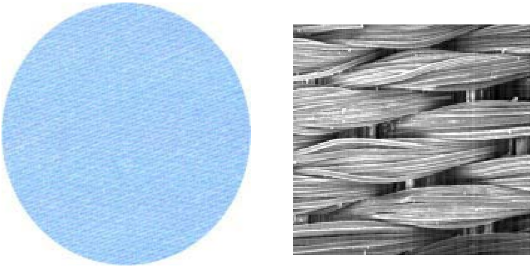


Figure 6-6 ATLANTIS polishing pad and SEM image of fibers.

TRICOTE polishing pad - a very tight-napped polishing pad. Its performance places it between the **MICROPAD** and the **ATLANTIS polishing pads**. It is very commonly used for polishing steels and other ferrous metals.

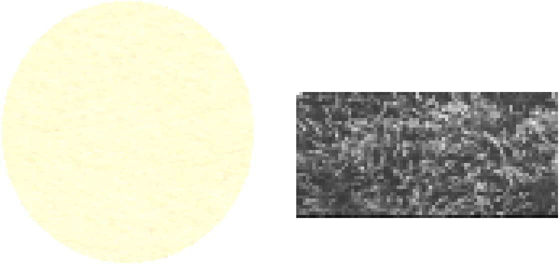


Figure 6-7 TRICOTE polishing pad and SEM image of fibers.

TIP: Polycrystalline alumina particles will electrostatically coat the specimen, leaving what appears to be a matted film. To remove, use a cotton ball with a cleaning solution, such as the **ULTRACLEAN 2,** and gently wipe off the specimen surface.

6.1.2 Calcined Alumina Polishing Abrasives

Although polycrystalline alumina is the premium alumina polishing abrasive, calcined alumina abrasives have been used for many years for polishing a wide range of materials, and are still the most commonly used alumina polishing abrasives. The primary advantage of polishing with calcined alumina abrasives over polycrystalline abrasives is cost. Typically, calcined abrasives are less expensive than polycrystalline abrasives and can be purchased in either a powder or slurry form.

Calcined alumina's typically have a plate-like (platey) structure and are available in either the harder alpha or softer gamma crystal structure. Often, the very fine calcined products (typically 1 micron or less) are jet milled to form a deagglomerated alumina powder. In general, deagglomerated calcined alumina powders have a very high bulk density and are very "fluffy."

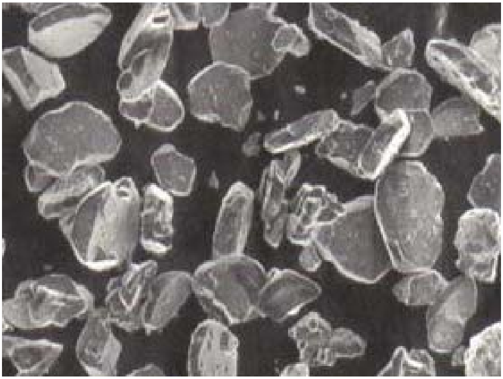


Figure 6-8 Calcined alumina particles.

Application of Calcined Alumina Polishing

Calcined alumina polishes are typically used on the same polishing pads as polycrystalline alumina, e.g. **MICROPAD**, **NAPPAD**, **ATLANTIS** and **TRICOTE**. However, they are also commonly used with the **MOLTEC 2 and FELT PAD polishing pads** which are very soft, canvas and thick felt types of polishing pad, respectively. **MOLTEC 2** pad has been a staple of the industry and is still a commonly used polishing pad with calcined alumina abrasives up to 5 microns in size. **FELT PAD** polishing pad is a very thick, 0.125" (1/8-inch) thick polishing pad.

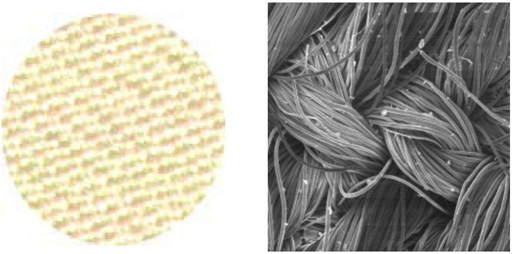


Figure 6-9 MOLTEC 2 polishing pad and SEM image of fibers.

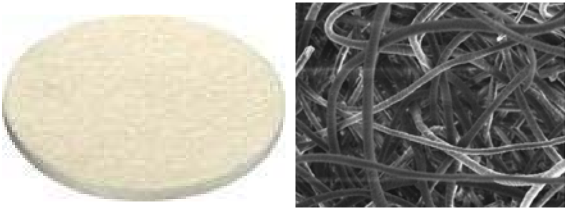


Figure 6-10 FELT PAD polishing pad image of thickness and SEM of fibers.

6.1.3 Colloidal Silica Polishing Abrasives

Colloidal silica abrasives for metallographic polishing typically range in particle size from 20 nm up to 70 nm (0.02-0.07 micron). The concentration of particles typically range from 25-50% solids. Even at these high concentrations, colloidal silica can be maintained in a nearly perfect suspension. The electrochemical stability of colloidal silica makes it an excellent chemical mechanical polishing abrasive for polishing ceramics. The main drawback to polishing with colloidal silica is that it will crystallize as it dehydrates. This is particularly troublesome around the lid of an open bottle. One way to reduce this crystallization is to add an ingredient to the colloidal silica suspension in order to reduce the evaporation rate of the water. A comparison of the two main types of colloidal silica, **SIAMAT** and **SIAMAT 2 Colloidal Silica's**, are listed in Table XX.

Figure 6-11 Colloidal silica suspensions for CMP polishing.

Table XX. Properties of Colloidal Silica

Properties	**SIAMAT colloidal silica**	**SIAMAT 2 colloidal silica**
Particle size	50-70 nm	20-40 nm
pH	9.5-10.0	9.5-10.0
Concentration	50% solids	40% solids
Specific gravity (@77°F/25°C)	1.39	1.25
Viscosity (@77°F/25°C)	15 cp	20 cp
Applications	Precision surface finishing for ceramics and glass. Ideal as a Chemical Mechanical Polishing (CMP) abrasive when used by itself or with diamond polishing abrasives	Precision surface finishing for metals and microelectronic materials
Polishing Pad recommendation	**TEXPAN** - As a polishing extender for intermediate polishing of ceramics and glass with diamond **BLACK CHEM 2** - Final polishing of glass and ceramics	**MICROPAD** - Metals and composites **BLACK CHEM 2** - Final polishing of microelectronics

Application of Colloidal Silica Polishing

Colloidal silica polishing can be used by itself as a polishing abrasive, or in combination with other harder abrasives. Colloidal silica is a unique abrasive, as it can be either a chemical agent or a mechanical abrasive. For polishing hard, and often times brittle materials (glass and ceramics) the primary polishing mechanism is chemical. For metals, composites and microelectronic components, colloidal silica functions more as a traditional abrasive and mechanically polishes the surface.

For chemical polishing with colloidal silica, the porous **BLACKCHEM 2 polishing pad** works very well because it allows the polishing process to generate more heat and thus enhances the chemical polishing rate. **GOLDPAD and TEXPAN polishing cloths** are also very useful for polishing glass, ceramics and composites utilizing a combination of colloidal silica and diamond.

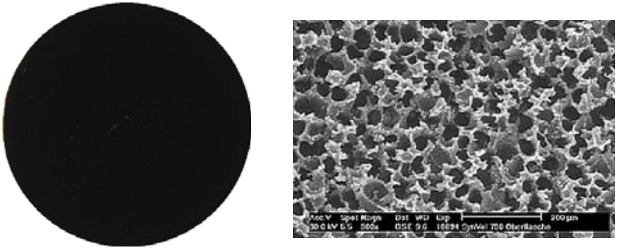


Figure 6-12 BLACKCHEM 2 Polishing Pad and SEM image.

Colloidal silica suspensions also have very good dispersion, thus the particles are usually very uniform and work well for polishing metals. However, for polishing metals, care must be taken so that the colloidal silica does not dry out because this can result in the colloidal silica crystallizing and producing much larger abrasive particles.

The other key to successful polishing with colloidal silica is to clean the specimen surface immediately after the polishing step. The most basic cleaning process should take place, which includes running clean particle free water on the polishing pad for the last 15-30 seconds of the polishing step.

If cleaning is not accomplished before the colloidal silica crystallizes on the specimen surface, then either the polishing process should be repeated or the sample should be cleaned with a caustic cleaning solution. A commonly used cleaning solution for most materials is a mixture of ammonia (NH_4OH) with hydrogen peroxide. **Note:** this solution will react (or etch) copper and copper alloys.

6.2 ALTERNATIVE POLISHING TECHNIQUES

Often there are materials which do not polish very well by conventional polishing techniques.   The most common materials which are difficult to polish by standard metallographic techniques include extremely soft materials which smear or recrystallize at very low temperatures. These materials, if not polished properly, can end up with erroneous microstructural features. To deal with these materials, a number of specialized polishing operations or techniques have been developed over the years. Several of these techniques can aid in polishing other types of materials, as well.

The three most common alternative polishing techniques include:

1. Electrolytic polishing
2. Attack polishing
3. Vibratory polishing

6.2.1 Electrolytic Polishing

Basically electrolytic polishing electrochemically corrodes the specimen in a controlled fashion. See Chapter 7 for more details regarding electrolytic polishing and etching.

6.2.2 Attack polishing

For materials which easily embed abrasives and the grinding swarf material, attack polishing is very useful. This process incorporates one or more etching steps between the grinding and polishing steps. For example, tin is a metal that is very soft and embeds fractured SiC abrasives easily (Figure 6-13a). Successful preparation can be accomplished with this material by etching the sample in between each SiC grit size in order to get rid of the embedded abrasives (Figure 6-13b). By doing this, the coarser abrasives are not carried over to the next grinding step.

For attack polishing techniques, the chemical etching solution can be a standard etchant for the material, or something a little more aggressive. The goal is not to actually etch the sample; instead, it is to dissolve the surface layer. Thus the etching process is usually longer and more aggressive than a common etch.

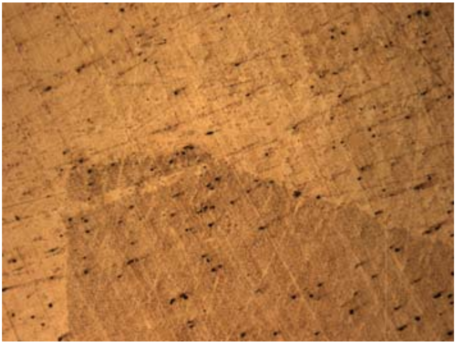


Figure 6-13a Tin with embedded abrasives, mag. 50X.

Figure 6-13b Tin after attack polishing with 2% Nital, mag. 50X.

6.2.3 Vibratory polishing

A unique mechanical polishing technique which still uses the standard polishing abrasives is known as vibratory polishing. Vibratory polishing uses a spring and motor to create the vibration. Essentially, the sample is vibrating up and down; however, by angling the spring mechanism, the specimen will rotate around the polishing bowl. Vibratory polishing is a very low deformation polishing technique; however, polishing action is usually very slow and it is not uncommon to polish samples for hours to days with this technique.

Figure 6-15 shows the effect of polishing a low carbon steel with standard polishing techniques compared to vibratory polishing. As can be seen in Figure 6-15b under DIC illumination, vibratory polishing is an effective polishing technique for removing surface deformation.

Figure 6-14 GIGA-0900 Vibratory Polisher.

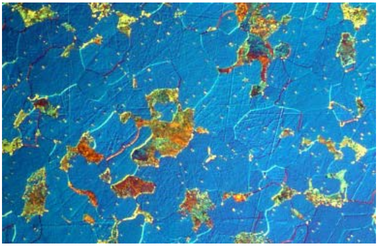


Figure 6-15a 1018 Steel, standard polishing, B.F., etchant 2% Nital, mag. 400X.

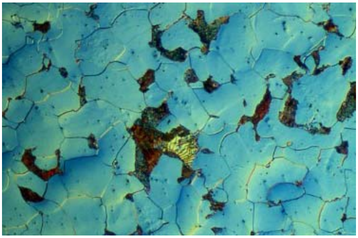


Figure 6-15b 1018 Steel, vibratory, polishing, 400X DIC., etchant 2% Nital.

6.3 FINAL POLISHING TROUBLESHOOTING

Although metallographic polishing artifacts can occur at any point within the polishing operation, they are commonly observed after final polishing.

Table XXI. Final Polishing Troubleshooting

Symptoms	**Cause**	**Action**
Scratches	-Abrasive contamination carry-over -Embedded abrasives -Friable inclusion or particles breaking loose	-Clean mount and specimen with an ultrasonic cleaner between preparation steps -Choose less friable abrasives (e.g. alumina) -Adjust polishing machine parameters so that the base and head speed are equal and in the same direction (e.g. 100/100 rpm)
Smearing	-Soft materials with low recrystallization temperatures do not work harden and can easily smear and cover up microstructural features	- Use a softer higher napped polishing cloth -Adjust polishing machine parameters so that the base and head speed are equal and in the same direction (e.g. 100/100 rpm) - Consider alternative polishing techniques such as etch-polishing, electrolytic polishing or vibratory polishing
Comet tails	-Fractured / embedded abrasives - Brittle inclusions -Too high a relative polishing velocity	-Choose a less friable fixed abrasive such as alumina or diamond lapping films -Combine CMP polishing with standard abrasive polishing -Adjust polishing machine parameters so that the base and head speed are equal and in the same direction (e.g. 100/100 rpm)
Polishing relief	-Materials of different hardness' polish at different rates -Too high a relative polishing velocity	-Use harder or lower-napped polishing pads -Plain backed lapping films also improve flatness -Incorporate CMP polishing with mechanical polishing -Adjust polishing machine parameters so that the base and head speed are equal and in the same direction (e.g. 100/100 rpm)

Table XXI. Final Polishing Trouble Shooting (continued)

Symptoms	**Cause**	**Action**
Embedded abrasives	-Abrasives fracturing and embedding into soft materials	-Choose a less friable abrasive such as alumina or diamond with a fixed bond (e.g. alumina grinding papers)
Edge rounding	-The edge of the specimen is polished faster then the body of the specimen	-Use harder or lower-napped polishing surfaces such as woven polishing pads or lapping films -Use a harder mounting resin
Pull-out	-Brittle constituents fracture and break out -Too high a relative polishing velocity	-Use CMP polishing for ceramic materials -Use finer abrasives for initial grinding -Use lapping films -Adjust polishing machine parameters so that the base and head speed are equal and in the same direction (e.g. 100/100 rpm)
Gaps	-Mounting does not adhere to the specimen	-Clean surface prior to mounting -Use glass-filled mounting compounds to reduce resin shrinkage -Cure castable mounting resins at lower temperatures
Porosity	-True porosity after correct polishing will have sharp edges. Rounded edges produce false porosity data	-Use harder or lower-napped polishing pads -Use CMP polishing with standard abrasives -Minimize final polishing time
Cracks	-Improper sectioning, mounting or rough grinding	-Minimize damage at cutting and rough grinding by using the smallest possible abrasive size -Reduce mounting pressure, or use castable mounting techniques
Staining	-Gaps between the mount and specimen -Unfilled cracks or porosity in specimen	-Use a lower shrinkage mounting compound -Use vacuum impregnation and castable mounting
Matted finish	-Polycrystalline alumina coating the specimen surface	-Clean with soapy water and cotton ball
Crystallized colloidal silica on surface of specimen	-Colloidal silica drying on the specimen surface	-Re-polish the specimen and clean the polishing cloth/specimen for the last 15-30 seconds with distilled water -Clean with an ammonia/hydrogen peroxide cleaning solution

6.3.1 Scratches

Description: Scratches remaining on the final polished surface are either due to previous abrasive contamination or from inclusions in the specimen breaking loose and damaging the surface.

Example: Very soft metals, such as a eutectic composition of 58% bismuth, 42% tin alloy, can scratch and smear very easily. Any fractured grinding abrasives can also easily embed into the samples.

Solution: Alternate between polishing with polycrystalline alumina and etching with 2% Nital and set machine parameters so that the base and head speeds are equal and in the same direction. Vibratory polishing will also eliminate the scratches.

Figure 6-16a Bismuth-tin alloy prior to polish-etch-polish.

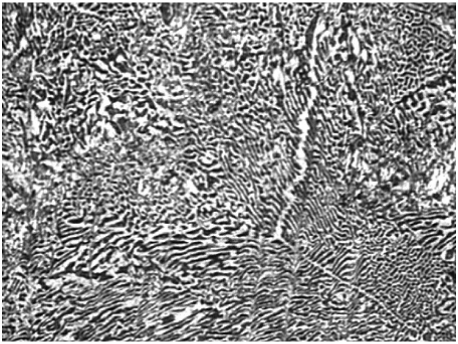


Figure 6-16b Bismuth-tin alloy after polish-etch-polish.

6.3.2 Smearing

Description: Very soft metals with very low recrystallization temperatures (tin, lead, zinc, austenitic steel) can smear during fine grinding and polishing. One of the problems with smearing is that it can cover up existing damage and is only revealed after later polishing steps or after specimen etching.

Example: Very soft metals with hard constituents , such as MMC composites, can smear very easily when using very hard polishing pads.

Solution: Use a softer or higher-napped polishing pad and increase lubrication during polishing. Adjust polishing machine parameters so that head and base speeds are equal and in the same direction.

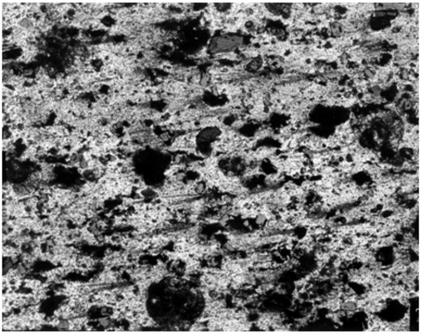


Figure 6-17a Smearing with hard polishing cloth.

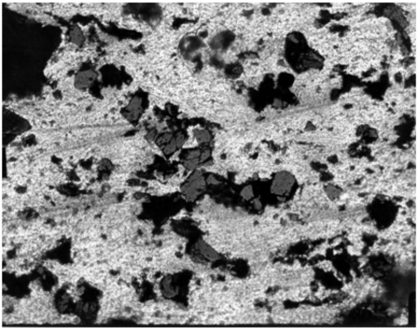


Figure 6-17b Smearing significantly reduced with softer polishing cloth.

6.3.3 Recrystallization

Description: Metals such as tin, lead, and zinc have recrystallization temperatures below room temperature. What this means is that these materials will not work harden at room temperature or above. Improper polishing of these metals can lead to false grain size analysis.

Example: Tin has a recrystallization temperature at -25°C, therefore specimen preparation must remove the recrystallized grains at the surface in order to obtain the correct microstructure.

Solution: Use a polish-etch-polishing technique to remove the recrystallized layer.

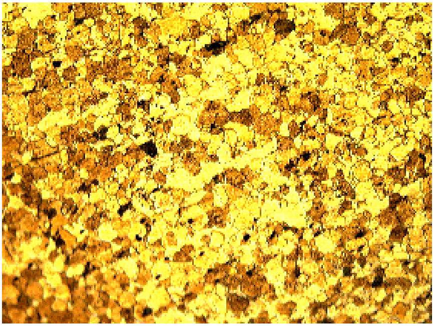


Figure 6-18a Recrystallized pure tin by standard polishing (fine grain structure).

Figure 6-18b Large tin grains following attack polishing with 2% Nital.

6.3.4 Comet Tails

Description: Comet tails are small scratches which can be traced back to either an embedded abrasive or particle in the specimen. It is quite common in very soft materials when SiC abrasives are used. Silicon carbide is normally a very good grinding abrasive; however, it fractures very easily. For very soft materials, the fractured abrasive can easily embed into the surface and completely change the ability to grind and polish these metals.

Example: Tin is a very soft metal that can easily be embedded with fractured SiC abrasives.

Solution: Use alumina grinding papers or lapping films. Adjust polishing machine parameters so that head and base speeds are equal and in the same direction.

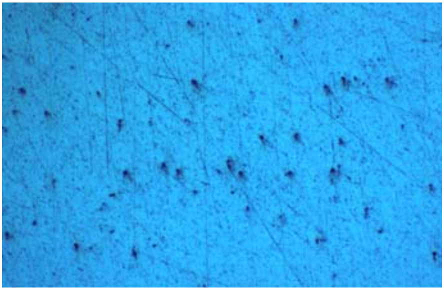


Figure 6-19a Comet tails in soft austenitic steel.

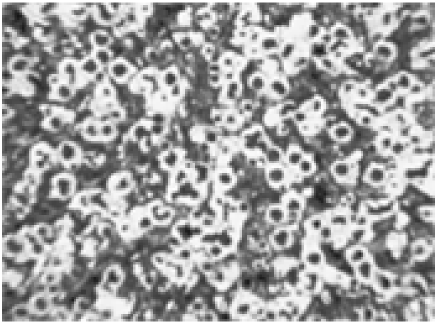


Figure 6-19b Retained brittle nodules in cast iron.

6.3.5 Embedded Abrasives

Description: Embedded abrasives can be very irritating because they are very difficult to remove after they get embedded. Embedded abrasives are quite a common problem in very soft materials (rhenium, niobium, aluminum, copper) when SiC abrasives are used.

Example: Rhenium is a very soft metal that can easily be embedded with fractured SiC abrasives.

Solution: Use fixed alumina grinding papers. Adjust polishing machine parameters so that head and base speeds are equal and in the same direction or use vibratory polishing.

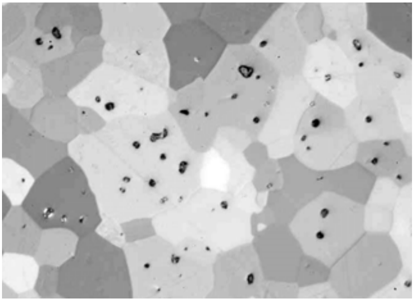


Figure 6-20a Embedded SiC abrasives in Rhenium.

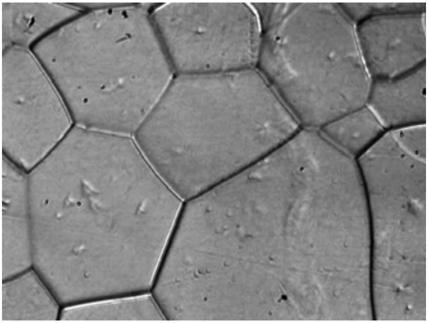


Figure 6-20b Rhenium polished with alumina abrasives (Photo courtesy of Climax Corporation).

6.3.6 Edge Rounding

Description: Edge rounding occurs when the edge of the specimen or a component of the specimen polishes at a different rate. When viewed at high optical magnifications, the specimen is not in focus across the entire plane.

Example: Titanium mounted in a soft mounting resin and over polished with a soft high-napped polishing pad.

Solution: Use low-napped polishing pads and CMP polishing conditions to equalize the polishing rates between the various components of the specimen. The use of lapping films can also improve flatness. Adjust polishing machine parameters so that head and base speeds are equal and in the same direction. Vibratory polishing is also an effective method to reduce edge rounding.

Figure 6-21 Edge rounding at edge of titanium and mount.

6.3.7 Polishing Relief

Description: Polishing relief occurs when two or more materials in the specimen polish at different rates. In some cases, if the relief is minimal, it can actually enhance the microstructural features without the need for etching.

Example: SiC particles in metal matrix. In this case, the slight polishing relief enhances the specimen microstructure.

Solution: To minimize relief, polishing on low-napped polishing pads using CMP polishing conditions can help equalize the polishing rates between the hard and soft components of the specimen. The use of lapping films can also improve flatness. Adjust polishing machine parameters so that head and base speeds are equal and in the same direction.

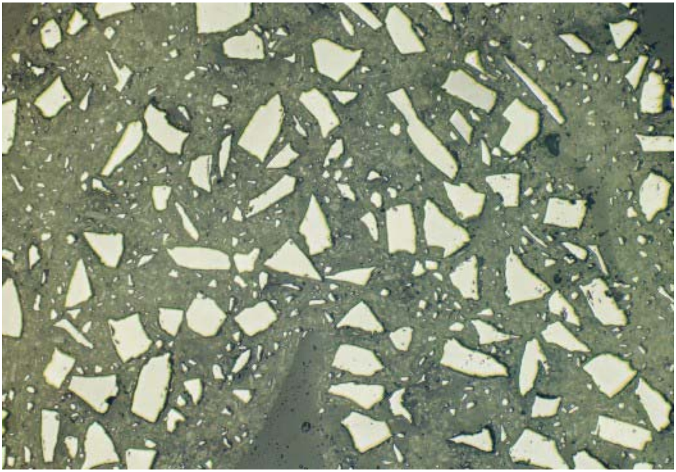


Figure 6-22 Specimen polishing relief for a SiC filter.

6.3.8 Pullout

Description: Pullout can refer to a number of artifacts, including grain pull-out in ceramics, inclusion pull-out in metals, and pull-out of brittle features such as nodules or flakes in cast iron.

Example: Silicon nitride is a very hard and tough ceramic. However, grinding with diamond abrasives can create a lot of grain pull-out which can be very difficult to remove.

Solution: Minimize initial damage during cutting and planar grinding. For ceramics, use colloidal silica to provide a CMP polishing action. For metals, use lapping films or low-napped/woven pads with polycrystalline diamond. Adjust polishing machine parameters so that head and base speeds are equal and in the same direction.

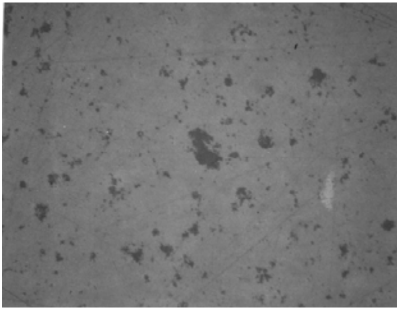


Figure 6-23a Si_3N_4 polished only with diamond abrasives.

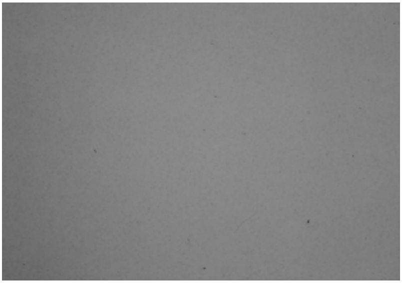


Figure 6-23b Si_3N_4 initially planar ground with diamond, followed by CMP polishing with colloidal silica.

6.3.9 Gaps and Staining

Description: Gaps usually refer to any spacing or void between the mount and specimen interface. One of the most common gaps is created when castable mounting resins get too hot and thus shrink away from the edge of the specimen. However, gaps can be inherent within the sample, and if, possible, these gaps or voids should be backfilled with vacuum impregnation. If not, the gaps or voids can entrap water, lubricants and/or etchants. When the sample is put under the intense light of the microscope, these fluids can leak out and corrode the sample.

Example: The following examples show the effect of having gaps in the material and the resulting staining and corrosion issues.

Solution: Improve mounting process to eliminate gaps at the specimen edge, use vacuum impregnation for internal voids and gaps, ultrasonically clean specimen with an alcohol cleaning solution and dry under vacuum.

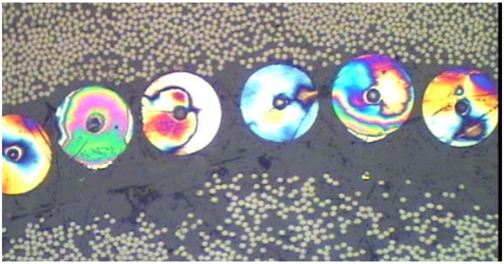


Figure 6-24a Boron-graphite golf shaft, stained.

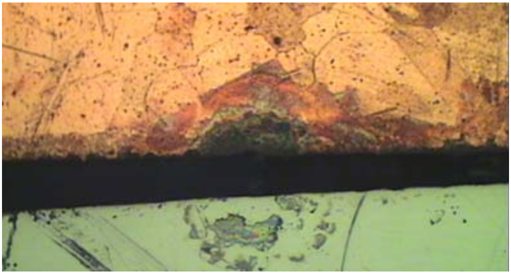


Figure 6-24b Gap and stain in copper/stainless steel weld.

6.3.10 Porosity and cracks

Description: Ceramics, powder metals and thermal spray components are examples of materials which can contain various levels of porosity. Marginal metallographic specimen preparation can influence this porosity by rounding the edges of the pores or cracks.

Example: The following examples (Figures 6-25a, 6-25b) show porosity in both a ceramic and powder metal.

Solution: Polish with low-napped polishing pads to reduce edge rounding at the pores.

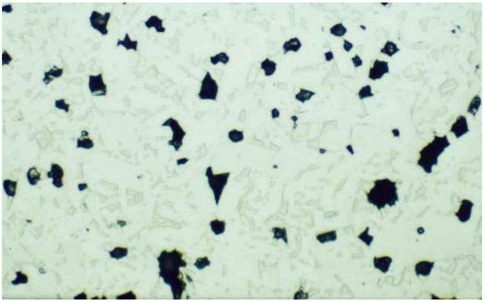


Figure 6-25a Cordierite ceramic with sharp pore edges for correct pore size analysis.

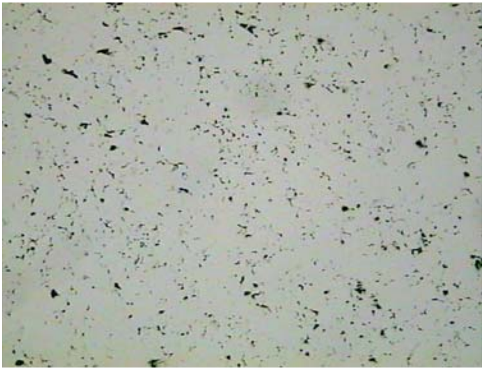


Figure 6-25b Powder metallurgy porosity for sintered iron specimen.

CHAPTER 7
Electrolytic Polishing

7.0 ELECTROLYTIC POLISHING

A less commonly used polishing technique is electrolytic polishing. Electrolytic polishing/etching, commonly known as anodic dissolution, is perhaps the best way to polish very soft materials which are prone to smearing and deformation. Materials that work well for electropolishing or etching include soft austenitic stainless steels, aluminum and aluminum alloys, copper and copper alloys, among others. The primary requirement for electropolishing is that the specimen be conductive.

Electrolytic polishing/etching essentially dissolves the higher surface energy sites associated with rough surfaces by smoothing the rough ridges or peaks. This is accomplished by making the specimen surface the anode in an electrolytic cell.

The voltage-current density plot shown in Figure 7-1 represents the mechanism by which electrolytic etching and polishing occurs. In Zone I, the primary mechanism is the direct dissolution of the metal. In this region, etching occurs. Zone II represents the voltage-current density conditions where the metal begins to form a passivation layer. In Zone III, the passivation layer is stable and dissolution of the metal is primarily by diffusion through the passivated layer. In this region, the higher surface area peaks dissolve preferentially to the lower surface area valleys, thus providing a polishing, or smoothing, effect to the surface. By increasing the voltage into Zone IV, the passivation layer breaks down as the oxygen evolution occurs at the surface. In this zone, the metal will begin to pit.

7.1 SPECIMEN PREPARATION

It is recommended that the specimen be rough polished down to a 600 (P1200) or 1200 (P4000) grit SiC surface finish. The specimen/mount must be conductive and in contact with the electrolyte, as well as the anode connection.

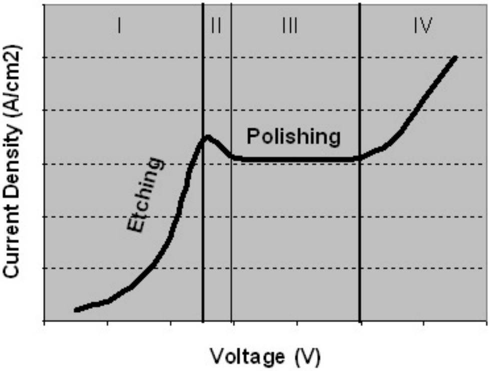


Figure 7-1 Electrolytic etching and polishing conditions.

7.2 SAFETY PRECAUTIONS

Electrolytes suitable for metallographic electrolytic polishing and etching are usually mixtures of acids (such as phosphoric, sulfuric and perchloric in ionizing solutions (such as water, acetic acid or alcohol). Glycerol, butyl glycol, urea, etc. are added to increase the viscosity. Metals which form highly soluble hydroxides are prepared with alkaline solutions, while those forming highly soluble cyanides are treated in cyanides. Most of the electrolytes used are relatively harmless when handled according to known common-sense precautions. However, mixtures of perchloric acid are particularly prone to decompose violently and should, therefore, be treated with extreme care.

7.2.1 Perchloric Acid ($HClO_4$) Electrolyte Precautions

Caution must be observed when electrolytically etching with perchloric acid electrolytes because of the possibility of explosion. The primary factors which lead to unsafe use of perchloric acid electrolytes include:

- Electrolyte temperature too high (>38°C)
- Perchloric acid concentration too high due to evaporation or improper mixing
- Reaction with certain common mounting materials (phenolics, acrylics and cellulose based resins).

The tendency of perchloric acid mixtures to explode are related to concentration and temperature. Concentrations above 35% perchloric acid become extremely dangerous. If the operator is not careful, a dangerous condition can occur through evaporation of the water or additives to the electrolyte. Likewise, temperatures greater than 38°C make perchloric acid less stable.

It is also recommended that specimens not be mounted in phenol-formaldehyde (phenolics), acrylic-resins or cellulose-base insulating lacquers. These materials produce very violent reactions with perchloric acid and may result in an explosion. However, polyethylene, polystyrene, epoxy resins, and polyvinyl chloride can be used as mounting materials for perchloric acid solutions without danger.

When working with perchloric acid, take precaution to avoid the explosive conditions listed above. In addition, before working with perchloric acid electrolytes they should be stirred and cooled for additional safety.

7.3 ELECTROLYTIC EQUIPMENT

Electrolytic polishers are composed of the following elements (Figure 7-2):
- Polishing cell
- Anode connection arm
- Cathode mask (stainless steel or platinum are most common)
- Electrolyte circulating pump
- Power controller for varying voltage and/or current

Figure 7-2 Electropolisher cell and control unit (Photo courtesy of Remet Italy).

7.4 ELECTROLYTE SOLUTIONS

Table XXII. Electrolyte Solution Guidelines

Material	Etchant	Procedure
Aluminum Pure Al, Al-Cu, Al-Mn, Al-Mg, Al-Mg-Si alloys (1)	90 ml DI water 10 ml H_3PO_4	5-10 s, 1-8 V DC, Stainless steel cathode
Beryllium and alloys (2)	294 ml ethylene glycol 4 ml HCl 2 ml HNO_3	6 min, 30°C (85°F), 13-20 V DC, Stainless steel cathode
Beryllium grain boundary etch. Also used to increase contrast in polarized light (2)	100 ml H_3PO_4 30 ml glycerol 30 ml ethanol (96 %) 2.5 ml H_2SO_4	1 min, cool (10°C, 50°F), 25 V DC, Stainless steel or Mo cathode
Boride ceramics, TaB_2, LaB4 (3)	10 ml DI water 1-2 gm NaOH	Few seconds to minutes, 10-15 V DC, Stainless steel cathode
Boron carbide (B_4C) and B_4C composites (4)	100 ml DI water 1 gm KOH	30-60 s, 30-60 V, 3 A/cm^2, V2A steel cathode, room temperature
Boron carbide (5)	10 ml DI water 0.1 gm KOH	40 V DC, 3 A/cm^2, Stainless steel cathode, Move specimen
Cadmium (Cd), In (6)	100 ml DI water 200 ml glycerol 200 ml H_3PO_4	5-10 min, 8-9 V DC, Cd cathode
Carbide ceramics - TiC, TaC (7)	10 ml DI water 2 gm KOH	2-30 s, 2 V DC, 30-60 mA/cm^2, Pt cathode, Move specimen

Table XXII. Electrolyte Solution Guidelines (Continued)

Material	Etchant	Procedure
Copper-Beta brasses, German silver, Monel, Cu-Ni alloys, Bronze (8).	950 ml DI water 50 ml H_2SO4 2 gm NaOH 15 gm iron (III) sulfate	Up to 15 s, 8-10 V DC, Cu cathode
Copper -All types of Cu. Cartridge brass. Tombac. Muntz metal, easily machinable brasses (8)	90 ml DI water 10 ml H_3PO_4	5-10 s, 1-8 V DC, Cu cathode
Copper and copper alloys (Cu), Beryllium copper and aluminum bronze (9)	1% CrO_3 99% water	3-6 s, 6 V, Al cathode
Copper -Al bronzes, Cu-Be alloys (8)	Aq. solution of chromium (VI) oxide (1 %)	3-6 s, 6 V DC, Al cathode
Germanium and its alloys. Grain boundaries (10)	100 ml DI water 10 gm oxalic acid	10-20 s, 4-6 V DC, Stainless steel cathode
Fe-Ni-Cr heat resistant casting alloys (Fe). Blackens sigma phase without outlining other phases (11).	5-6 gm KOH 100 ml DI water	1 s, 1.5 V, Stainless steel cathode, Room temperature
Fe-Ni-Cr heat resistant casting alloys (Fe). Stains austenite, then sigma phase, then carbide particles (11)	38 gm $Pb(C_2H_2O_2)^2x3H_2O$ DI water to make 100 ml	30 s, 1.5 V, Stainless steel cathode, Room temperature

Table XXII. Electrolyte Solution Guidelines (Continued)

Material	Etchant	Procedure
Cobalt base alloys (Co) Cobalt wrought alloys (13)	95 ml HCl (conc.) 5 ml H_2O_2	3 V, 10 s
Cobalt base alloys (Co) Heat resistant high temperature (superalloys) (Co-Cr-X type) (13)	25 ml HCl (conc.) 5-50 ml 10 % solution of chromic acid	6 V, 10 s, amount of CrO_3 determines activity
Co-Cr (40 %)-Ni-Fe alloys (Co) Co-Cr (40 %) - Ni-Fe alloys (13)	92 ml DI water 8 gm oxalic acid	6 V, 25-35°C (77-95°F), 200 mA/cm^2, 5-15 s
Copper and copper alloys (Cu) (14)	5 -14 % H_3PO_4 remainder water	10 s, 1-4 V
Copper and copper alloys (Cu) Coppers, brasses, bronzes, nickel silver.; color by electrolytic etching or with $FeCl_3$ etchants (15)	50 ml CrO_3 (10-15 %) 1-2 drops HCl	Immersion (add HCl at time of use).
Copper and copper alloys (Cu) Copper and alpha brass (16)	500 ml ortophosphoric acid (conc.) 500 ml DI water	A few seconds to 1 min., 0.8 V, 0.05 A/cm^2 (polishing 1.8 V, 0.12 A/cm^2), Cu cathode
Copper and copper alloys (Cu) Cartridge brass, free-cutting brass, admirality, gilding metal (14)	5 - 14 % H_3PO_4 remainder water	5-7 s, 1-8 V

Table XXII. Electrolyte Solution Guidelines (Continued)

Material	Etchant	Procedure
Niobium, molybdenum (17)	65 ml DI water 17 ml HNO_3 17 ml HF (40%)	Few seconds to minutes, 12-30 V DC, Pt cathode, Toxic
NiO ceramic (17)	60-70 ml DI water 25 ml HF (40 %) 25 ml glacial acetic acid	30-45 s, 2-4 mA/cm^2, 6-12 V DC, Stainless steel cathode, Toxic
Nickel and Ni base superalloys; gamma precipitates; Ti and Nb microsegregations (18)	85 ml H_3PO_4 5 ml H_2SO_4 8 gm chromium (VI) oxide	5-30 s, 10 V dc, Pt cathode, Toxic
Wrought Fe-Ni-Cr heat resisting alloys, Inconel X-750 (AISI 688), general structure, no pitting (12)	5 ml HNO_3 95 ml methanol Use colorless acid and absolute methanol	15-20 s, 5-10 V, Stainless steel cathode, Room temperature
Nickel-Grain contrast in Ni. Ni-Ag, Ni-Al, Ni-Cr, Ni-Cu, Ni-Fe, and Ni-Ti alloys (18)	85 ml DI water 10 ml HNO_3 5 ml glacial acetic acid	20-60 s, 1.5 V DC, Pt cathode, Do not store, Toxic
Nickel, Ni-Al alloys (18)	85 ml DI water 10 ml glycerol 5 ml HF	2-10 s, 2-3 V DC, Ni cathode, Toxic
Nickel and Ni base alloys, Ni-Cr, Ni-Fe alloys; Superalloys of the Nimonic type (19)	30 ml DI water 70 ml H_3PO_4 15 ml H_2SO_4	5-60 s, 2-10 V dc, Ni cathode
Nickel -Ni and Ni base alloys; Ni-Cr and Ni-Cr alloys; carbide inclusions (19)	100 ml DI water 2-50 ml H_2SO_4	5-15 s, 6 V DC, Pt cathode

Table XXII. Electrolyte Solution Guidelines (Continued)

Material	Etchant	Procedure
Nickel -Carbides in Ni-Cr alloys (19)	100 ml DI water 10 gm KCN	Approx. 3 min, 6 V DC, Pt cathode, Toxic
Nickel superalloy, Inconel 718 alloy (20)	50 ml HCl 40 ml HF 10 ml HNO_3 30 ml H_2O	3-5 s, 5 V DC, Stainless steel cathode
Nickel superalloy, Inconel 718 alloy (20)	10 ml HNO_3 90 ml ethanol (10 % Nital)	3-5 s, 5 V DC, Stainless steel cathode
Nickel superalloy, Inconel 718 alloy (20)	10 ml H_3PO_4 40 ml H_2SO_4 10 ml HNO_3 10 ml DI H_2O	3-5 s, 5 V DC, Stainless steel cathode
Nickel superalloy 718, Inconel 718 alloy (20)	1 part HCl 1 part H_2O	3-5 s, 5V DC, Stainless steel cathode
Nimonic alloys - Nimonic PK 31 (21)	45 parts of HCl 15 parts of HNO_3 40 parts of glycerol	5-15 s, 2-4 V DC, 0.5 A/dm^2, Nickel, stainless steel or 80Cr-20Ni cathode
Nimonic alloys - Nimonic PK 33, PK 50 Nimonic 901 (21)	20 % KOH solution	5-15 s, 2-4 V DC, 0.5 A/dm^2, Nickel, stainless steel or 80Cr-20Ni cathode
Nimonic alloys (Ni) - Nimonic alloys 75, 80A, 90, 93, 105 (21)	5 parts of HF 10 parts of glycerol 85 parts of DI water	5-15 s, 2-4 V DC, 0.5 A/dm^2, Nickel, stainless steel or 80Cr-20Ni cathode

Table XXII. Electrolyte Solution Guidelines (Continued)

Material	Etchant	Procedure
Osmium, palladium , iridium -Os base alloys, pure Pd and Pd alloys, Pt-Au alloys, Ir (22)	90 ml ethanol (96 %) 10 ml HCl	90 s, 10 V dc, Graphite cathode
Plutonium (Pu) - Pu and Pu base alloys (23)	20 ml methanol (95 %) 50 ml ethylene glycol 5 ml HNO_3	2 min, 0.05 A/cm^2, Stainless steel cathode
Silicon carbide (24)	10 % aqueous oxalic acid	0.5 min, 10 V DC, 1 A/cm^2, Stainless steel cathode
Silicon carbide (25)	10 ml DI water 2 gm KOH	20 s, 6 V dc, 1 A/cm^2, Pt cathode, Move specimen
Silver (Ag) - Ag alloys (26)	10 ml DI water 10 gm citric acid	15 s to 1 min, 6 V DC, Ag cathode.; possibly 2-3 drops nitric acid
Sintered carbides with high content of Ti and Ta carbide; the carbides are etched (27)	2 gm KOH 10 ml DI water	2-30 s, 2 V, 30-60 A/cm^2, Pt cathode; agitate specimen or electrolyte
Austenitic stainless steels and high-alloy nickel steel (28)	8 gm oxalic acid 100 ml DI water	5-60 s, Pt or stainless steel cathode

Table XXII. Electrolyte Solution Guidelines (Continued)

Material	Etchant	Procedure
Titanium (Ti) - Pure Ti and Ti base alloys (29)	80 ml glacial acetic acid 5 ml perchloric acid (70 %)	1-5 min, 20-60 V DC, Stainless steel cathode, Toxic
Titanium (pure) (30)	25 ml DI water 390 ml methanol (95 %) Ethylene glycol 35 ml perchloric acid (70 %)	10-40 s, 5-10°C (40-50°F), 30-50 V DC, Stainless steel cathode, Toxic

CHAPTER 8
Metallographic Etching

8.0 ETCHING

The purpose of etching is to optically enhance the microstructural features such as grain size, phase identification and other microstructural features. Etching selectively alters these microstructural features based on composition, stress, or crystal structure. The most common technique for etching is selective chemical etching, and numerous formulations have been used over the years. Other techniques such as molten salt, electrolytic, thermal, plasma and magnetic etching have also found specialized applications.

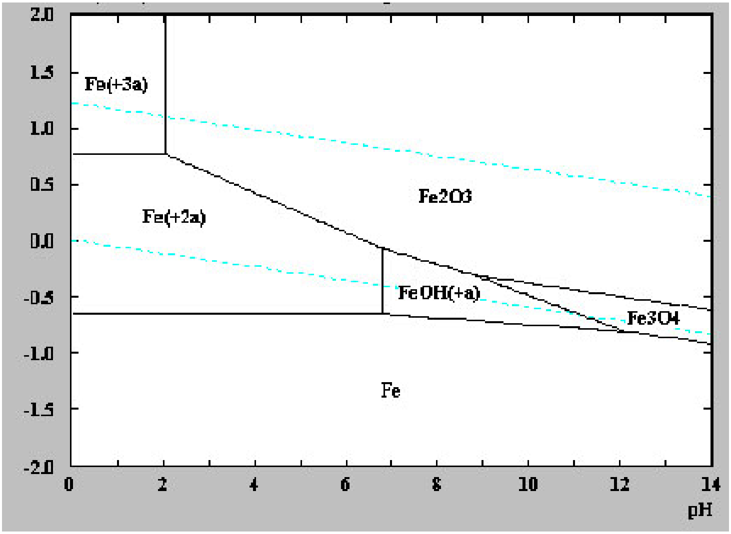


Figure 8-1 Pourbaix electrochemical diagram for iron.

8.1 CHEMICAL ETCHING

Chemical etching selectively attacks specific microstructural features. It generally consists of a mixture of acids or bases with oxidizing or reducing agents. For more technical information on selective chemical etching, consult corrosion books which discuss the relationship between pH and Eh (oxidation/reduction potentials). These diagrams are often known as Eh-pH diagrams or Pourbaix diagrams. Figure 8-1 shows the Pourbaix diagram for the iron-water system. As seen in Figure 8-1, iron dissolves over a wide range of pH values, however, only at a very limited oxidation potential for most of this range. Controlling this oxidation potential within this range could be difficult, so the best etching condition for iron would be at pH values below 2 and at high oxidation potentials (Eh). At these Eh and pH values, the most stable species is the ferric ion (Fe^{+3}). Over the years, numerous chemical etchants have been formulated. For specific etchant recommendations, refer to Chapter 11.

8.2 ELECTROLYTIC ETCHING

Electrolytic etching is another fairly common etching technique. It is similar to chemical etching in that acids and bases are used for modifying the pH. However, the electrochemical potential is controlled electrically by varying either the voltage or current externally as opposed to chemically. Electrolytic etching is often used for harder-to-etch specimens that do not respond well to basic chemical etching techniques. Electrolytic techniques require that the specimen be conductive and therefore they are limited primarily to metals.

The most common electrolytic etching equipment uses a two-electrode design (anode and cathode) with acids or bases used for the electrolyte. Procedures for this type of electrolytic etching are fairly common and can be found in Section 7.4.

8.3 MOLTEN SALT ETCHING

Molten salt etching is a combination of thermal and chemical etching techniques. Molten salt etching is useful for grain size analysis for hard to etch materials such as ceramics. The technique takes advantage of the higher internal energy associated at a material's grain boundaries. As a result of the higher melting temperature of molten salts, the higher energy at the grain boundaries are relieved, producing a rounded grain boundary edge; this can be observed by optical or electron microscope techniques (Figure 8-2).

Figure 8-2 SiAlON etched in KCl molten salt, mag. 5000X.

8.4 THERMAL ETCHING

Thermal etching is a useful technique for etching ceramic materials. Thermal etching is a technique that relieves the higher energy areas associated at the grain boundaries of a material. By heating and holding the temperature to just below its sintering temperature, the grain boundaries will seek a level of lower energy. The result is that the grain boundary edges become rounded, which are observable by optical or electron microscope techniques (Figure 8-3).

Depending upon the ceramic material, the atmospheric condition of the furnace may need to be controlled. For example, etching silicon nitride will require either a vacuum or an inert atmosphere of nitrogen or argon to prevent oxidation of the surface to silicon dioxide.

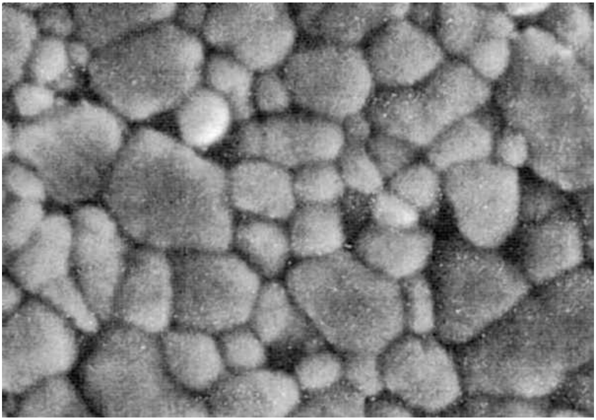


Figure 8-3 ZrO_2 thermally etched in air atmosphere, mag. 5000X.

TIP: To increase the contrast and reflectivity of ceramic materials, the specimen can be sputter coated with a metallic coating (Figure 8-4). This is particularly useful for higher magnification analysis.

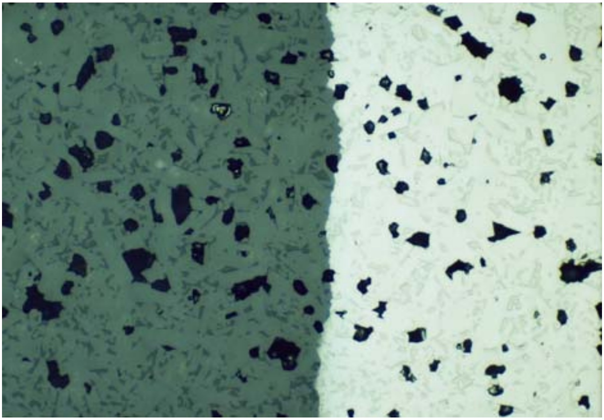


Figure 8-4 Cordierite ceramic as polished, right side sputter coated with gold to increase optical contrast, 400X.

CHAPTER 9
Microscopy and Image Analysis

9.0 MICROSCOPY

Figure 9-1 Inverted Metallurgical Microscope.

Optical microscopy using metallographic microscopes is a widely used technique for analyzing metallographic specimens. The typical magnification range for optical microscopes is 50 to 1000X, however higher magnifications are possible with specialized oil immersion lenses. The standard resolution for optical microscopes using air immersion lenses is between 0.5 to 10 micron. Optical microscopes use a number of different optical techniques to reveal specific microstructural features, including the following illumination techniques: brightfield, darkfield, polarized light, oblique (stereo) and differential interference contrast (DIC). Scanning electron microscopy is also used for metallographic analysis and has a resolution ranging from Angstroms to microns.

9.0.1 Definitions

Brightfield – an image condition where the background is light and the features are dark (high angle of illumination)
Darkfield – an image condition where the background is dark and the features are bright (low angle of illumination)
Depth of Field – the distance or depth at which the specimen surface will be in focus
Empty Magnification – the magnification limit where no additional information is obtained; increasing magnification beyond this limit only magnifies existing features
Numerical Aperture (N.A.) – measure of objective lens light-gathering ability (also determines the quality of the lens)
Resolution – the distance at which two individual features can be seen as individual objects
Working Distance – the distance between the objective lens and the specimen surface when the image is in focus

9.0.2 Resolution and Numerical Aperture (N.A.)

The most important components of the optical microscope are its objective lenses. The quality of these lens ability to gather light is characterized by the numerical aperture (N.A.)

N.A. = $\mu \sin \theta$

Where:
μ - refractive index of the medium in front of the objective ($\mu = 1$ for air)
θ - the half-angle subtended by the objective in front of the objective at the specimen (see Figure 9-2).

Figure 9-2 N.A. is the light-gathering capacity of the objective lens.

Resolving Power = $(2 * N.A.)/\lambda$

λ = wavelength of light used
λ = 0.54 micron – green light
λ = 0.1 Angstrom – electron beam

Limit of Resolution = $\lambda/(2 * N.A.)$

Total magnification = objective mag. * eyepiece mag. * tube factor mag.

9.0.3 Optical Filters

Optical filters are used to enhance the definition of the specimen image, especially for photographic film. The main types of optical filters include:

Neutral Density Filters - reduce the illumination intensity without affecting the color temperature
Green Monochromatic Filters - produce a single wavelength of light to ensure a sharp focus on black and white film
Blue Color Correction Filters - allow the operator to use daylight film with tungsten illumination and vice versa.
Color Compensating Filters - used to compensate for minor color temperature differences between the film and the illumination source

9.1 BRIGHTFIELD

Brightfield (B.F.) illumination is the most common illumination technique for metallographic analysis. The light path for B.F. illumination is from the source, through the objective, reflected off the surface, returning through the objective, and back to the eyepiece or camera. This type of illumination produces a bright background for flat surfaces, with the non-flat features (pores, edges, etched grain boundaries) being darker as light is reflected back at a different angle.

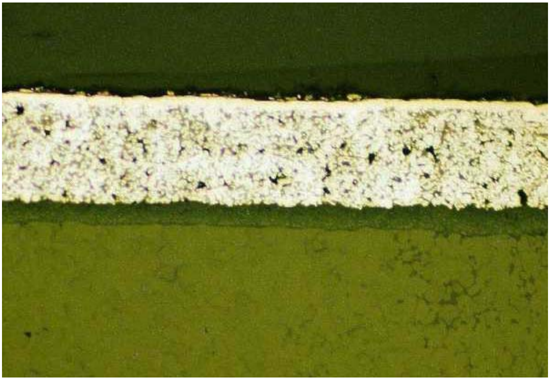


Figure 9-3 Aluminum nitride electronic substrate - Brightfield, 400X.

9.2 DARKFIELD

Darkfield (D.F.) illumination is a lesser known but powerful illumination technique. The light path for D.F. illumination is from the source, down the outside of the objective, reflected off the surface, returned through the objective and back to the eyepiece or camera. This type of illumination produces a dark background for flat surfaces, with the non-flat features (pores, edges, etched grain boundaries) being brighter as light is reflected at an angle back into the objective.

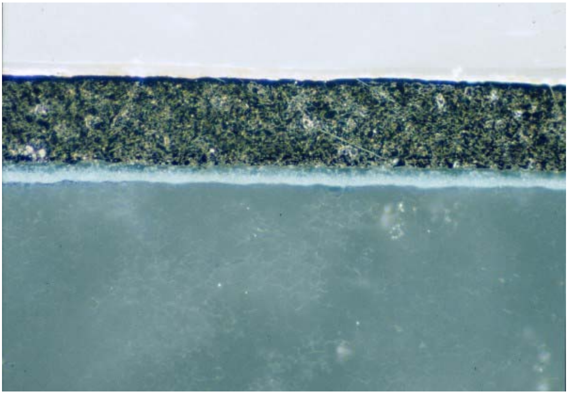


Figure 9-4 Aluminum nitride electronic substrate - Darkfield, 400X.

9.3 DIFFERENTIAL INTERFERENCE CONTRAST

Differential Interference Contrast (DIC) is a very useful illumination technique for providing enhanced specimen features. DIC uses a Normarski prism along with a polarizer in the 90° crossed positions. Essentially, two light beams are made to coincide at the focal plane of the objective, thus rendering height differences more visible as variations in color.

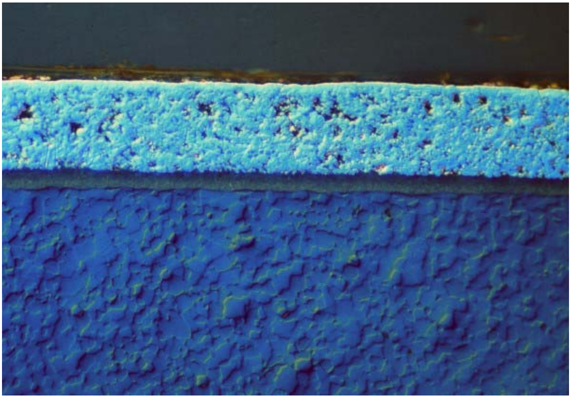


Figure 9-5 Aluminum nitride electronic substrate - Differential Interference Contrast (DIC), 400X.

9.4 METALLOGRAPHIC IMAGE ANALYSIS

Quantifying and documenting a materials microstructure can provide very useful information for process development, quality control and failure analysis applications. Stereological techniques are used to analyze and characterize 3-dimensional microstructural features from 2-dimensional images or planar specimen cross sections.

The most common stereological analysis includes: point counting, length, area and volume measurements; although, for automated image analysis, counting picture points has recently been added. The following list of measurements or calculations are used for determining a number of metallographic features:

$\bar{A}$ = average area of inclusions or particles, (μm^2)
A_A = area fraction of the inclusion or constituent

A_i = area of the detected feature
A_T = measurement area (field area, mm^2)
H_T = total project length in the hot-working direction of an inclusion or constituent in the field, microns
Ƚ = average length in the hot-working direction of the inclusion or constituent, (µm)
L_T = true length of scan lines, pixel lines, or grid lines (number of lines times the length of the lines divided by the magnification), mm
n = the number of fields measured
N_A =number of inclusions or constituents of a given type per unit area, mm^2
N_i = number of inclusions or constituent particles or the number of feature interceptions, in the field
N_L = number of interceptions of inclusions or constituent particles per unit length (mm) of scan lines, pixel lines, or grit lines
PP_i = the number of detected picture points
PP_T = total number of picture points in the field area
s = standard deviation
t = a multiplier related to the number of fields examined and used in conjunction with the standard deviation of the measurements to determine the 95% CI
V_V = volume fraction
X = mean of a measurement
X_i = an individual measurement
$\sum X$ = the sum of all of a particular measurement over n-fields
$\sum X^2$ = sum of all of the squares of a particular measurement over n-fields
λ = mean free path (µm) of an inclusion or constituent type perpendicular to the hot-working direction

95% CI = 95% confidence interval
% RA = relative accuracy, %

For stereological measurements:

Volume fraction = $V_V = A_A = A_i / A_T = PP_i / PP_T$

Number per unit area (inclusions) = $N_A = N_i / A_T$

Average length of each inclusion = $L = \sum H_T / \sum N_i$

Average area of each inclusion or particle = $\bar{A} = \bar{A}_A / \bar{N}_{\bar{A}}$
Mean free path or the mean edge-to-edge distance between inclusions (oxide and sulfide) or particle types, perpendicular to the hot-working axis:

$$\lambda = (1-\bar{A}_A)/ \bar{N}_{\bar{L}}$$

Several commonly used metallographic quantification procedures include the following:
- Grain size (ASTM E112, E930, E1181 and E1382)
- Phase analysis (ASTM E562, E1245)
- Nodularity (ASTM A247)
- Porosity (ASTM 562)
- Inclusion (ASTM E45, E1245)
- Decarburization (ASTM E1077)
- Coating thickness (ASTM B487)
- Weld analysis

9.4.1 Grain size (ASTM E112, E930, E1181)

A grain is defined as the individual crystal in a polycrystalline material. Although grain size is a 3-dimensional feature, it is measured from a 2-dimensional cross section of the material. ASTM (American Society for Testing Materials) provides a number of internationally recognized standards for measuring and classifying a materials grain size.

- **ASTM E112** - Standard Test Methods for Determining Average Grain Size (31)
- **ASTM 930** - Standard Test Methods for Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size) (32)
- **ASTM E1181** - Standard Test Methods for Characterizing Duplex Grain Sizes (33)
- **ASTM E1382** - Standard Test Methods for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis (34)

ASTM E112 describes several procedures for measuring grain size, including the Comparison procedure, Planimetric (Jeffries) procedure, and general Intercept procedures. The Comparison procedure is useful for completely recrystallized materials with equiaxed grains and uses a set of standardized charts that can be obtained or purchased from ASTM. These charts are used to compare the etched specimens microstructure, at the same magnification, to the appropriate comparison chart (31).

For the Planimetric method, a rectangle or circle having a known area (5000 mm^2) is placed over a micrograph of the etched specimen and the number of full grains are counted and the number of grains that intersect the circumference of the area are counted and multiplied by 1/2, this gives the total number of grains. This number is then multiplied by the Jeffries multiplier which is based on the magnification (note: proper magnification requires at least 50 grains).

The Intercept procedure is recommended for all structures which do not have uniform equiaxed grain structure. The Heyn Lineal Intercept Procedure (31) counts the number of grain boundary intercepts along a straight line. Another intersect technique utilities a circular test line.

Note: for determining grain size, twin boundaries should be removed from the calculation.

ASTM E930 is used to measure the grain size for materials with very large grain structures when there are not enough grains to use ASTM E112. For example, galvanized coatings can have very large grain structures (32). This standard determines the largest observed grain in the sample, often referred to ALA (as large as) grain size. The methods used to determine the ALA grain size include measuring the largest grain with a caliper or by photographing the largest grain at the highest magnification which shows the entire grain. For the caliper method the largest diameter and the largest diameter perpendicular to this line are measured. These two numbers are multiplied together and then multiplied by 0.785 to give an elliptical area. This number is divided by the square of the magnification to give the grain size at a magnification of 1X. Using the appropriate ASTM table, the ASTM grain size number can be determined. Another techniques uses a photograph with an ASTM overlay. The number of grid intersections are counted and converted to grain size number.

ASTM E1181 is the standard used for characterizing grain sizes for materials which have two or more distinctive grains sizes (33).

ASTM E1382 is the standard which covers the procedures for automatically determining the mean grain size, the distribution of grain intercept length, or grain areas. The primary issue for semi-automatic and automatic image analysis is proper specimen preparation, including proper grinding, polishing and etching. The resulting microstructure should fully and uniformly reveal the grain boundaries (34).

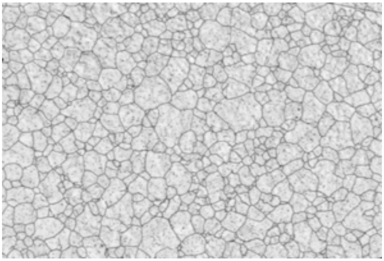


Figure 9-6a. Grain size analysis - polished and etched.

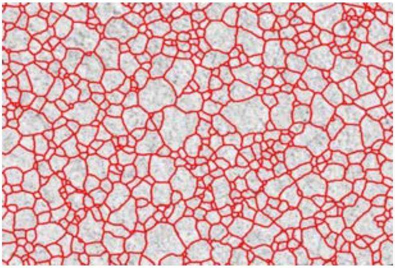
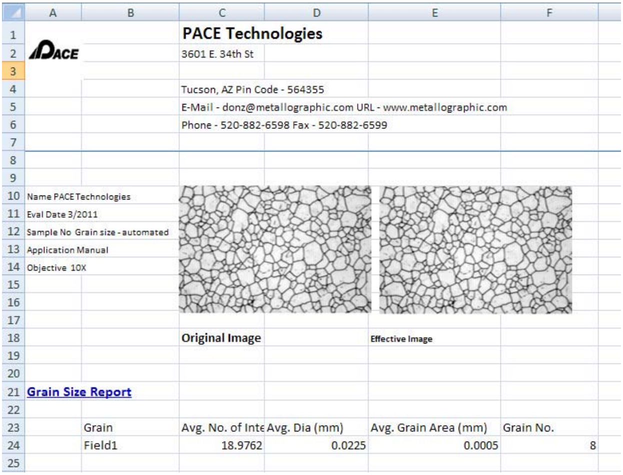


Figure 9-6b. Grain size analysis - detected image.



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Name PACE Technologies
Eval Date 3/2011
Sample No Grain size - automated
Application Manual
Objective 10X

Grain Size Report

Grain	Avg. No. of Inte	Avg. Dia (mm)	Avg. Grain Area (mm)	Grain No.
Field1	18.9762	0.0225	0.0005	8

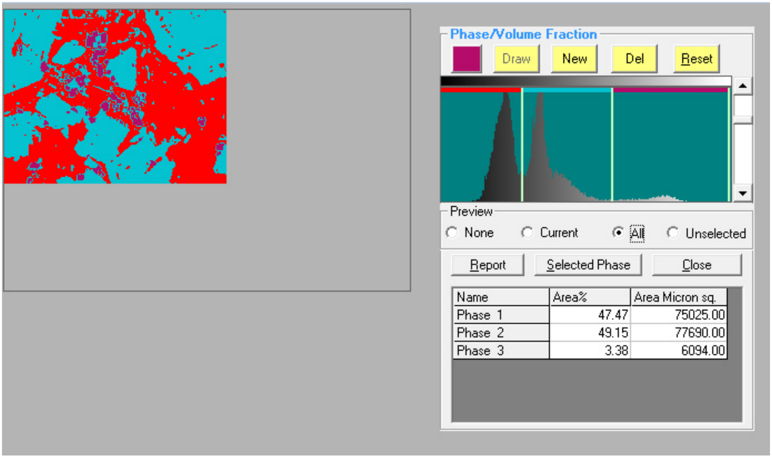
Figure 9-6c. Grain size analysis - report.

9.4.2 Phase Analysis (ASTM E562, E1245)

Phases are defined as physically homogenous and distinct constituents of the material. Phase analysis can be characterized and measured using area or volume fraction measurements per ASTM E562 (Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count) (35) or ASTM E1245 (Standard Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis) (36).

Common measurements used in phase analysis include: length, area, number, volume fraction, mean free path, number of detected picture points, and 95% CI – confidence interval.

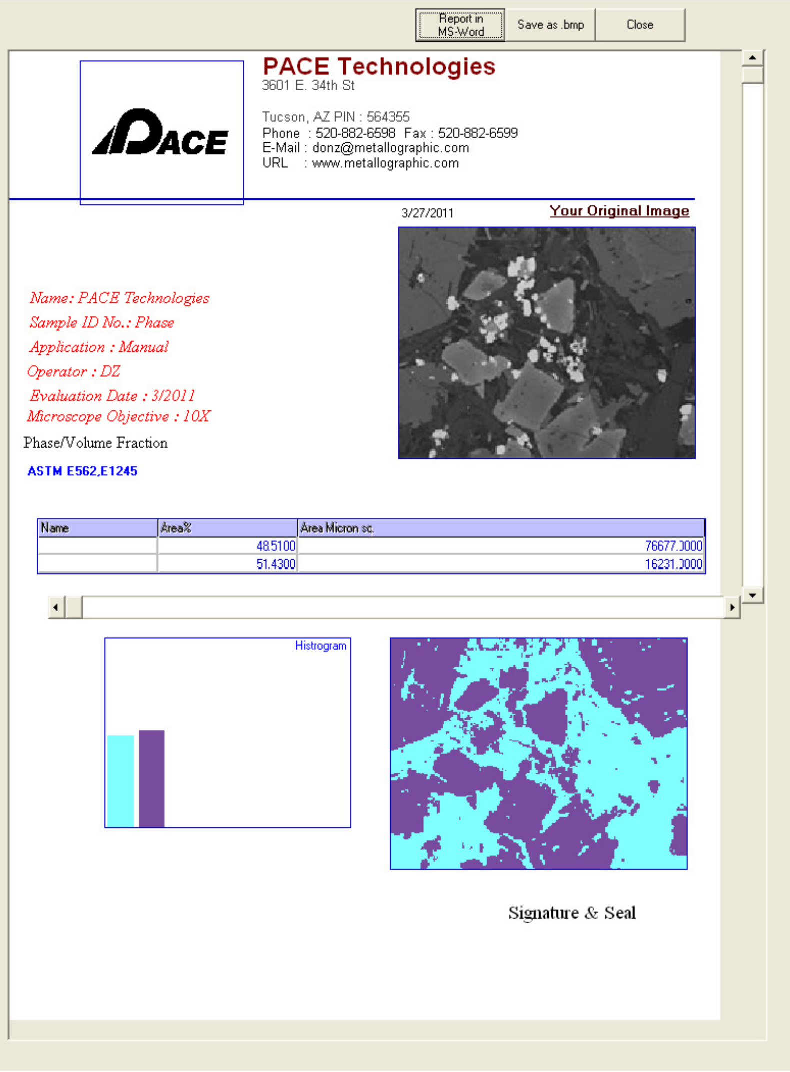
Examples where phase analysis are used include stereological measurements that describe the amount, number, size, and spacing of the indigenous inclusions (sulfides, oxides and silicates) in steel, porosity, and the analysis of any discrete second-phase constituent in the material.



Preview
○ None ○ Current ● All ○ Unselected

Name	Area%	Area Micron sq.
Phase 1	47.47	75025.00
Phase 2	49.15	77690.00
Phase 3	3.38	6094.00

Figure 9-7a. Phase analysis - detected image.



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3/27/2011

Your Original Image

Name: PACE Technologies
Sample ID No.: Phase
Application : Manual
Operator : DZ
Evaluation Date : 3/2011
Microscope Objective : 10X

Phase/Volume Fraction

ASTM E562,E1245

Name	Area%	Area Micron sq.
	48.5100	76677.3000
	51.4300	16231.3000

Signature & Seal

Figure 9-7b. Phase analysis - report.

9.4.3 Nodularity (ASTM A247)

Nodularity describes the type and distribution of graphite in cast irons. ASTM A247 (Standard Test Method for Evaluating the Microstructure of Graphite in Iron Castings) (37) is used to classify and characterize the graphite for all iron-carbon alloys containing graphite particles. This method can be applied to gray irons, malleable irons, and ductile (nodular) irons.

Quantification of cast irons can be described with three classifications: graphite form (Roman number I through VII), graphite distribution (letter A-E), and graphite size (1-largest to 8-smallest). Types I-VI are for nodular cast iron and Type VII would be for the graphite flakes in gray cast irons. Classification of the graphite is typically accomplished by comparison with ASTM Plate I for the type, ASTM Plate II for the distribution of the graphite, and ASTM Plate III would reference the size of the graphite.

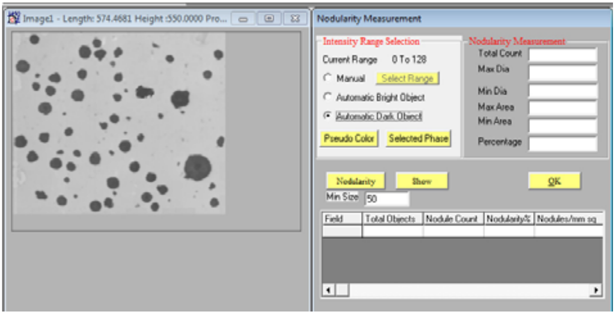


Image1 - Length: 574.4681 Height :550.0000 Pro...

Nodularity Measurement

Intensity Range Selection

Current Range 0 To 128

○ Manual

○ Automatic Bright Object

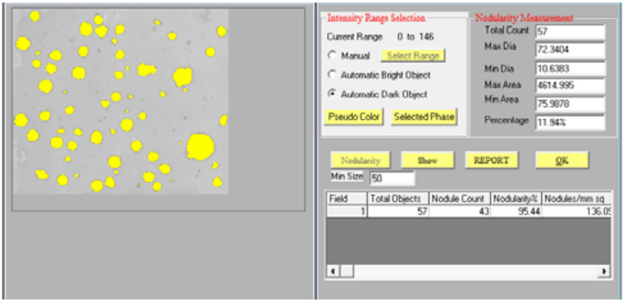
● Automatic Dark Object

Nodularity Measurement

Min Size 50

Field	Total Objects	Nodule Count	Nodularity%	Nodules/mm sq

Figure 9-8a. Nodular graphite Type I - as polished.



Intensity Range Selection

Current Range 0 to 146

○ Manual

○ Automatic Bright Object

● Automatic Dark Object

Nodularity Measurement

Total Count 57

Max Dia 72.3404

Min Dia 10.6383

Max Area 4614.995

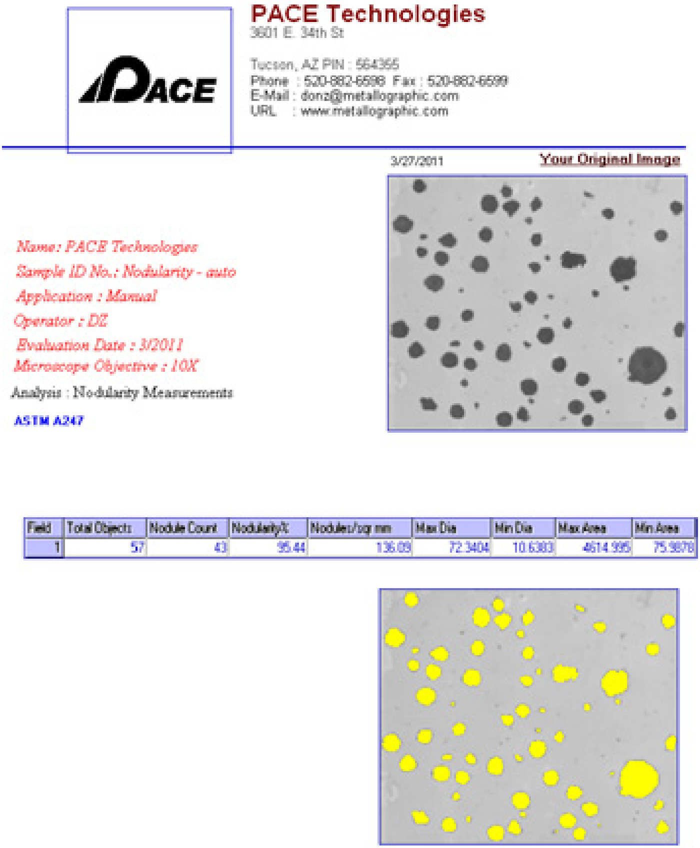
Min Area 75.9878

Percentage 11.94%

Min Size 50

Field	Total Objects	Nodule Count	Nodularity%	Nodules/mm sq
1	57	43	95.44	136.0

Figure 9-8b. Nodular graphite Type I - as detected.



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3/27/2011

Your Original Image

Name: PACE Technologies
Sample ID No.: Nodularity - auto
Application : Manual
Operator : DZ
Evaluation Date : 3/2011
Microscope Objective : 10X
Analysis : Nodularity Measurements

ASTM A247

Field	Total Objects	Nodule Count	Nodularity%	Nodules/sq mm	Max Dia	Min Dia	Max Area	Min Area
1	57	43	95.44	136.09	72.3404	10.6383	4614.995	75.9878

Figure 9-8c. Nodular graphite Type I size and distribution - report.

9.4.4 Porosity (ASTM E562, E1245)

Porosity are voids in the material caused by entrapped air and incomplete or poor sintering. Porosity can be measured as a volume fraction, either manually using ASTM E562 (35) or with automated image analysis using ASTM E1245.

ASTM E562 (Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count) (35) is a point counting method using a clear plastic test grid or an eyepiece reticle with a regular array of test points overlaid on the image. The number of test point falling within the phase or constituent of interest are counted and divided by the total number of grid points.

P_i – point count on the ith field

$P_p(i) = P_i/P_T*100\%$ = percentage of grid points, in the constituent observed on the ith field

P_T = total number of points in the test grid (ASTM standard test grits $P_T = 16$, 25, 49 or 100 points)

n = number of fields counted

t = a multiplier related to the number of fields examined and used in conjunction with the standard deviation of the measurements to determine the 95% CI (see table 1 p. 630 ASTM Standard 3.01, 2010)(35)

Arithmetic average of $Ᵽ_p = 1/n(\sum P_p(i))$

s = estimate of the standard deviation $s = [1/(n-1) \sum\{P_p(i) - Ᵽ_p\}^2]^{1/2}$

95% CI = 95% confidence interval = $t * s/(n)^{½}$

Volume fraction $V_V = Ᵽ_p$ +/- 95% CI

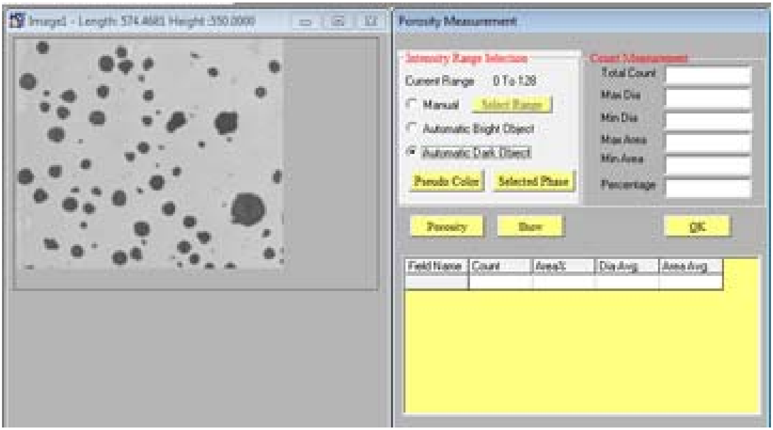


Image1 - Length: 574.4681 Height :550.0000

Porosity Measurement

Intensity Range Selection

Current Range 0 To 128

● Automatic Dark Object

Count Measurement

Total Count

Max Dia

Min Dia

Max Area

Min Area

Percentage

Field Name	Count	Area%	Dia Avg.	Area Avg.

Figure 9-9a. Porous sample - as polished.

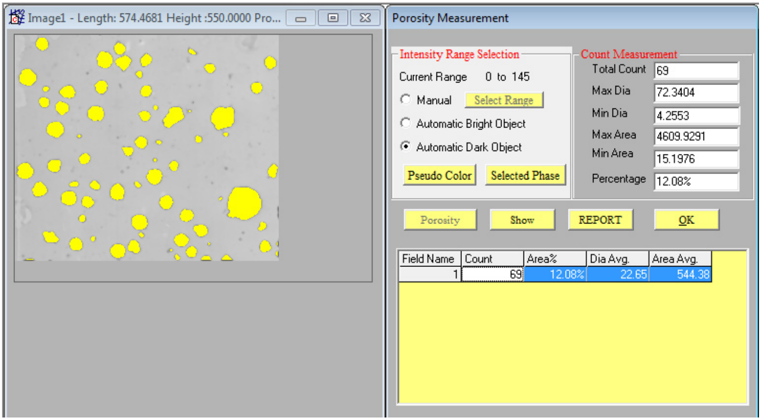


Image1 - Length: 574.4681 Height :550.0000 Pro...

Porosity Measurement

Intensity Range Selection

Current Range 0 to 145

● Automatic Dark Object

Count Measurement

Total Count 69

Max Dia 72.3404

Min Dia 4.2553

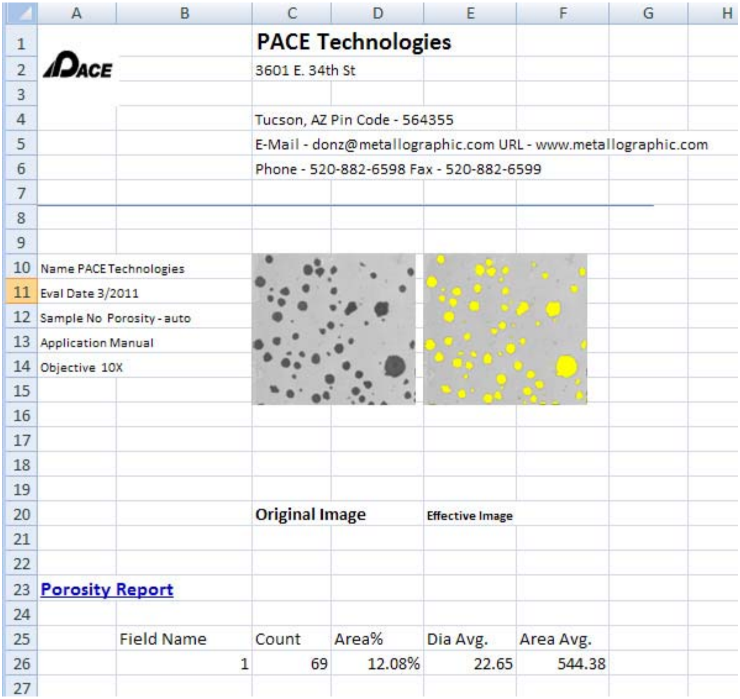
Max Area 4609.9291

Min Area 15.1976

Percentage 12.08%

Field Name	Count	Area%	Dia Avg.	Area Avg.
1	69	12.08%	22.65	544.38

Figure 9-9b. Porous sample - as detected.



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Name PACE Technologies

Eval Date 3/2011

Sample No Porosity - auto

Application Manual

Objective 10X

Original Image

Effective Image

Porosity Report

Field Name	Count	Area%	Dia Avg.	Area Avg.
1	69	12.08%	22.65	544.38

Figure 9-9c. Porous sample - report.

9.4.5 Inclusion rating (ASTM E45)

Inclusions are particles of foreign material that are insoluble in the metal or materials matrix. For steels, common inclusions include oxides, sulfides or silicates; however, any foreign substance can be classified as an inclusion.

ASTM E45 (Standard Test Methods for Determining the Inclusion Content of Steel) (38) is used to characterize the type, size and severity of the inclusions in wrought steel. ASTM E45 describes the JK-type inclusion rating system. The JK-type inclusion rating system first characterizes the type of inclusion (Type A-D):

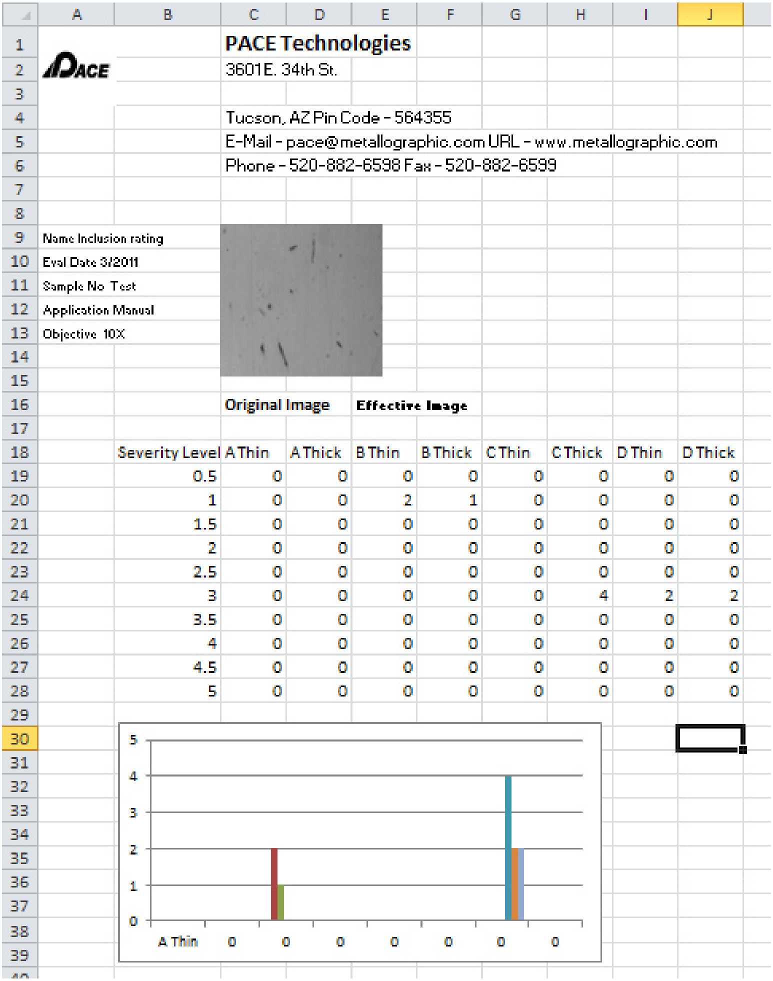
Type A-sulfide type
Type B-alumina type
Type C-silicate type
Type D-globular oxide type

Type A and C are very similar in size and shape, however Type A-Sulfide are light gray which Type S-Silicate are black when viewed under Brightfield illumination.

Type B stringers consist of a number (at least three) round or angular oxide particles with aspect ratios less than 2 that are aligned nearly parallel to the deformation axis.

The second characterization parameter is thickness: designated H-heavy, T-thin.

The third characterization parameter is “Severity Level” and are partitioned based on the number or length of the particles present in a 0.50 mm^2 field of view (38).



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Name Inclusion rating
Eval Date 3/2011
Sample No Test
Application Manual
Objective 10X

Original Image **Effective Image**

Severity Level	A Thin	A Thick	B Thin	B Thick	C Thin	C Thick	D Thin	D Thick
0.5	0	0	0	0	0	0	0	0
1	0	0	2	1	0	0	0	0
1.5	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0
2.5	0	0	0	0	0	0	0	0
3	0	0	0	0	0	4	2	2
3.5	0	0	0	0	0	0	0	0
4	0	0	0	0	0	0	0	0
4.5	0	0	0	0	0	0	0	0
5	0	0	0	0	0	0	0	0

Figure 9-10. Inclusion rating - report.

9.4.6 Decarburization (ASTM E1077)

Decarburization is the loss of carbon at the metals surface due to chemical reaction(s) with the contacting media. Decarburization can over time significantly change the surface properties of the metal. ASTM E1077 (Standard Test Methods for Estimating the Depth of Decarburization of Steel Specimens) provides the guidelines for estimating the average or greatest depth of decarburization in hardened or non-hardened steel products (39).

Metallographic analysis of a properly polished and etched sample is considered an acceptable technique for determining decarburization for heated-treated, spherodize-annealed, cold-worked, as-hot rolled, as-forged, annealed, or normalized steel specimens. The depth of decarburization can be determined by the observed changes in the microstructural cross-section due to changes in the carbon content.

ASTM defines the following terms (39):
Average depth of decarburization – the mean value of 5 or more measurements of the total depth of decarburization.

Average free-ferrite depth – the mean value of 5 or more measurements of the depth of complete decarburization

Complete decarburization – loss of carbon content at the surface of a steel specimen to a level below the solubility limit of carbon in ferrite so that only ferrite is present.

Partial decarburization – loss of carbon content at the surface of a steel specimen to a level less than the bulk carbon content of the unaffected interior by greater then the room temperature solubility limit of carbon in ferrite. The partial decarburization zone would contain both ferrite and pearlite.

Total depth of decarburization – the perpendicular distance from the specimen surface to that location in the interior where the bulk carbon content is reached; that is, the sum of the depths of complete and partial decarburization. For heat-treated specimens, the presence of non-martensitic structures in the partially decarburized zone is used to estimate the total depth of decarburization.

Maximum depth of decarburization – the largest measured value of the total depth of decarburization.

● Manualy

○ Automatic

DECARBURIZING

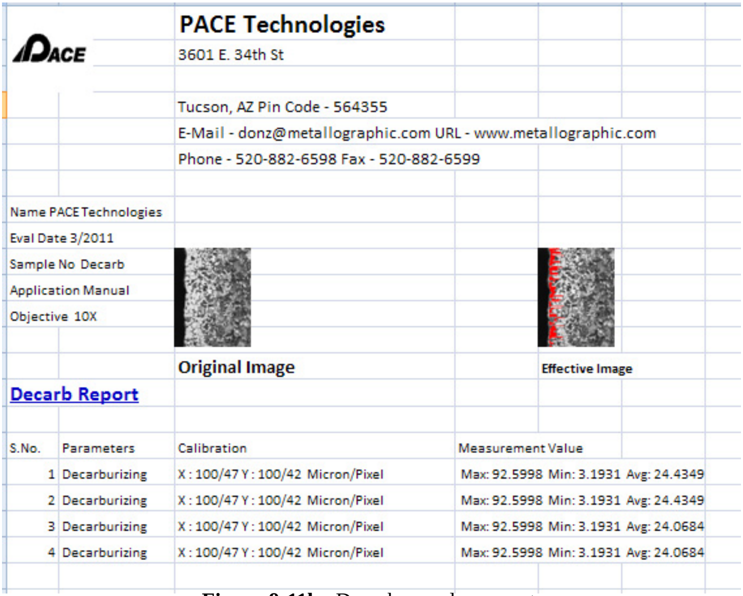
Calibration: Cal10X

Minor Width : 61.7021 Micron

Major Width : 61.7021 Micron

Average Width : 61.7021 Micron

Figure 9-11a. Decarburization analysis.



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Name PACE Technologies

Eval Date 3/2011

Sample No Decarb

Application Manual

Objective 10X

Decarb Report

S.No.	Parameters	Calibration	Measurement Value
1	Decarburizing	X : 100/47 Y : 100/42 Micron/Pixel	Max: 92.5998 Min: 3.1931 Avg: 24.4349
2	Decarburizing	X : 100/47 Y : 100/42 Micron/Pixel	Max: 92.5998 Min: 3.1931 Avg: 24.4349
3	Decarburizing	X : 100/47 Y : 100/42 Micron/Pixel	Max: 92.5998 Min: 3.1931 Avg: 24.0684
4	Decarburizing	X : 100/47 Y : 100/42 Micron/Pixel	Max: 92.5998 Min: 3.1931 Avg: 24.0684

Figure 9-11b. Decarb sample - report.

9.4.7 Coating thickness (ASTM B487)

Measurement of coating thickness is very important for characterizing the performance of many materials. Such coatings can have very important wear, heat resistance, and corrosion resistant properties. ASTM B487 (Standard Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of Cross Section) describes the recommended acceptance testing procedures for measuring coating thickness.

As with other metallographic analysis, proper specimen preparation is required to obtain a meaningful quantitative number. In general, the specimens need to be mounted, polished and etched so that the cross section is perpendicular to the coating as to avoid any geometrical errors in measuring the coating thickness. It is important that the surface be flat across the entire sample so that the boundaries are sharply defined. The cross section should also be prepared to eliminate deformation, smearing and other polishing artifacts.

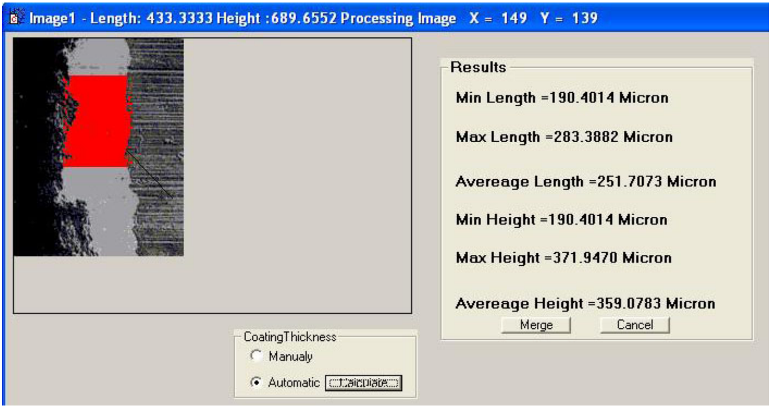

Image1 - Length: 433.3333 Height :689.6552 Processing Image X = 149 Y = 139

Results

Min Length =190.4014 Micron

Max Length =283.3882 Micron

Avereage Length =251.7073 Micron

Min Height =190.4014 Micron

Max Height =371.9470 Micron

Avereage Height =359.0783 Micron

CoatingThickness

○ Manualy

● Automatic

Figure 9-12a. Coating thickness - as detected.

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3/27/2011

Your Original Image

Name: PACE Technologies
Sample ID No.: Coating Thickness - auto
Application : Manual
Operator : DZ
Evaluation Date : 3/2011
Microscope Objective : 10X

Measurement Report

ASTM E1077

S.No.	Parameters	Calibration	Measurement Value
1	Coating	X : 100/47 Y : 100/42 Micron/Pixel	MinL:166.0 MaxL:258.6 AvgL:208.0 MinH:12.8 MaxH:309.7

Figure 9-12b. Coating thickness - report.

9.4.8 *Weld analysis*

Metallographic cross sectional analysis of welded components are listed in a number of SAE and AWS standards; however, no specific general standard is presently known. A number of common measurements include:

-Distance from the foot of the fillet to the center of the face (or throat)

-Distance from the root of the joint to the junction between the exposed surface of the weld and the base metal (leg)

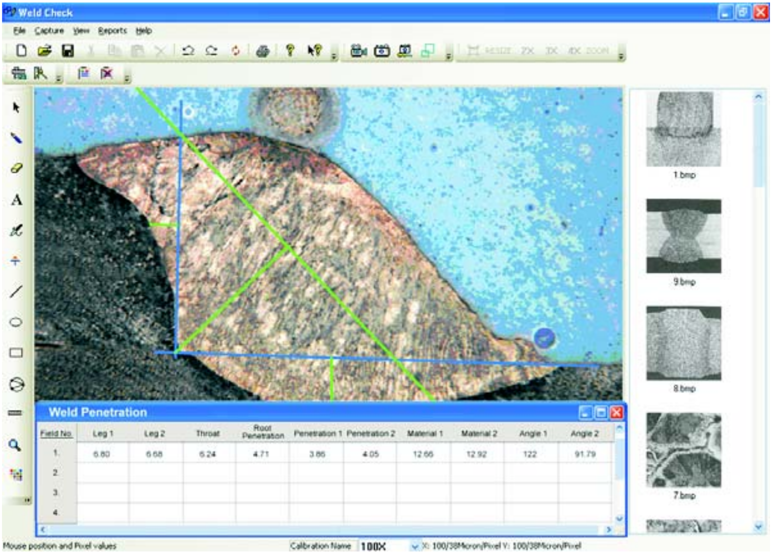
-Angles and the root penetration

-Depth of HAZ (heat affected zone)

-Area of HAZ

-Joint penetration

-Phase counting, etc.



Field No	Leg 1	Leg 2	Throat	Root Penetration	Penetration 1	Penetration 2	Material 1	Material 2	Angle 1	Angle 2
1.	6.80	6.68	6.24	4.71	3.86	4.05	12.66	12.92	122	91.79
2.										
3.										
4.										

Mouse position and Pixel values

Calibration Name 100X

X: 100/38Micron/Pixel Y: 100/38Micron/Pixel

Figure 9-13. Weld analysis - report.

CHAPTER 10
Hardness Testing

10.0 HARDNESS

Hardness Testing provides useful information, which can be correlated to tensile strength, wear resistance, ductility, and other physical characteristics of the material. Hardness testing is therefore useful for monitoring quality control and for aiding in the materials selection process. Table XXIII compares the various hardness testing applications.

Figure 10-1 Microhardness Tester (MHT) and Rockwell Tester.

Table XXIII. Common Hardness Testing Applications

Type	Penetrator	Typical range of loads, kg	General hardness range covered	Surface preparation before testing	Typical applications
Rockwell C	Diamond cone	150	Medium to very hard	Fine grinding	Production testing of finished parts
Rockwell B	1/16 inch steel ball	100	Soft to medium	Fine grinding	Production testing of finished parts
Superficial Rockwell	Steel ball or diamond cone	15, 30 and 45 kgf	>HRC 20 and <HRB 100	Fine polishing	Brittle and very thin materials
Brinell	10 mm steel ball	500-3000	Soft to hard	Coarse grinding	Production testing of unfinished parts
Vickers	Diamond pyramid	5-100	Very soft to very hard	Fine grinding	Laboratory investigations
Microhardness	Diamond pyramid	0.01-50	Very soft to very hard	Fine polishing	Test micro-constituents for alloys, ceramics

10.1 ROCKWELL HARDNESS

Rockwell hardness (HR) is an indentation hardness test that is determined with a spheroconical penetrator, or hard steel ball, that is forced into the specimen surface. The test is accomplished by bringing the specimen into contact with the penetrator and allowing the penetrator to be slowly forced into the specimen surface by a series of weights acting through a system of levers. After the load is released, a dial pointer or LED screen indicates the hardness number.

Typical Applications:
- Quality control for metal heat treatment
- Materials receiving inspection
- Evaluation of welds in steels and other metal alloys
- Failure analysis

10.2 BRINELL HARDNESS

To determine a Brinell hardness number (BHN), a 10 mm diameter steel ball is typically used as an indenter with a 3,000 kgf (29 kN) force. For softer materials, a smaller force is used; for harder materials, a tungsten carbide ball is used. The BHN can also be converted into the ultimate tensile strength (UTS), although the relationship is dependent on the material, and therefore is only an empirically based value.

10.3 VICKERS HARDNESS

The Vickers test is often easier to use than other hardness tests since the required calculations are independent of the size of the indenter, and the indenter can be used for all materials irrespective of hardness. The Vickers test can be used for all metals and has one of the widest scales among hardness tests. The unit of hardness given by the test is known as the Vickers Pyramid Number (HV) or Diamond Pyramid Hardness (DPH).

Table XXIV provides a relative scaling between the various hardness testing values, however these comparisons are not exact since the values will vary for different materials.

Table XXIV. Relative Hardness Scale Comparisons

Brinell (10 mm Ball, 3000 kg load)	Vickers (120 kg)	Rockwell C (150 kg)	Rockwell B (1/16" ball, 100 kg)
800	-	72	-
780	1220	71	-
725	1060	67	-
652	867	63	-
601	476	60	-
514	587	53	120
451	489	47	117
401	423	43	114
351	361	38	111
301	305	33	107
249	250	25	101
200	199	16	93
152	150	-	82
105	110	-	62
76	80	-	37

10.4 MICROHARDNESS

Microhardness testers are both mechanical and optical measuring tools. The indent is produced by applying a known load to the specimen and then measuring the size of the appropriate diagonals either optically or with image analysis software.

Microhardness is primarily determined with either a Knoop or Vickers indenter under test loads in the range of 1 to 2000 gram-force. Microhardness is used to measure the hardness of specific phases, small particles, and for brittle materials. Figure 10-2 shows the Knoop microhardness indents for a heat treated steel specimen.

Knoop hardness (HK) number is based on the size of the indent that a rhombic-based, pyramidal diamond indenter produces under a known applied load. The HK number is calculated by dividing the applied load (kilogram-force) by the projected area of the indentation (square millimeters).

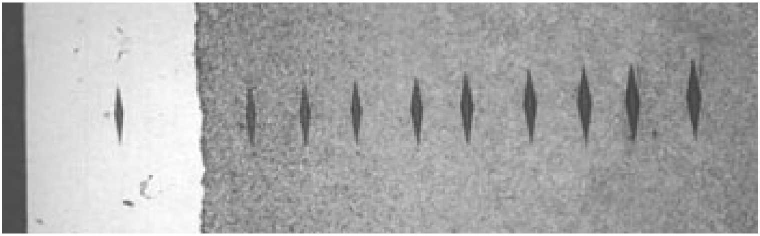


Figure 10-2 Knoop hardness indents in a hardened steel.

The Vickers hardness (HV) number is obtained by dividing the applied load in kilogram-force by the surface area of the indentation. The area of the indentation produced from the Vickers square-based pyramidal diamond is determined by the mean distance between the two diagonals of the indentation (Figure 10-3).

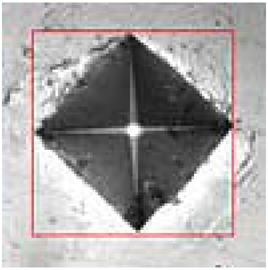


Figure 10-3 Vickers indent.

CHAPTER 11
Metallographic Specimen Preparation

11.0 PROCEDURES / ETCHANTS

Metallographic specimen preparation techniques can vary significantly depending on the hardness or ductility of the material. Figure 11-1 shows a relative guideline chart based on the hardness and ductility for most materials analyzed by metallographic techniques. The specimen procedures suggested in this manual are fundamentally derived from this chart.

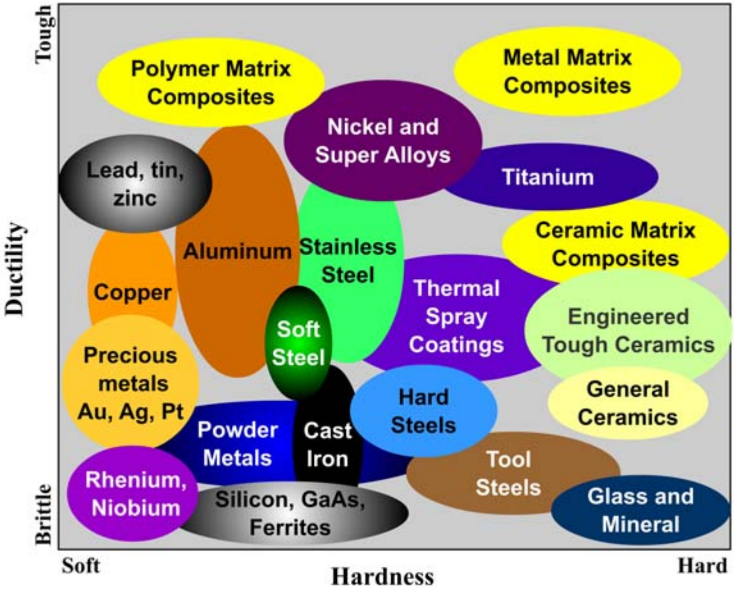


Figure 11-1 Relative hardness and ductility materials chart.

Figure 11-2 breaks this guidelines chart down into specific material classes, and Table XXV provides a basic overview of these classes and their metallographic specimen preparation procedures.

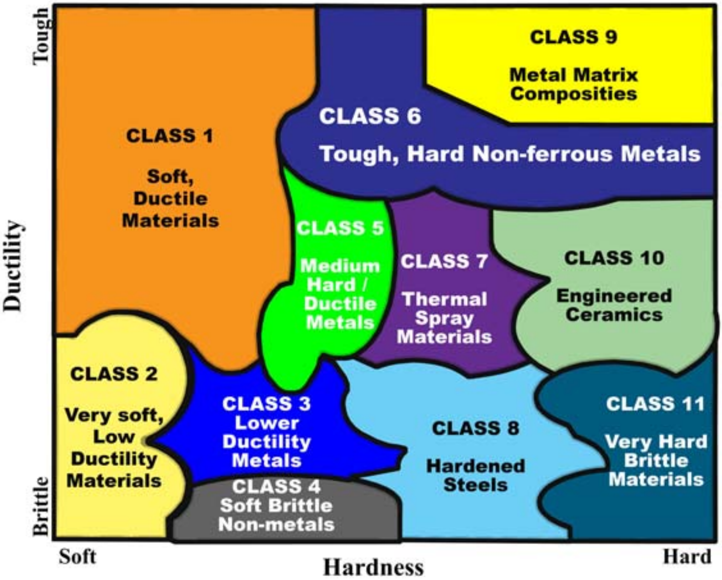


Figure 11-2 Guidelines chart for material classes.

Table XXV. Metallographic Preparation Breakdown by Material Classes

CLASS	MATERIALS	OVERVIEW
1	Soft, ductile materials (aluminum, copper, lead, tin, zinc, PMC's)	-Relatively easy to prepare most materials in this class with the use of Alumina abrasives and 1-2 polishing steps.
2	Very soft, low ductility materials (rhenium, niobium, gold, silver)	-Can be difficult to prepare because these materials are very soft and abrasives can become embedded.
3	Low ductility metals (powder metals and cast irons)	-Initial grinding with alumina abrasive is recommended, however rough polishing must minimize pull-out.

Table XXV. Metallographic Preparation Breakdown by Material Classes

CLASS	MATERIALS	OVERVIEW
4	Soft, brittle non-metals (silicon, GaAs, electronic devices, ferrites)	-Subsurface damage must be minimized in cutting, grinding and polishing of these materials. This requires the proper selection and application of the abrasive.
5	Medium hard, ductile metals (soft steels, stainless steels)	-Relatively easy to prepare most materials in this class with the use of SiC abrasives and 1-2 polishing steps.
6	Tough, hard non-ferrous metals (super alloys, titanium)	-Relatively easy to prepare most materials in this class with the use of SiC abrasives and 1-2 polishing steps.
7	Thermal spray materials	-Can pose significant preparation challenges depending upon the properties of the coating.
8	Hardened steels (high carbon and tool steels)	-Relatively easy to prepare most materials in this class with the use of SiC abrasives and 1-2 polishing steps.
9	Metal Matrix Composites	-Very dependent upon both the matrix and filler material properties. See specific preparation procedures.
10	Engineered Ceramics (zirconia, silicon nitride)	-Successful preparation is typically accomplished with colloidal silica by CMP polishing.
11	Very hard, brittle materials (glass, minerals)	-Minimize initial damage and CMP polish with colloidal silica abrasives.

11.1 CLASS 1 - DUCTILE MATERIALS

Examples of ductile materials, include aluminum, copper, brass, lead, tin, solder, zinc, polymers, polymer matrix composites (PMC's).

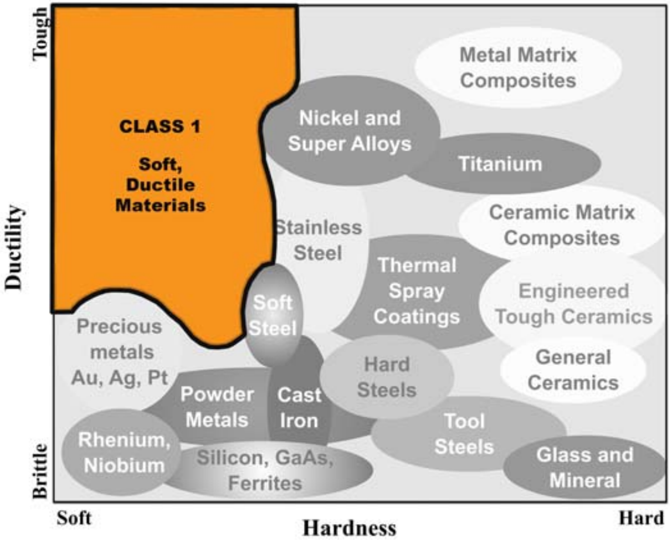


Figure 11-3 Class 1 - Ductile Materials.

Table XXVI. Preparation Guidelines for Class 1 Materials

Preparation Step	Basic Recommendation
Rough Grinding	P500 grit ALO paper or P1200 grit ALO paper
Rough Polishing	1 or 2 diamond polishing steps on a woven polishing pad
Final Polishing	Polycrystalline alumina on a woven polishing pad

11.1.1 Aluminum and Aluminum Alloys

Description:
Aluminum and its alloys are relatively soft materials which have a wide range of alloy applications.

Preparation Challenge:
The difficulty in preparing these materials arises from the fact that these materials have oxide inclusions in the microstructure. Proper specimen preparation retains these oxides and thus eliminates scratching and smearing from these particles. If the specimen is not prepared properly, the microstructure will not be accurately represented.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

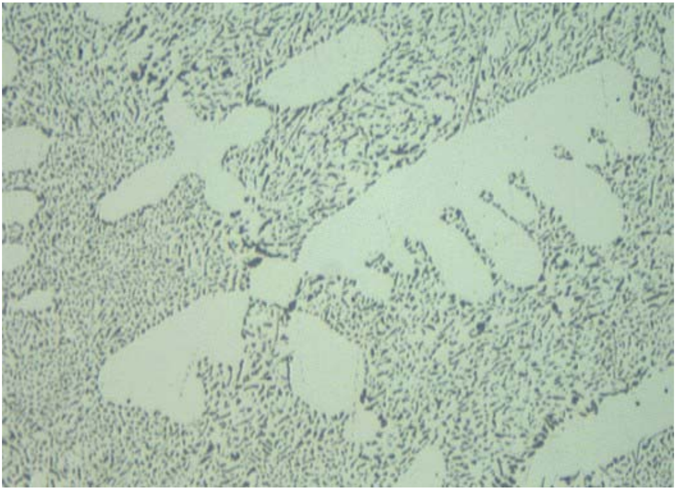


Figure 11-4 Cast aluminum-silicon, Kellers reagent, 200X.

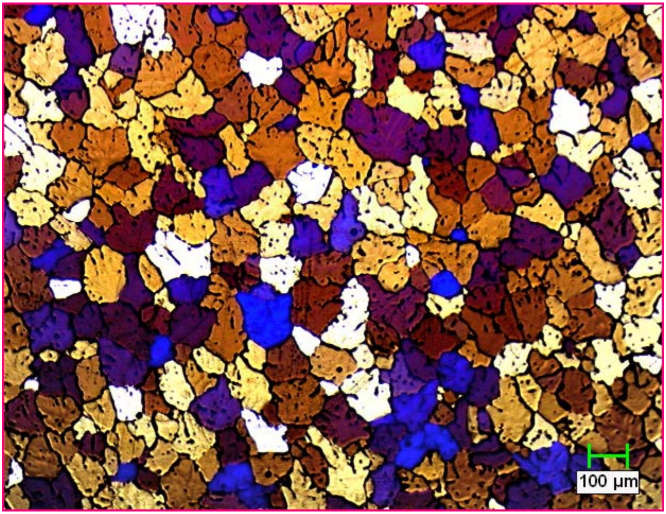


Figure 11-5 Anodized aluminum (photo courtesy of Clemex Technologies).

Figure 11-6 6061 aluminum, Kellers reagent, 200X.

Figure 11-7 7075 aluminum, Kellers reagent, 200X.

Table XXVII. Selected Microetchants for Aluminum

Name/Type	Application	Composition	Conditions
Keller's Reagent (Microetchant)(41)	Very popular general purpose reagent for Al and Al alloys; outlines all common constituents, reveals grain boundaries in certain alloys	2.5 ml HNO_3, 1.5 ml HCl, 1.0 ml HF, 95 ml DI water	Immerse sample for 10-20 seconds. Clean in warm water. Use only fresh.
Graff and Sargent (Microetchant)(42)	Wrought aluminum alloys	84 ml water, 15.5 ml HNO_3, 0.5 ml HF, 3 gm CrO_3	Immerse sample 20-60 seconds with mild agitation.
Al-Bronze (Microetchant)(43)	Al-bronzes, Cu, brasses, bronzes, Cu-Ni and Cu-Ag alloys, German silver; grain contrast of alpha brasses; Cu welds	100 ml DI water, 10 gm ammonium persulfate, 10 ml HCl	Etch for a few seconds to minutes. Possibly heated. Use only fresh.
Al-Cu (Microetchant)(45)	Grain contrast or grain-boundary lines	2 gm NaOH, 5 gm NaF, 93 ml DI water	Immerse for 2-3 minutes.

Table XXVIII. Selected Macroetchants for Aluminum

Name/Type	Application	Composition	Conditions
Tucker's Reagent (Macroetchant) (44)	Commercial-purity aluminum; Al-Mn alloys; Al-Si alloys; Al-Mg alloys; Al-Mg-Si and casting alloys	45 ml HCl (conc.), 15 ml HNO_3 (conc.), 15 ml HF (48%), 25 ml DI water	Mix fresh before using. Immerse or swab specimen for 10-15 seconds, rinse in warm water, dry and examine for desired effect; repeat until desired effect is obtained.
Poulton' reagent (Macroetchant) (45)	High-purity aluminum; commercial-purity aluminum; Al-Mn alloys; Al-Si alloys and casting alloys; Al-Mg alloys and casting alloys	12 ml HCl (conc.), 6 ml HNO_3 (conc.), 1 ml HF (48%), 1 ml DI water	Etch by brief immersion or by swabbing; rinse in cool water, and do not allow the etchant or the specimen to heat during etching
Caustic Etch (Macroetchant) (45)	Commercial-purity aluminum; all high-copper alloys and casting alloys; Al-Mn alloys; Al-Mg alloys; Al-Mg-Si and casting alloys; Al-Cu-Mg-Zn alloys and casting alloys	10 gm NaOH, 90 ml DI water	Immerse specimen 5-15 minutes in solution heated to 60-70°C, rinse in water, dip in 50% HNO_3 solution to desmut, rinse in water, dry.

11.1.2 Copper

Description: Copper and its alloys are relatively soft materials which have a wide range of alloy applications.

Preparation Challenge:
Copper and copper alloys are difficult to prepare because they have a tendency to work harden and have oxide inclusions. The result is that the oxide inclusions can become dislodged and scratch the surface. This scratching is more apparent following etching.

To minimize oxide pull-out, preparation damage can be minimized by using alumina grinding papers and polishing with polycrystalline diamond for the intermediate polishing step, followed by polishing with polycrystalline alumina.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

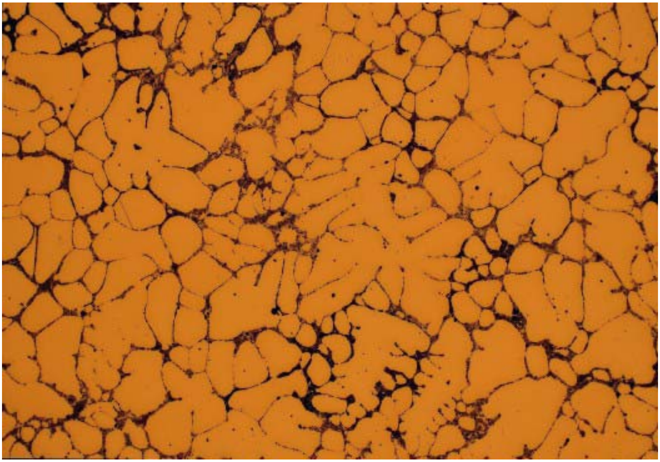


Figure 11-8 Tough Pitch Copper, B.F. 100X, Etchant NH_4OH / H_2O_2.

Figure 11-9 Diffusion of Stainless Steel (gray) into copper, 200X B.F., (as polished).

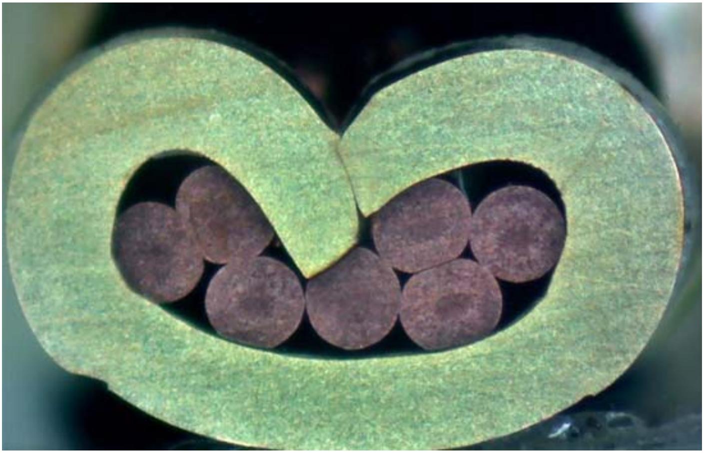


Figure 11-10a Inadequate crimping of copper wires.

Figure 11-10b Proper crimping of copper wires.

Table XXIX. Selected Microetchants for Copper & Copper Alloys

Name/Type	Application	Composition	Conditions
Copper (Macroetchant) (46)	For copper; reveals grain boundary and macroetch by polish attack	45 ml acetic acid, 45 ml HNO_3	Immerse at room temperature, rinse in warm water, dry
Copper (Macroetchant) (46)	Reveals grains; cracks and other defects; also reveals grain contrast	50 ml HNO_3, 50 ml DI water	Immerse at room temperature, rinse in warm water, dry
Copper and brass (Macroetchant) (46)	For coppers and all brasses; reveals grains; cracks and other defects	10 ml HNO_3, 90 ml DI water	Immerse at room temperature, rinse in warm water, dry
Copper and brass (Macroetchant) (46)	For all coppers and copper alloys; produces a brilliant, deep etch	50 ml HNO_3, 0.5 gm $AgNO_3$, 50 ml DI water	Immerse at room temperature, rinse in warm water, dry
Copper and brass (Macroetchant) (46)	For coppers and all brasses; reveals grains; cracks and other defects	30 ml HCl, 10 gm $FeCl_3$, 120 ml water or methanol	Immerse at room temperature, rinse in warm water, dry
Copper and brass (Macroetchant) (46)	For all brasses; produces a brilliant, deep etch	20 ml acetic acid, 10 ml 5% CrO_3, 5 ml 10% $FeCl_3$, 100 ml DI water	Immerse at room temperature, rinse in warm water, dry

Table XXIX (conti.) Selected Microetchants for Copper Alloys

Name/Type	Application	Composition	Conditions
Copper and Brass (Microetchant) (47)	Cu and all types of brasses; grains and cracks	90 ml DI water, 10-60 ml HNO_3	Etch for a few minutes
Copper, brass, bronze (Microetchant) (48)	Cu, brasses, bronzes, Al bronzes, Cu-Ni and Cu-Ag alloys, German silver; grain contrast of alpha brasses; Cu welds.	100 ml DI water, 10 gm ammonium persulfate, 10 ml HCl	Etch for a few seconds to minutes; possibly heated; use only fresh
Copper, solder, brass, bronze (Microetchant) (49)	Most types of Cu and Cu alloys; Cu-Ag solder layers; Mn, P, Be, Al-Si bronzes.	25 ml DI water, 25 ml ammonia water, 5-25 ml H_2O_2 (30%)	Use only fresh; possible addition of 1-5 ml solution of potassium hydroxide (20%)
Klemm's reagent - (Microetchant) (49)	Color and grain contrast etchant for Cu. For alpha and beta brass.	50 ml saturated aqueous solution of sodium thiosulfate, 1 gm potassium metabisulfite	Etch for 3 minutes
Berahs's tint etch (Microetchant) (50)	For copper, brass and bronze alloys	200 gm CrO3, 20 gm anhydrous sodium sulfate, 17 ml HCl , 1000 ml DI water	Dip sample 2-20 seconds; can dilute to slow down rate of attack

11.1.3 Brass

Description:
Brass is a copper-zinc alloy, whereas, bronze is a copper-tin alloy. Brass is stronger than copper and has a higher malleability then either copper and zinc. Brass is also a good conductor of heat, has excellent acoustic properties and is generally resistant to corrosion in salt water. Brass is commonly rolled and extruded; however, these processes also work harden the metal which can be quantified by metallographic analysis.

Preparation Challenge:
Brasses and bronzes are relatively easy to prepare.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

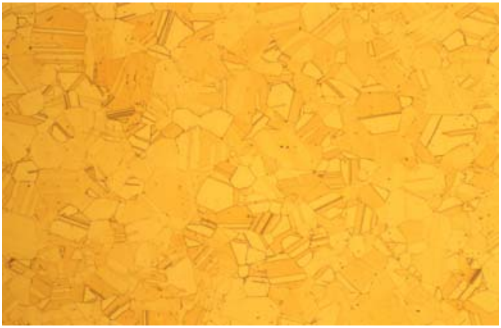


Figure 11-11a 70-30 Brass, 200X (B.F.), Etchant H_2O_2 / NH_4OH

Figure 11-11b 70-30 Brass cold rolled, 200X (B.F.), Etchant H_2O_2 / NH_4OH

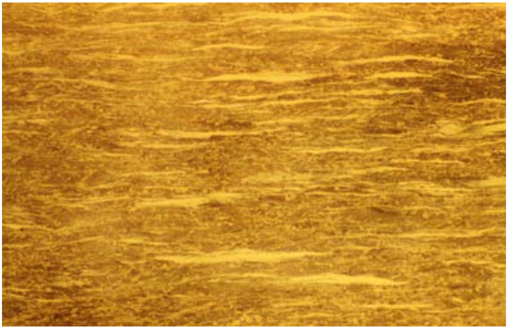


Figure 11-11c 70-30 Brass deep rolled, 200X (B.F.), Etchant H_2O_2 / NH_4OH

11.1.4 Bronze

Description:
Bronzes are essentially copper-tin alloys which can contain additional alloying elements. Bronzes are also harder and tougher than wrought iron.

Preparation Challenge:
The specimen preparation procedures for brasses and bronzes are relatively straightforward.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P500 grit ALO paper P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane 1 minute
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

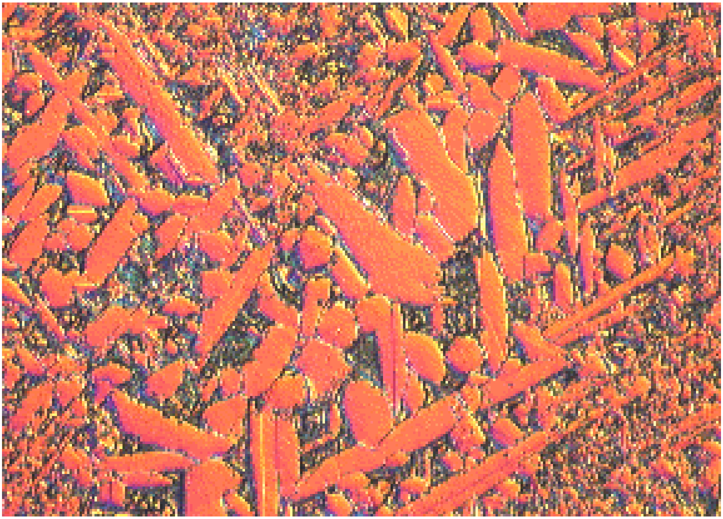


Figure 11-12 Manganese-Aluminum Bronze, 400X (DIC), Etchant: ammonium persulfate, HCl, DI water.

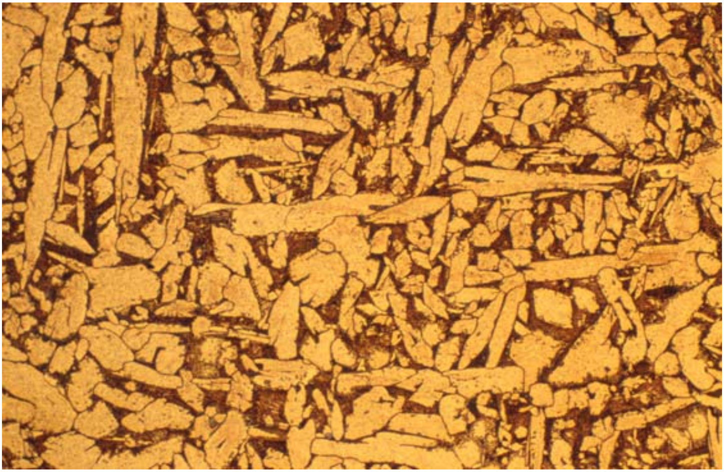


Figure 11-13 Nickel-Iron Bronze, 200X B.F., Etchant NH_4OH, H_2O_2.

11.1.5 Tin and Tin Alloys

Description:
Tin and tin alloys are relatively soft materials. Pure tin has a recrystallization temperature below room temperature, and thus can easily smear.

Preparation Challenge:
The difficulty in preparing these metals for microstructural analysis is because they do not work harden and thus their microstructure can be altered simply by incorrect specimen preparation. For example, pure tin has a relatively large grain size; however, if not prepared properly, the microstructure of the pure tin will actually appear to be quite small due to recrystallization when, in fact, the grain structure is actually quite large.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

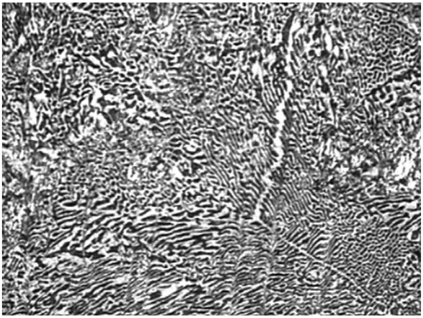


Figure 11-14a Tin-Bismuth eutectic alloy, 200X, Etchant 2% Nital.

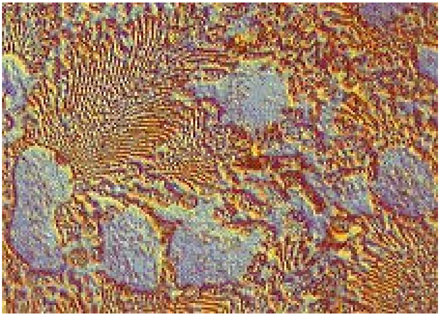


Figure 11-14b Tin-antimony-copper (hard tin) alloy, 100X, Etchant 2% Nital.

Figure 11-14c Pure tin, 100X, as polished.

Table XXX. Selected Microetchants for Tin & Tin Alloys

Name/Type	Application	Composition	Conditions
Klemm's II reagent (Microetchant) (51)	For etching Sn. Colors grains	50 ml saturated aqueous sodium thiosulfate, 5 gm potassium metabisulfite	Immerse sample 60-90 seconds
Nital (Microetchant) (52)	Recommended for etching tin-antimony alloys; darkens tin-rich matrix, leaving intermetallics unattacked; often used for etching specimens of babbitted bearings	2 ml HNO_3 98 ml ethanol (95%) or methanol (95%)	Immerse sample 60-90 seconds
Picral (Microetchant) (52)	For etching tin-coated steel and tin-coated cast iron	4 gm picric acid 100 ml methanol (95%) or ethanol (95%)	Immerse sample 60-90 seconds
Tin and lead solder joints (Microetchant) (52)	General etchant for lead-tin alloys and for soldered joints	3 parts acetic acid 4 parts HNO_3 16 parts water	Etch for a few seconds to minutes.
Tin (Macroetchant) (53)	Macroetchant for all types of Sn and Sn-base alloys; grain distribution	Saturated aqueous solution of ammonium polysulfide	Etch for 20-30 minutes; wipe off with cotton after etching

Table XXX. (conti.) Selected Microetchants for Tin & Tin Alloys

Name/Type	Application	Composition	Conditions
Tin General (Macroetchant) (53)	Macroetchant for Sn-rich bearing metals and white metals	100 ml DI water 2 ml HCl 10 gm $FeCl_3$	Etch for 30 seconds to 5 minutes
Tin General (Microetchant) (51)	Grain contrast etchant for tin and pewter	2 ml HCl 5 ml HNO_3 93 ml DI water	Etch for 30 seconds to 5 minutes
Tin General (Microetchant) (51)	General use for tin and tin alloys	5 ml HCl 2 gm $FeCl_3$ 30 ml DI water 60 ml methanol (95%) or ethanol (95%)	Etch for 30 seconds to 5 minutes
Tin General (Microetchant) (51)	Darkens primary and eutectic lead in lead-rich tin-lead alloys	5% $AgNO_3$ in water	Etch for 30 seconds to 5 minutes
Tin General (Microetchant) (51)	Darkens the lead in the eutectic of tin-rich tin-lead alloys	10 ml HNO_3 10 ml acetic acid 80 ml glycerol	Etch for 30 seconds to 5 minutes
Tin-coated copper (Microetchant) (51)	For etching tin-coated copper and copper alloys (polish attack)	NH_4OH diluted with few drops of 30% H_2O_2	Etch for 30 seconds to 5 minutes

11.1.6 Lead and Lead Alloys

Description:
Lead and lead alloys are very soft materials. Pure lead has a recrystallization temperature below room temperature, and thus can easily smear.

Preparation Challenge:
The difficulty in preparing these metals for microstructural analysis is because they do not work harden and thus their microstructure can be altered simply by incorrect specimen preparation.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

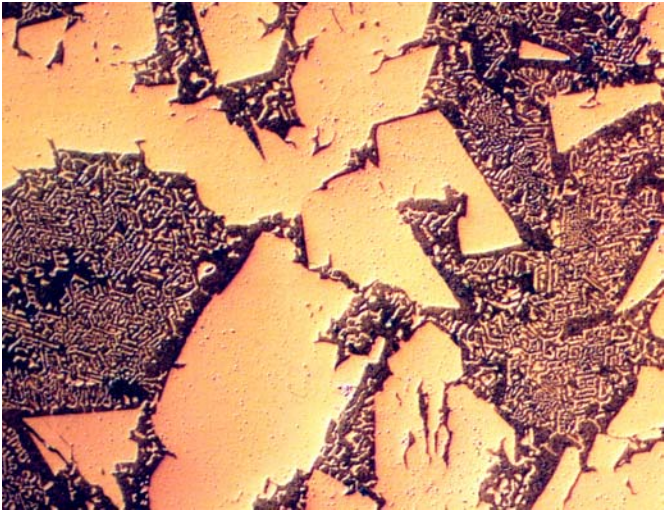


Figure 11-15a Type metal (54% Pb, 28% Sb, 18% Sn), 400X (DIC), Etchant 90 ml ethanol, 20 ml HCl.

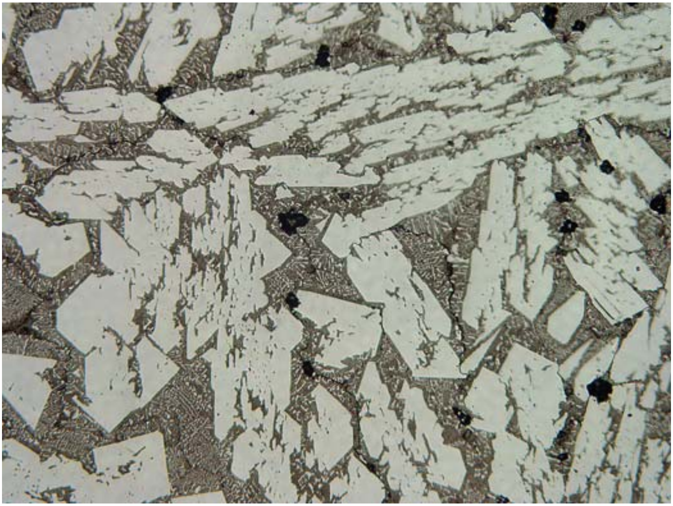


Figure 11-15b Type metal (54% Pb, 28% Sb, 18% Sn), 200X (BF), Etchant 90 ml ethanol, 20 ml HCl.

Table XXXI. Selected Etchants for Lead & Lead Alloys

Name/Type	Application	Composition	Conditions
Lead (Macroetchant) (54)	Macroetchant for grain contrast, welded seams and layer for Pb	80 ml DI water, 20 ml HNO_3	Swab for 10 minutes
Lead (Macroetchant) (55)	Macroetching of lead	1 part acetic acid (glacial), 1 part HNO_3, 4 parts glycerol	Use freshly prepared solution at 80°C, discard after use; etch for several minutes, rinse in water
Lead (Microetchant) (55)	For etching lead and lead-tin alloys containing up to 3% tin	3 parts acetic acid (glacial), 4 parts HNO_3, 16 parts DI water	Use freshly prepared solution at 40-42°C; immerse specimen for 4-30 minutes until disturbed layer is removed; clean with cotton in running water
Lead (Microetchant) (56)	For etching Pb alloys and solders	90 ml water or ethanol, 20-30 ml HCl, 0-10 gm $FeCl_3$	Immerse sample 1-10 minutes
Beckinsdale and Waterhouse (Microetchant) (56)	For etching Pb and alloys; grain contrast etch	90 ml DI water, 10 ml HNO_3	Can add CrO_3

11.1.7 Zinc and Zinc Alloys

Description:
Zinc and zinc alloys are relatively soft materials. Pure zinc has a recrystallization temperature below room temperature, and thus can easily smear. Zinc alloys are commonly used because they can be diecast and easily machined.

Preparation Challenge:
The difficulty in preparing these metals for microstructural analysis is because they do not work harden and thus their microstructure can be altered simply by incorrect specimen preparation. For example, pure zinc has a relatively large grain size; however, if not prepared properly, the microstructure of the pure zinc will actually appear to be quite small due to recrystallization.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

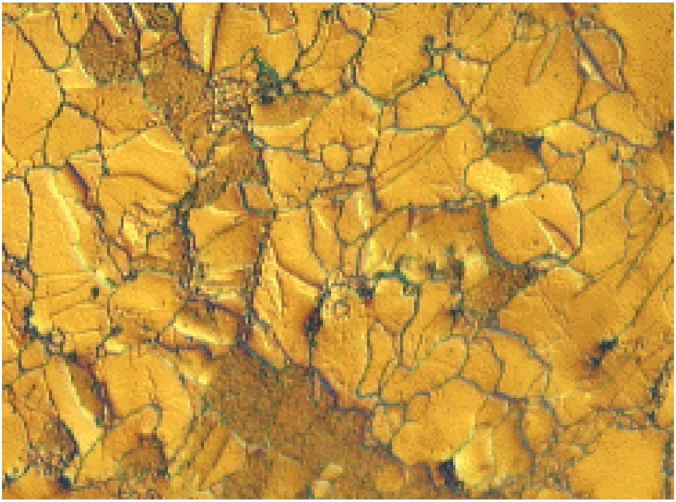


Figure 11-16 Pure zinc, 100X (DIC), Etchant: 10 gms NaOH in 100 ml DI water.

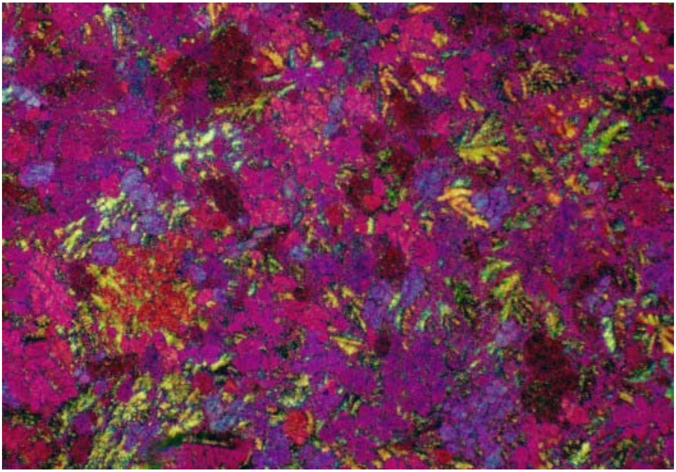


Figure 11-17 Zinc-Aluminum alloy, 100 X Polarized Light, Etchant 40 gms CrO_3, 3 gms Na_2SO_4, 200 ml DI water.

Table XXXII. Selected Microetchants for Zinc

Name/Type	Application	Composition	Conditions
Palmerton's reagent (Microetchant) (57)	Very popular for pure Zn and alloys	40 gm CrO_3, 3 gm Na_2SO_4, 200 ml DI water	Immerse sample up to 3 minutes. Rinse in 20% aq. CrO_3
Klemm's I reagent (Microetchant) (57)	For Zn and zinc alloys	50 ml sat. aq. sodium thiosulfate, 1 gm potassium metabisulfite	Immerse sample 30 seconds
Zinc Die cast (Microetchant) (58)	For Zn Die-cast	5 ml HNO_3, 100 ml DI water	Immerse sample 5-30 seconds
Gelles (Microetchant)(57)	For Zn and zinc alloys	98 ml lactic acid, 2 ml HNO_3	Immerse sample 15 minutes
Gennone and Kersey (Microetchant) (59)	For Zn alloys; reveals grain boundaries	84 ml water, 15 ml H_2SO_4, 1 ml HF	Immerse sample 10 seconds at 100°F (38°C)
Zinc (Microetchant) (59)	For Zn and zinc alloys	5 gm $FeCl_3$, 10 ml HCl, 240 ml alcohol	Immerse sample 5-30 seconds

Table XXXIII. Selected Macroetchants for Zinc

Name/Type	Application	Composition	Conditions
Palmerton's Reagent (Macroetchant) (59)	Macroetchant for Zn alloys containing Cu.	100 ml DI water, 20 gm CrO_3, 3.5 gm Na_2SO_4-$10H_2O$	Etch for seconds to minutes.
Zinc (Macroetchant) (60)	Macroetchant for pure Zn, Zn alloys without Cu, and cast materials	Concentrated HCl	Etch for approximately 15 seconds; rinse off film under running water
Zinc (Macroetchant) (60)	Macroetchant for pure Zn, Zn alloys without Cu, and cast materials	Concentrated HNO_3	Etch for approximately 15 seconds; rinse off film under running water
Zinc (Macroetchant) (61)	Macroetchant for pure Zn, Zn alloys without Cu, and cast materials	50 ml DI water, 50 ml HCl	Etch for approximately 15 seconds; rinse off film under running water

11.1.8 Carbon-Carbon PMC Composites

Description:
Polymer matrix composites (PMCs) are extremely useful materials because they combine lightweight polymers (plastics) with materials such as graphite and boron carbide. PMCs are used in applications ranging from sports equipment to stealth fighter airplanes.

Preparation Challenge:
Proper preparation of carbon composites is required because the properties of the matrix material and the carbon fibers vary significantly in both hardness and fracture toughness. As a general rule, the most difficult components to prepare in composites are the most brittle components (e.g. carbon fibers). To properly prepare carbon-carbon composites, initial sectioning must minimize damage. Proceeding preparation steps must not result in additional damage.

SECTIONING
Diamond wafering blade - medium grit/low concentration

MOUNTING
Castable mounting with Epoxy or Acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
P500 grit ALO paper P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane 1 minute
6 micron DIAMAT diamond on TEXPAN polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	3 minutes
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on MICROPAD / MICROPAD 2 polishing pad		5-10 lbs	100/100 rpm	1 minute

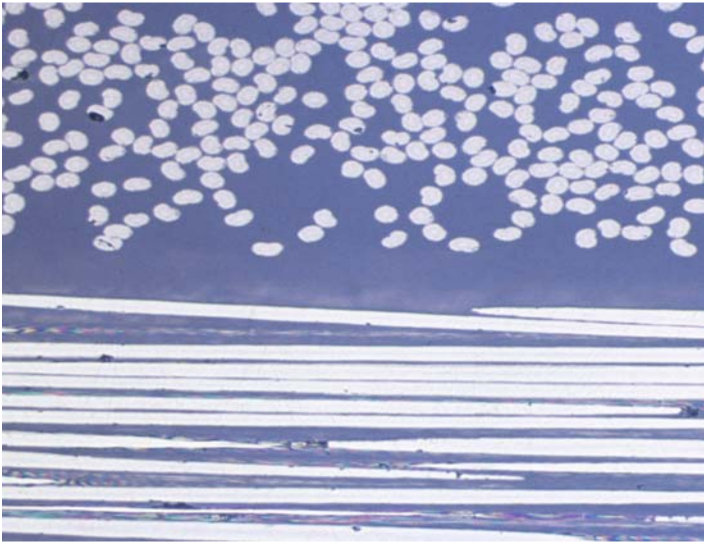


Figure 11-18a Carbon fibers in polymer matrix, 400X, as polished.

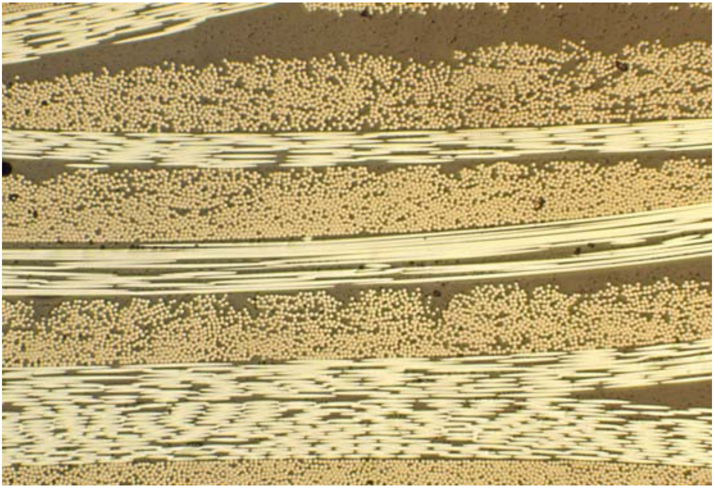


Figure 11-18b Carbon fibers in polymer matrix, 100X, as polished.

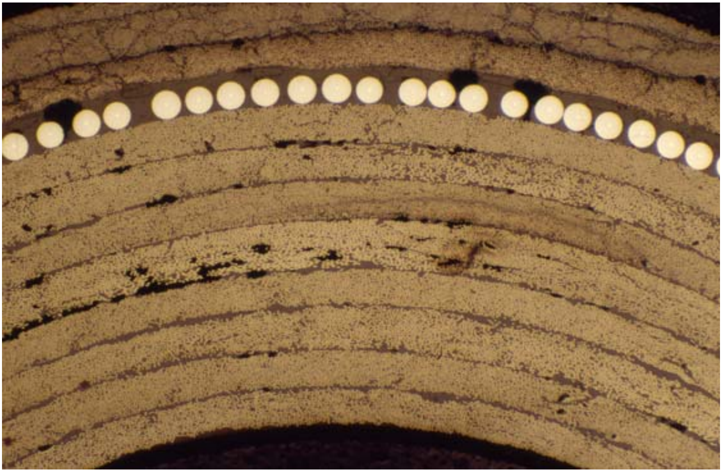


Figure 11-19a Boron-carbon golf shaft, 100X, as polished.

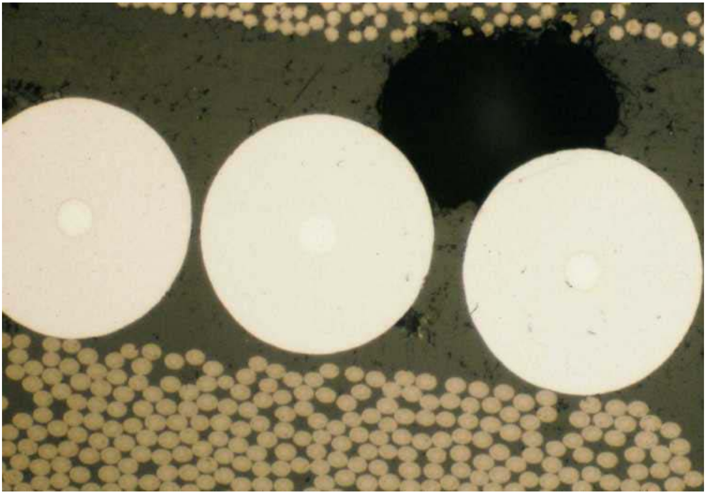


Figure 11-19b Boron-carbon golf shaft, 1000X, as polished.

11.2 CLASS 2 - VERY SOFT, LOW DUCTILITY MATERIALS

This class of materials includes: refractory metals (tungsten, rhenium, niobium, molybdenum), rare earths and precious metals (gold, silver, platinum).

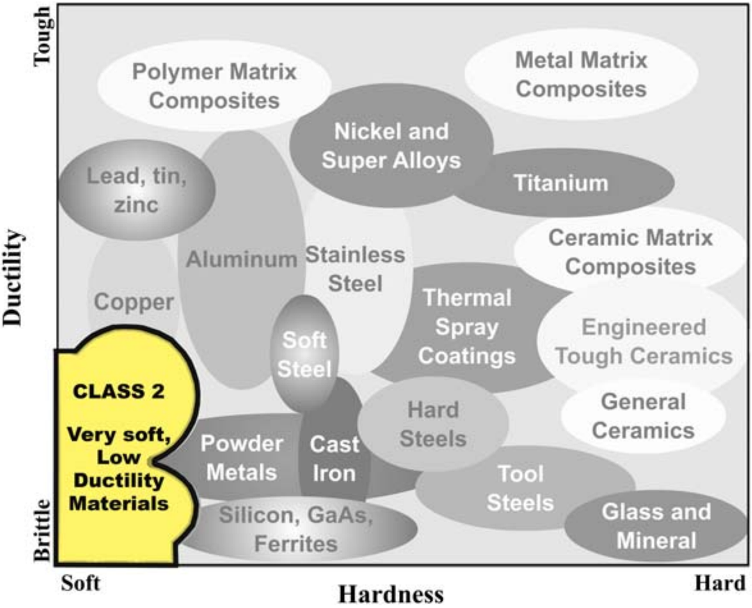


Figure 11-20 Class 2 - Very soft, low ductility materials.

Table XXXIV. Preparation Guidelines for Class 2 Materials

Preparation Step	Basic Recommendation
Rough Grinding	P1200 grit ALO grinding papers
Rough Polishing	1 micron diamond on a woven polishing pad
Final Polishing	Etch polish with polycrystalline alumina

11.2.1 Refractory Materials (Rhenium, Niobium, Tungsten)

Description:
Refractory metals such as rhenium, niobium and tungsten have very high melting temperatures; however, they are typically very soft.

Preparation Challenge:
Since refractory metals are very soft, any loose or fractured abrasive particles can easily get embedded. This makes specimen preparation very difficult because it gums up diamond grinding disks or the specimen can become embedded with fractured abrasive particles. The key to preparation of these materials is to use tougher alumina abrasives and to chemically etch the specimen between each grinding step. The purpose of etching is to remove the embedded particles so as not to carry them over to the next grinding step as contamination.

SECTIONING
MAXCUT abrasive blade (MAX-A or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

Etch Polish -10% Diluted etchant below:

Niobium - 30 ml HCl, 15 ml HNO_3, 30 ml HF (40%)

Rhenium - 30 ml lactic acid, 30 ml HNO_3, 1 ml HF

Tungsten - 15 ml HNO_3, 3 ml HF, 80 ml water

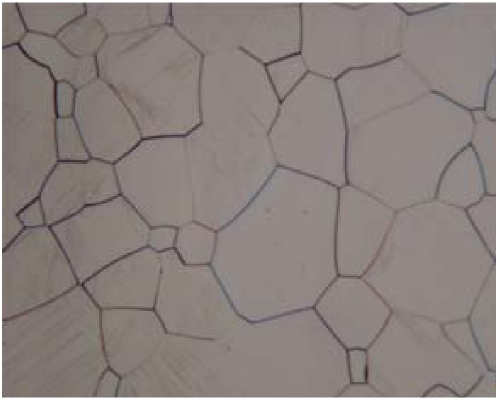


Figure 11-21 Niobium alloy (B.F.), Etchant 30 ml HCl, 15 ml HNO_3, 30 ml HF (40%).

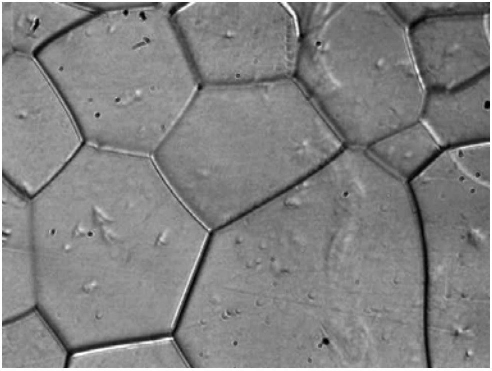


Figure 11-22 Rhenium alloy, 1000X (B.F.), Etchant 30 ml lactic acid, 30 ml HNO_3, 1 ml HF.

Table XXXV. Selected Niobium Etchants

Name/Type	Application	Composition	Conditions
Niobium (Macroetchant) (62)	For Mo, W, V, Nb, Ta and their alloys	30 ml HCl, 15 ml HNO_3, 30 ml HF (40%)	Etch for seconds to minutes
Niobium (Microetchant) (63)	Etchant for niobium and niobium alloys	10 ml HNO_3, 10 ml HF, 30 ml lactic acid	Swab for 5 seconds; repeat if necessary
Niobium (Microetchant) (63)	Etchant for niobium and niobium alloys	10 ml HNO_3, 10 ml HF, 30 ml lactic acid	Swab for 5 seconds; repeat if necessary
Niobium (Microetchant) (63)	Etchant for niobium and niobium alloys	20 ml HF, 15 ml H_2SO_4, 5 ml HNO_3, 50 ml DI water	Immerse up to 5 minutes
Niobium (Microetchant) (63)	Etchant for niobium and niobium alloys	50 ml HNO_3, 30 gm NH_4HF_2, 20 ml DI water	Swab 3-10 seconds; use in fume hood
Niobium (Microetchant) (64)	Etchant for niobium and niobium alloys	70 ml DI water, 20 ml H_2O_2 (30%), 10 ml NH_4OH	Seconds to minutes

Table XXXV (conti.). Selected Rhenium Etchants

Name/Type	Application	Composition	Conditions
Buchheit (Microetchant) (65)	For etching Re	30 ml lactic acid, 30 ml HNO_3, 1 ml HF	Swab sample
Murakami's reagent (Microetchant) (65)	For etching Re	10 gm KOH, 10 gm $K_3Fe(CN)_6$, 100 ml DI water	Swab or immerse sample
Murakami's reagent (modified) (Microetchant) (65)	For etching Re	15 gm $K_3Fe(CN)_6$, 2 gm KOH or NaOH, 100 ml DI water	Swab or immerse sample
Rhenium (Microetchant) (66)	For etching Re silicides	50 ml water, 50 ml HNO_3, 50 ml HF	Swab or immerse sample
Rhenium (Microetchant) (66)	CAUTION: Toxic. Ta, Nb, and their alloys; Cr and Cr silicide; Re silicide; W-Th alloys	50 ml DI water, 50 ml HNO_3, 50 ml HF	Few seconds to minutes

11.2.2 Rare Earth - Neodymium

Description:
Neodymium is a rare earth metal which has some very interesting properties. Applications for neodymium include: magnets (strongest permanent magnets known), colorant for welder's glasses, cyrocoolers (high specific heat capacity), light filters, fertilizers and for lasers.

Preparation Challenge:
Neodymium is both a soft and ductile material. It is most often alloyed with other elements, which gives it a wider range of properties. Preparation concerns include avoiding embedded abrasives by using alumina grinding papers.

SECTIONING
MAXCUT abrasive blade (MAX-A or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

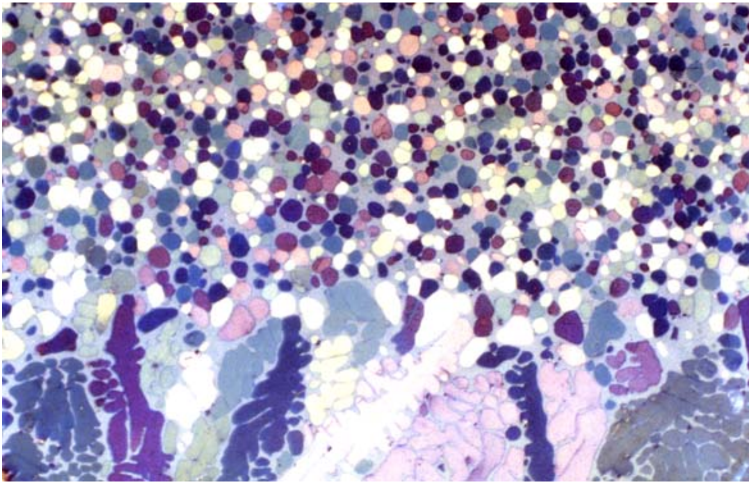


Figure 11-23 Neodymium-Aluminum-Titanium casting, (polarized light).

Table XXXVI. Selected Rare Earth Etchants

Name/Type	Application	Composition	Conditions
Hergert and Altenhoff (Microetchant) (65)	For etching Re-Co rare earth alloys	10 gm ammonium persulfate, 100 ml DI water	Use boiling, short immersion time
Myklebust and Daane (Microetchant) (65)	For etching Y-Mn rare earths	99.25 ml ethanol, 0.75 ml HNO_3	Rinse with acetone or alcohol, dry. Immerse sample 2-3 minutes
Lundin and Klodt (Microetchant) (65)	Rare earth etch for Gd, Er, Ho, and Dy	10 ml H_3PO_4, 10 ml lactic acid, 30 ml HNO_3, 20 ml acetic acid	Etches with slight chemical polishing action
Geiselman (Microetchant) (65)	Rare earth etch for scandium grain boundary	100 ml water, 5 gm CrO_3	Use polarized light
Oak Ridge (Microetchant) (65)	Rare earth etch for Y	30 ml acetic acid, 30 ml H_3PO_4, 30 ml HNO_3	Immerse sample
Rosen and Sprang (Microetchant) (65)	Rare earth etch for Y-Al-C alloys; carbide phase reacts with water	30 ml lactic acid, 10 ml HNO_3, 2 ml HF	Swab sample

11.2.3 Tungsten

Description:
Tungsten in its raw form is very brittle; however, pure tungsten can be cut with a hacksaw. Tungsten is a remarkable material because it has the highest melting temperature of all non-alloy metals and is second only to carbon's melting temperature for all elements. It is also a very dense metal.

Preparation Challenge:
Tungsten is another metal that can present some very unique preparation challenges due to its physical properties. Factors to consider during preparation are embedding of fracture abrasives and the ease at which the metal can smear. Specimen preparation should include alumina abrasive grinding papers and CMP polishing with hydrogen peroxide.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit AlO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

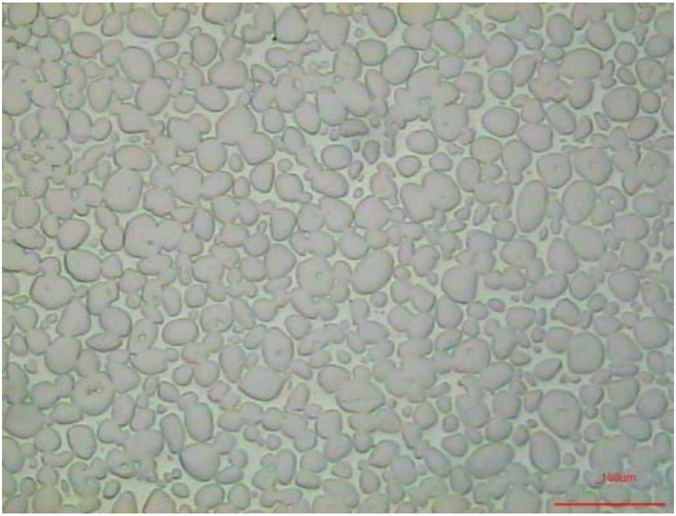


Figure 11-24 Tungsten refractory metal, 200X (DIC), etched with Murakami's reagent.

Table XXXVII. Selected Tungsten and Molybdenum Etchants

Name/Type	Application	Composition	Conditions
Tungsten, Molybdenum (Macroetchant) (66)	Macroetchant Mo, W, and V	75 ml distilled water, 35 ml HNO_3, 15 ml HF (40%)	Etch for 10-20 minutes
Murakami's reagent (modified A) (Microetchant) (63)	For W, Mo and their alloys	30 gm $K_3Fe(CN)_6$, 10 gm KOH or NaOH, 200 ml DI water	Swab or immerse sample
Murakami's reagent (modified) (Microetchant) (63)	For W, Mo and their alloys	15 gm $K_3Fe(CN)_6$, 2 gm KOH or NaOH, 100 ml DI water	Swab or immerse sample
Murakami's reagent (Microetchant) (63)	For W, Mo and their alloys	10 gm $K_3Fe(CN)_6$, 10 gm KOH or NaOH, 100 ml DI water	Swab 5 to 60 seconds; immersion will produce a stain etch; follow with water rinse, alcohol rinse, dry
Tungsten, Molybdenum (Microetchant) (64)	Mo and Mo-Ni alloys; W and W alloys	50 ml DI water, 50 ml H_2O_2 (30%), 50 ml ammonia water	Few seconds to minutes
Tungsten-cobalt (Microetchant) (64)	W-Co	100 ml DI water, 2 gm picric acid, 25 gm NaOH	Etch for 15 seconds; boiling

11.2.4 Precious Metals (Gold, Silver, Platinum)

Description:
Precious metals such as gold, silver and platinum are very soft.

Preparation Challenge:
Precious metals are relatively soft metals and any loose or fracture abrasive particles can easily embed. This makes specimen preparation very difficult because it gums up diamond grinding disks or the specimen becomes embedded with fractured abrasives. The key to preparation of these materials is to use the tougher alumina abrasives to minimize embedded abrasives.

SECTIONING
MAXCUT abrasive blade (MAX-A or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
P1200 grit ALO paper	Water	5-10 lbs	100/100 rpm	Until plane
800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

Table XXXVIII. Selected Silver Etchants

Name/Type	Application	Composition	Conditions
Silver (Microetchant) (67)	For pure Ag, Ag-Ni alloys, Ag-Pd alloys	50 ml ammonia water, 50 ml H_2O_2 (3 %)	Etch up to 11 minutes; use fresh!
Silver (Microetchant) (68)	For macroetching pure Ag and low-alloy Ag; reveals grain contrast	95 ml methanol (95 %), 10 ml H_2O_2	Several minutes
Silver (Microetchant) (69)	For pure Ag and Ag alloys, Ag solders	10 ml H_2SO_4, 100 ml saturated aqueous solution of potassium dichromate, 2 ml saturated aqueous solution of NaCl	Etch several seconds to minutes, 1:9 dilution with distilled water; possibly without sulfuric acid
Silver (Microetchant) (67)	Pure Ag and Ag composites with other metals	100 ml NH_4OH, 5-10 gm potassium cyanide	Etch for several seconds
Silver solder (Microetchant) (67)	For Ag solders	100 ml DI water, 2 gm iron (III) chloride	Etch for 5-30 seconds

Table XXXIX. Selected Gold, Palladium, Platinum Etchants

Name/Type	Application	Composition	Conditions
Gold, Palladium, Platinum (Microetchant) (70)	For Au alloys with high content of precious metals; white gold; Pd and Pt	100 ml DI water, 10 gm ammonium persulfate	Etch for 30 seconds to 2 minutes
Gold, Palladium, Platinum (Microetchant) (70)	For pure Pt and Pd, Au alloys	30 ml DI water, 25 ml HCl, 5 ml HNO_3	Etch for 1-5 minutes. Use hot! Remove precipitate of gold chloride with NH_4OH
Gold alloys (Microetchant) (71)	For Au-Cu-Ag alloys	100 ml water, 100 ml H_2O_2 (3%), 32 gm $FeCl_3$	Immerse sample for up to a few minutes
Gold alloys (Microetchant) (71)	For gold and high noble metal alloys	60 ml HCl, 40 ml HNO_3	Use in fume hood
Gold, Palladium, Platinum (Microetchant) (70)	Pure Au and Au-rich alloys, Pd and Pd alloys	100 ml HCl, 1-5 gm chromium (VI) oxide	Etch several seconds to minutes

11.3 CLASS 3 - LOWER DUCTILITY METALS

Examples of lower ductility metals include powder metals and cast iron.

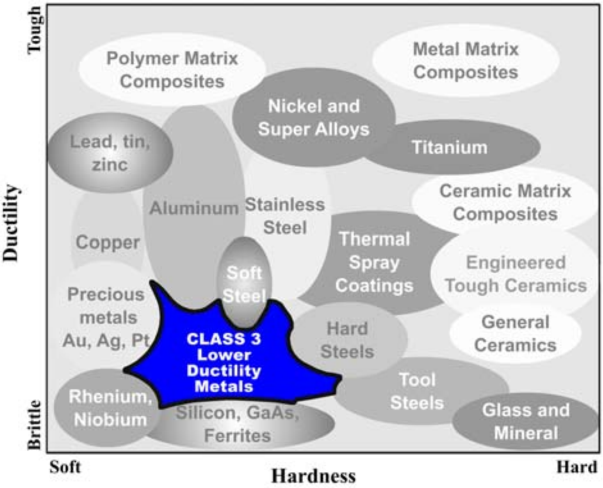


Figure 11-25 Class 3 - Low ductility metals.

Table XL. Preparation Guidelines for Class 3 - Powder Metals

Preparation Step	Basic Recommendation
Rough Grinding	Diamond on a metal mesh polishing cloth
Rough Polishing	Diamond on woven polishing pads
Final Polishing	Polycrystalline alumina on a napped polishing pad

Table XLI. Preparation Guidelines for Class 3 - with Brittle Components

Preparation Step	Basic Recommendation
Rough Grinding	240 (P220), 360 (P500), 600 (P1200), 800 (P2400), 1200 (P4000) grit SiC paper
Rough Polishing	1 or 2 diamond polishing steps with a woven polishing pad
Final Polishing	Polycrystalline alumina on a woven polishing pad

11.3.1 Sintered Iron - Powder Metallurgy

Description:
Powder metallurgy is a very useful manufacturing process for parts designed with hard-to-machine geometries. The process includes pressing a metal powder into the rough shape and then sintering it just below the melting temperature of the alloy.

Preparation Challenge:
The metallographic specimen preparation of powder metallurgy samples is affected by the specimen’s composition, packing and sintering conditions. Microstructural features of interest include: porosity, grain size, inclusions and orientation of voids. Proper metallurgical preparation takes into account that the microstructure may be porous and perhaps somewhat brittle due to inadequate sintering conditions.

Vacuum impregnation is often used to support voids and porosity before initial grinding. In addition, metal smeared into pores can be removed by an intermediate etch between polishing steps.

SECTIONING
MAXCUT abrasive blade (MAX-C, MAX-D or MAX-E series)

MOUNTING
Castable epoxy or acrylics with vacuum impregnation

ETCHANT
See recommendations for specific alloys.

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on a Metal Mesh Cloth	DIALUBE Diamond Extender	5-10 lbs	200/200 rpm	Until plane
9 micron DIAMAT diamond on POLYPAD polishing pad	DIALUBE Diamond Extender	5-10 lbs	200/200 rpm	3 minutes
6 micron DIAMAT diamond on TEXPAN polishing pad	DIALUBE Diamond Extender	5-10 lbs	200/200 rpm	3 minutes
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Diamond Extender	5-10 lbs	200/200 rpm	3 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

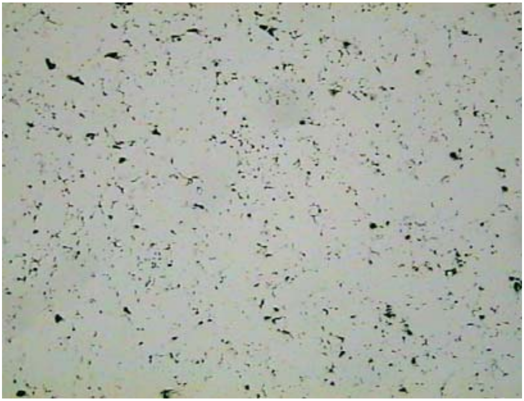


Figure 11-26 Sintered Iron, Mag. 100X (B.F.), as polished.

11.3.2 Cast Irons

Description:
Cast irons are used for many applications. The reliability of cast iron depends on the microstructure of the graphite in the material. If the graphite forms flakes, then the cast iron will be brittle, becoming more so over time. Cast iron with the addition of either magnesium or cerium, results in the graphite forming nodules. Nodular cast iron has much better ductility than gray cast iron (see figures 11-27a and 11-27b).

Preparation Challenge:
Cast Irons are difficult materials to prepare properly because the graphite nodules or the graphite flakes are easily fractured and pulled out during preparation. By minimizing the sectioning damage and starting with a modest grit size alumina paper, retaining these difficult particles can be accomplished.

SECTIONING
MAXCUT abrasive blade (MAX-D or MAX-I series)

MOUNTING
Compression mounting with Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
P500 grit ALO paper P1200 grit ALO paper	Water	5-10 lbs	200/200 rpm	Until plane 1 minute
1 micron DIAMAT diamond on GOLDPAD polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

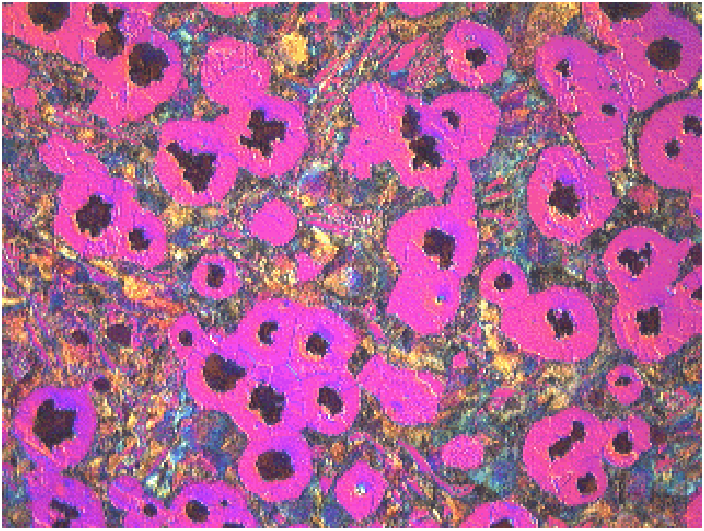


Figure 11-27a Nodular cast iron, 100X (DIC), Etchant 2% Nital.

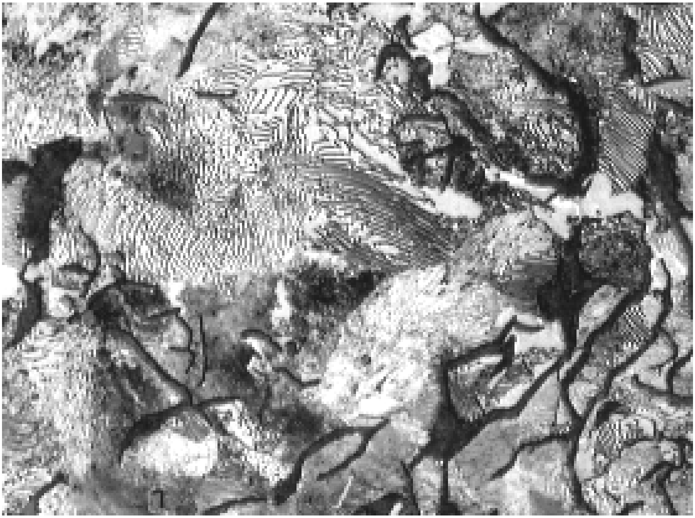


Figure 11-27b Gray cast iron, 500X (B.F), Etchant 2% Nital.

11.3.3 White Irons

Description:
White cast irons are hard and brittle wear-resistant metals consisting of pearlite and cementite. White cast irons are manufactured by chilling the surface of the cast mold in order to prevent the formation of graphite during solidification. White cast irons are used for train car wheels, brake shoes, shot blasting nozzles, mill liners, crushers, pump impellers and other abrasion resistant parts.

Preparation Challenge:
Specimen preparation is relatively straight forward for white cast iron.

SECTIONING
MAXCUT abrasive blade (MAX-D or MAX-I series)

MOUNTING
Phenolic, Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
240 (P220) grit SiC paper 360 (P500) grit SiC paper 600 (P1200) grit SiC paper 800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	200/200 rpm	Until plane 1 minute 1 minute 1 minute 1 minute
3 micron DIAMAT diamond on TEXPAN polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	3 minutes
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

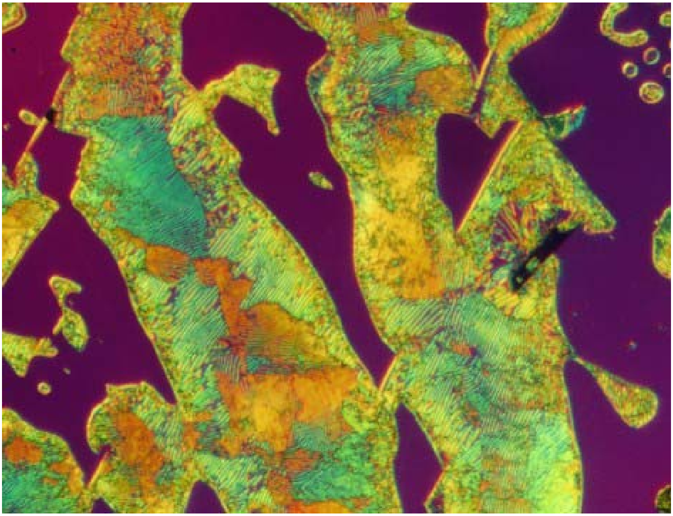


Figure 11-28a White Iron - Hypo eutectic, 1000X (DIC), Etchant: Picral.

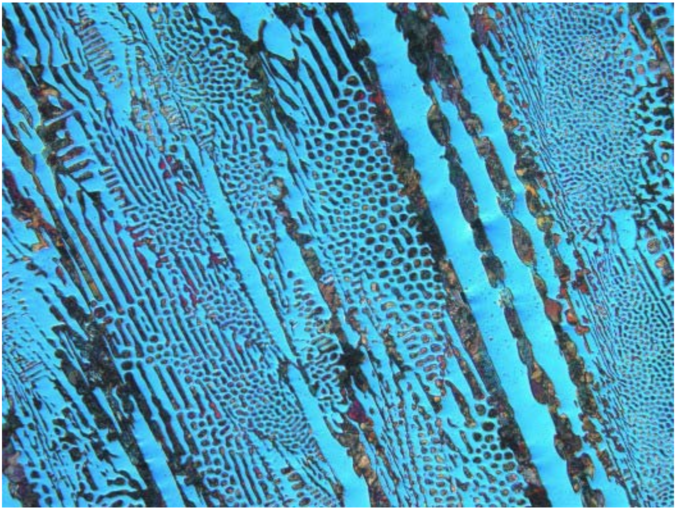


Figure 11-28b White Iron - Hyper eutectic, 200X (DIC), Etchant: Picral.

Table XLII. Selected Cast Iron Etchants

Name/Type	Application	Composition	Conditions
Nital (Microetchant) (72)	Most common etchant for pure iron, low carbon steels, alloy steels, and gray cast iron; segregation can cause uneven attack	100 ml ethanol or methanol (95 %), 1-10 ml HNO_3	Etch for a few seconds to minutes: **EXPLOSIVE - do not exceed 10 % solution**
Ferrite etchant (Microetchant) (73)	Color etching; cementite colored red-bright; ferrite remains bright; phosphides and silicon colors blue-green	2 ml HCl (35 %), 0.5 ml selenic acid, 100 ml ethyl alcohol (95%)	After polishing, immerse for 5-6 minutes; if pre-etching in 2% Nital, immersion time is shorter 2-3 minutes at room temperature
Beraha's tint etch (73)	Beraha's tint etchant for cast iron, carbon, alloy steels and Mn steels	100 ml DI water, 6 gm potassium metabisulfite, 2 gm sulfamic acid	Use at 20°C; reagents active for 2-4 hours; discard when yellow; Cd and Zn coatings inhibit staining; etch for 45 seconds to 4 minutes
Ferrite etchant (Microetchant) (74)	Color etching; after short immersion time (20-40 sec), only ferrite is colored (red or violet); after longer immersion time, all phases are colored: phosphides (brown-orange), ferrite (yellow or light blue), cementite (red-violet or blue)	240 gm sodium thiosulfate, 30 gm citric acid, 20-25 gm cadmium chloride, 1000 ml DI water	Pre-etch in 2 % Nital; immersion at room temperature (after filtering sulfur particles) for 20-90 seconds

Table XLII (conti.) Selected Cast Iron Etchants

Name/Type	Application	Composition	Conditions
Ferrite etch (Microetchant) (73)	Color etching; spectacular coloration of ferrite grains as a function of crystallographic orientation	3 gm potassium metabisulfite, 10 gm sodium thiosulfate, anhydrous, 100 ml DI water (use after 4% Picral pre-etch)	Pre-etch with 4% Picral, 1-2 minute; immersion at room temperature for 2 minutes (until surface turns bluish -red)
Picral etchant (Microetchant) (74)	Generally used for iron and heat treated steels; pearlite, martensite and bainite; uniform etching, even with segregations; Fe_3C stained light yellow	100 ml ethanol (96 %), 2-4 gm picric acid	Etch for a few seconds to minutes. **CAUTION: TOXIC**
Klemm's reagent (Microetchant) (75)	Phosphorus distribution in cast steel and cast iron	50 ml saturated aqueous solution of thiosulfate, 1gm potassium metabisulfite	Etch for a few seconds to minutes

11.4 CLASS 4 - SOFT, BRITTLE NONMETALS (Electronics)

Examples include silicon, GaAs, ferrites, PZT's, MEMS devices.

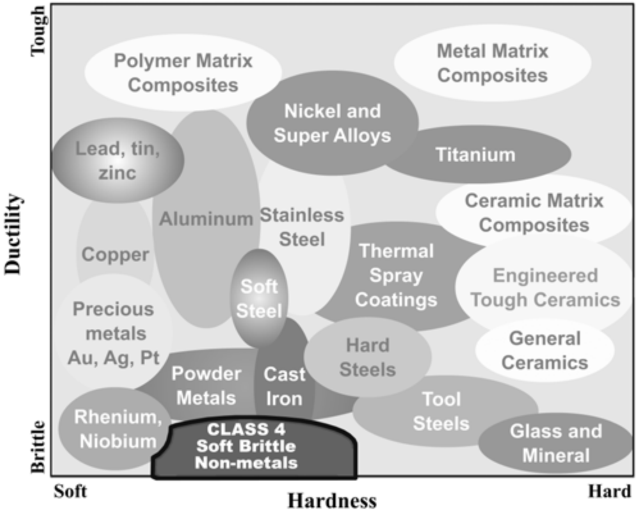


Figure 11-29 Class 4 - Soft, brittle nonmetals and electronics.

Table XLIII. Preparation Guidelines for Class 4 Materials

Preparation Step	Soft Substrates	Hard Substrates
Rough Grinding	320 (P360) grit SiC paper 400 (P800) grit SiC paper 600 (P1200) grit SiC paper 800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	45 micron Lapping films 30 micron Lapping films 15 micron Lapping films 9 micron Lapping films
Rough Polishing	Diamond on a woven polishing pad	1-3 steps of diamond with woven polishing pads
Final Polishing	Polycrystalline alumina on a woven polishing pad	Colloidal silica on woven or porous urethane polishing pads

11.4.1 Multilayer Ceramic Capacitors

Description: Ceramic capacitors are typically very small, inexpensive devices used in cell phones, MP3 players, computers and other electronic products. MLC devices are constructed of alternating layers of metal and ceramic, with the ceramic material acting as the dielectric.

Preparation Challenge:
Microstructural analysis of a $BaTiO_3$ ceramic capacitor includes looking for missing metal layers, as well as for examining for any gaps and voids in the ceramic substrate. Microstructural preparation of multilayer $BaTiO_3$ ceramic capacitors requires minimizing polishing relief and damage to the coating or ceramic substrate. This is accomplished by filling the existing voids with a castable resin under vacuum and then curing at a higher pressure. Initial grinding with alumina lapping films is required to open up the capacitor, as well as to minimize microstructural damage. Rough and final polishing are recommended on low napped woven polishing pads using diamond and colloidal silica, respectively.

MOUNTING
Castable acrylic resins (CASTAMOUNT, Premium Acrylic)

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
45 micron alumina lapping film 30 micron alumina lapping film	Water	5-10 lbs	100/100 rpm	Until plane 1 minute
15 micron DIAMAT diamond on POLYPAD polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	3 minutes
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	3 minutes
1 micron DIAMAT diamond on ATLANTIS polishing pad	SIAMAT colloidal silica	5-10 lbs	100/100 rpm	2 minutes

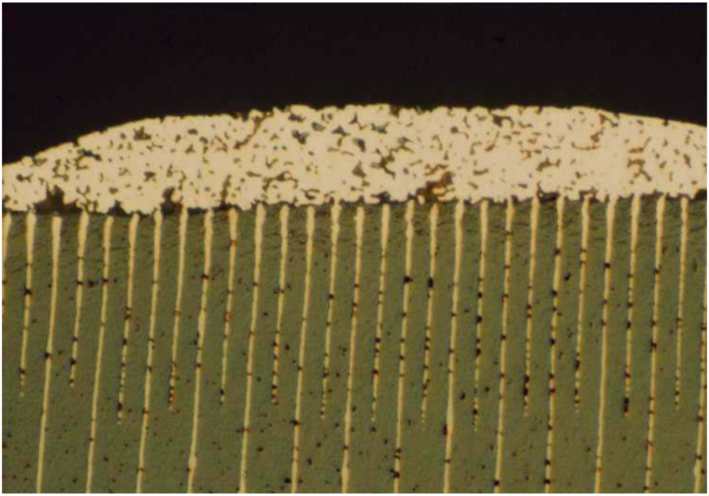


Figure 11-30a BaTiO$_3$ multilayer capacitor, 100X, as polished.

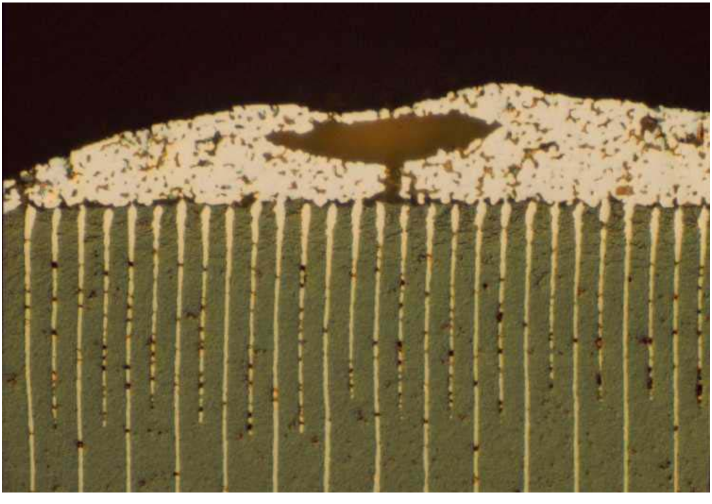


Figure 11-30b BaTiO$_3$ multilayer capacitor with void in metal layer, 100X, as polished.

11.4.2 Electronic Die Packages (Silicon, Plastic, Solder Joints)

Description: Electronic packages can consist of materials having very different mechanical and chemical properties. Materials in electronic packages range from brittle silicon, soft plastics, and solder to very hard dielectric materials, such as alumina.

Preparation Challenge:
The microstructural preparation of electronic packages presents some very difficult challenges, such as abrasives embedding in very soft solder joints, edge rounding, polishing relief between very hard (ceramic) or brittle (silicon) materials and the very soft plastic and metal solders. The use of alumina lapping films are very useful for maintaining flatness and for minimizing fractured abrasive embedding for non-ceramic substrates. For specimens with ceramic substrates, diamond lapping films are recommended.

Table XLIV. Electronic Packaging Components

Typical Material	Package Component	Physical Properties
Alumina, Aluminum Nitride, Beryllium oxide	Packaging body	Hard, brittle
Au/Si	Die attach	Soft
Ni/Fe/Co	Leads/lids	Tough, ductile
Au and/or Ni	Plating	Soft, ductile
Silicon	IC chip	Brittle
Ag/Cu	Lead braze	Soft, ductile
Pb/Sn	Solder	Soft
Tungsten	Coating refractory	Hard, tough, smears easily
Aluminum	Bonded wires	Soft

SECTIONING

Diamond wafering blade - medium grit/low concentration

MOUNTING

Castable epoxy or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ Sample	Speed (Head/Base)	Time
15 micron alumina or diamond lapping film	POLYLUBE Extender	5-10 lbs	100/100 rpm	Until plane
9 micron alumina or diamond lapping film	POLYLUBE Extender	5-10 lbs	100/100 rpm	2 minutes
6 micron alumina or diamond lapping film	POLYLUBE Extender	5-10 lbs	100/100 rpm	2 minutes
3 micron alumina or diamond lapping film	POLYLUBE Extender	5-10 lbs	100/100 rpm	2 minutes
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	1 minute
SIAMAT colloidal silica on MICROPAD 2 polishing pad		5 lbs	100/100 rpm	30 seconds to several minutes

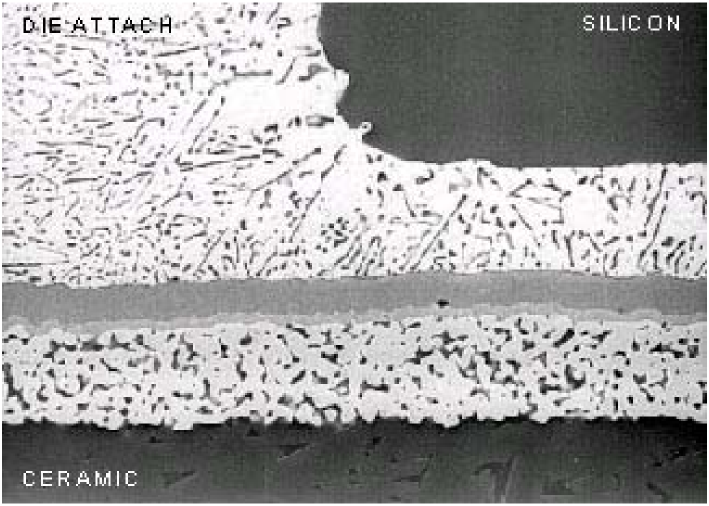


Figure 11-31a Electronic die cross section (compliments of Analog Devices).

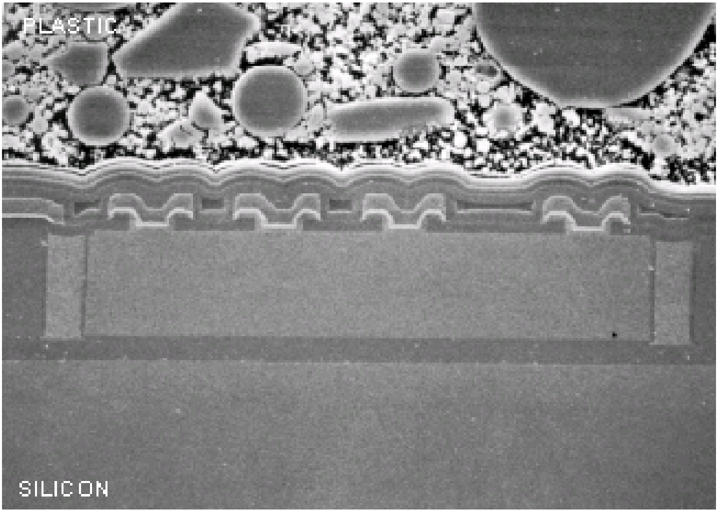


Figure 11-31b Electronic die cross section (compliments of Analog Devices).

11.4.3 MEMS (Microelectromechanical System) Devices

Description: Microelectromechanical systems (MEMS) devices are also referred to as micromachines. MEMS devices are made up of components varying in size between 1 micron to 0.1 mm.

Preparation Challenge:
Microstructural cross section preparation of MEMS devices presents the same challenges as polishing other specimens having material properties ranging from very hard and brittle (silicon, ceramics, etc.) to soft and ductile (soft metals, metallized layers, sputter coating, etc.). Specimen preparation must eliminate any preparation induced artifacts, as well as maintain the planarity of the specimen.

SECTIONING
Diamond wafering blade - medium grit/low concentration (if required)

MOUNTING
Castable epoxy or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
600 (P1200) grit SiC grinding paper	Water	5-10 lbs	100/100 rpm	Until plane
1 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	100/100 rpm	3-5 minutes
SIAMAT colloidal silica on BLACKCHEM 2 polishing pad		10 lbs	100/100 rpm	3-5 minutes

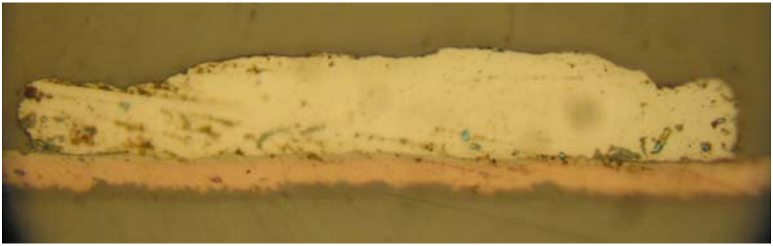


Figure 11-32a Aluminum-silicon substrate MEMS device with a gold bond on a nickel intermediate layer, Mag. 200X, as polished.

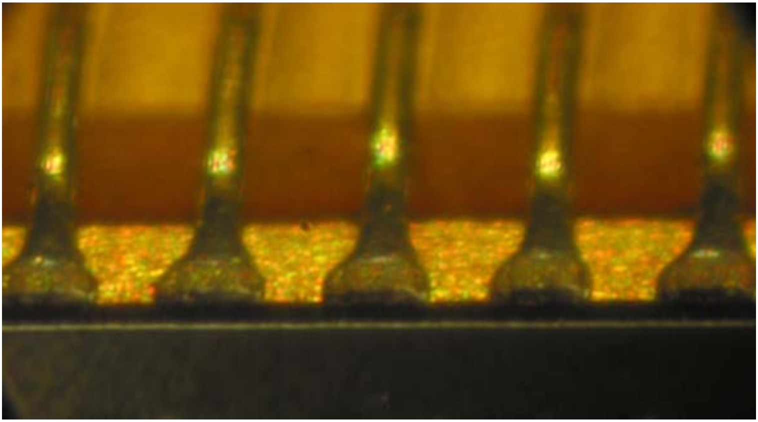


Figure 11-32b Wire bond for MEMS device, 200X, as polished.

11.4.4 PZT (piezoelectric) Devices

Description: PZT, or piezoelectric, devices are very useful because they generate a voltage when mechanically deformed or vice versa.

Preparation Challenge:
PZT's are typically composed of lead, zirconium and titanate, which are processed at extremely high temperatures. PZT devices present the same challenges as polishing other brittle materials; however, they present the additional challenge of preparing other materials with significantly different properties (packaging materials, solders, coating, etc.). Specimen preparation must eliminate the preparation induced artifacts, as well as maintain the planarity of the specimen.

SECTIONING
Diamond wafering blade - medium grit/low concentration (if required)

MOUNTING
Castable epoxy or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
1200 (P4000) grit SiC grinding paper	Water	5 lbs	100/100 rpm	Until plane
1 micron DIAMAT diamond on GOLDPAD polishing pad		10 lbs	100/100 rpm	3-5 minutes
0.05 micron Nanometer Alumina on BLACKCHEM 2 polishing pad		10 lbs	100/100 rpm	3-5 minutes
SIAMAT 2 colloidal silica on BLACKCHEM 2 polishing pad		10 lbs	100/100 rpm	1 minute

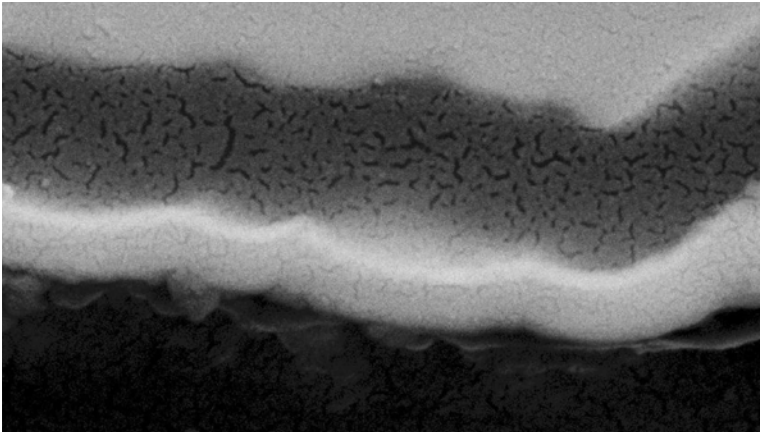


Figure 11-33a PZT cross section with sputter coating, 200,000X, as polished.

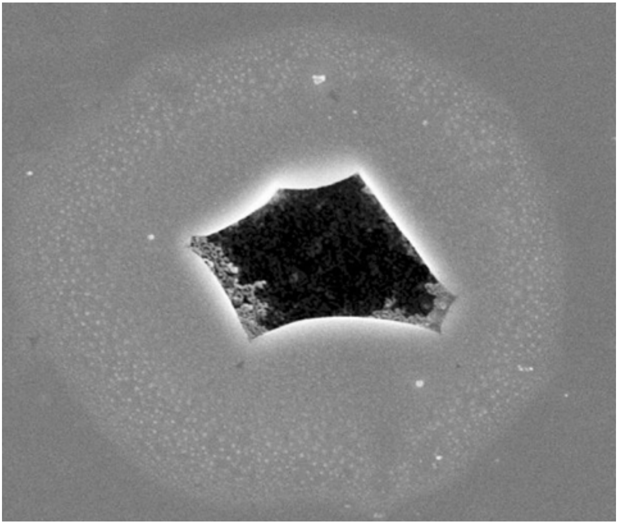


Figure 11-33b Undesirable void is PZT, 1,000X, as polished.

11.4.5 Gallium Arsenide substrates

Specimen preparation of extremely friable or brittle materials, such as GaAs, offer a microstructural preparation challenge. Proper microstructural preparation of these materials must minimize fracturing of the GaAs. This is accomplished by first cutting or sectioning with fine grit / low concentration diamond blades. Often times microelectronic cross sections are not encapsulated and are only mounted using a hot melt tape. If the specimens are encapsulated, a castable mounting compound such as an acrylic or an epoxy is recommended. Grinding and polishing is recommended with diamond lapping films to prevent undue damaging the GaAs substrate. Polishing is accomplished on low napped polishing cloths using the addition of hypochlorite to diamond for a CMP polishing operation.

SECTIONING
Diamond wafering blade - medium grit/low concentration

MOUNTING
Castable epoxy or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ Sample	Speed (Head/Base)	Time
15 micron alumina or diamond lapping film	POLYLUBE Extender	5-10 lbs	100/100 rpm	Until plane
9 micron alumina or diamond lapping film	POLYLUBE Extender	5-10 lbs	100/100 rpm	2 minutes
6 micron alumina or diamond lapping film	POLYLUBE Extender	5-10 lbs	100/100 rpm	2 minutes
3 micron alumina or diamond lapping film	POLYLUBE Extender	5-10 lbs	100/100 rpm	2 minutes
1 micron DIAMAT diamond on ATLANTIS polishing pad	HOCl- (hypochlorite-bleach)	5-10 lbs	100/100 rpm	1 minute

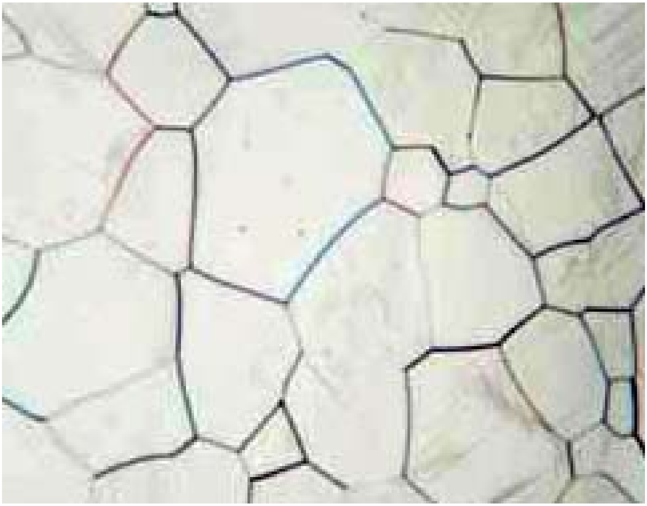


Figure 11-34 GaAs, etched with HOCl-, mag. 1000X, DIC.

11.4.5 Electronic Metallized Ceramics (Alumina, BeO, AlN)

Description: Ceramics such as alumina, beryllia (BeO) and aluminum nitride (AlN) are commonly used ceramic substrate materials for metallizing because they provide thermal conductivity and electrical resistivity.

Preparation Challenge:
Microstructural cross section preparation of metallized layers are difficult because of edge rounding and phase relief. In order to maintain the integrity of the metallic layers, the specimen must first be sectioned properly to avoid chipping and cracking of the ceramic substrate/metal interface. The use of SIAMAT colloidal silica also provides a chemical mechanical polishing (CMP) action, which is the most effective means for eliminating both surface and subsurface damage.

SECTIONING
Diamond wafering blade - medium grit / low concentration

MOUNTING
Castable epoxy or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	5-10 lbs	200/200 rpm	5 minutes
1 micron DIAMAT diamond on ATLANTIS polishing pad	SIAMAT colloidal silica	5-10 lbs	200/200 rpm	5 minutes
SIAMAT colloidal silica on TEXPAN polishing pad		5-10 lbs	100/100 rpm	2 minutes

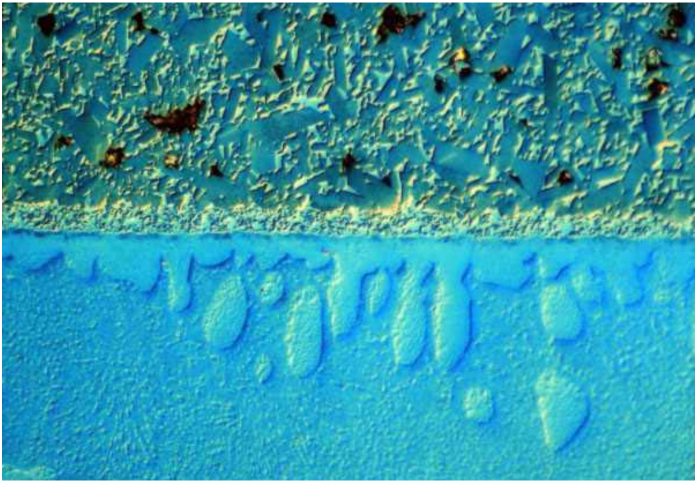


Figure 11-35a BeO substrate with nickel, copper metallized coating, 400X (DIC), as polished.

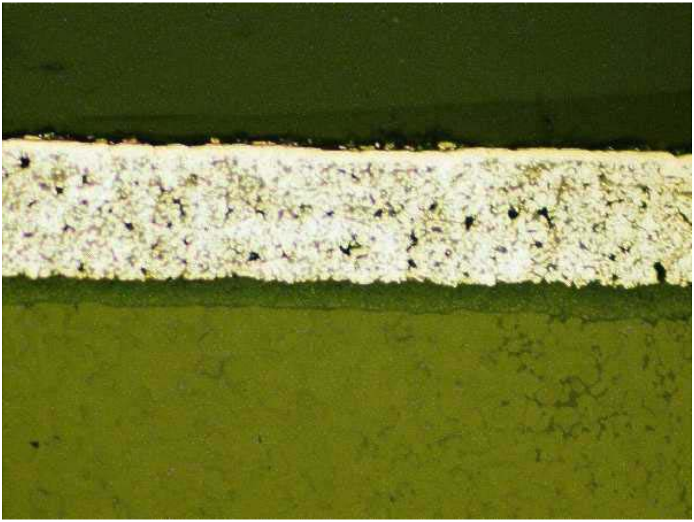


Figure 11-35b AlN substrate with molybdenum/nickel, gold metallized coating, 400X (Brightfield), as polished.

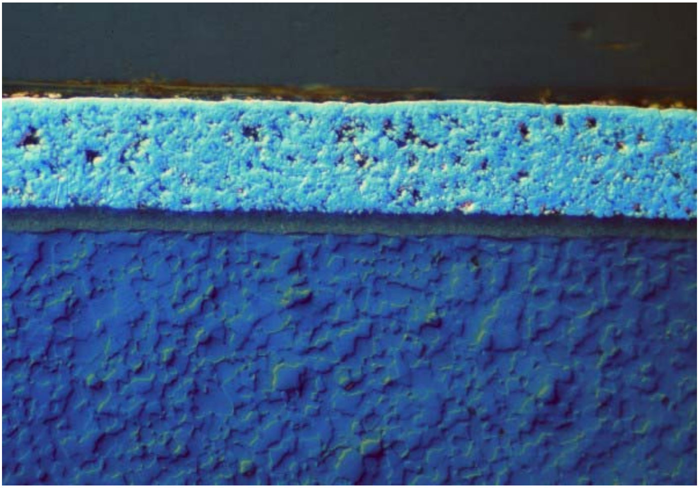


Figure 11-36 AlN substrate with molybdenum/nickel, gold metallized coating, 400X (DIC), as polished.

11.4.6 Magnetic Ceramics (Ferrite)

Description:
Ferrite ceramics produce one of the strongest types of magnetics, and consist of iron, boron and barium, or strontium and molybdenum. Ferrite ceramic magnets have high magnetic permeability, which allows them to store stronger magnetic fields than iron.

Preparation Challenge:
Specimen preparation of friable or brittle materials such as ferrites can be difficult. Proper microstructural preparation of these materials must maintain the structure of the Ni-Fe ferrite structure. This is accomplished by mounting the ferrite in a castable mounting compound such as epoxy. Initial grinding with a 320 (P360) grit or finer SiC paper is required to prevent pulling out the ferrite particles, especially the smaller particles. Rough polishing is accomplished on woven polishing cloths using diamond, with final polishing on high napped cloths using a polycrystalline alumina abrasive.

MOUNTING
Castable mounting resins (epoxies or acrylics)

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
240 (P220) grit SiC paper 360 (P500) grit SiC paper 600 (P1200) grit SiC paper 800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	100/100 rpm	Until plane 1 minute 1 minute 1 minute 1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on NAPPAD polishing pad		5-10 lbs	100/100 rpm	1 minute

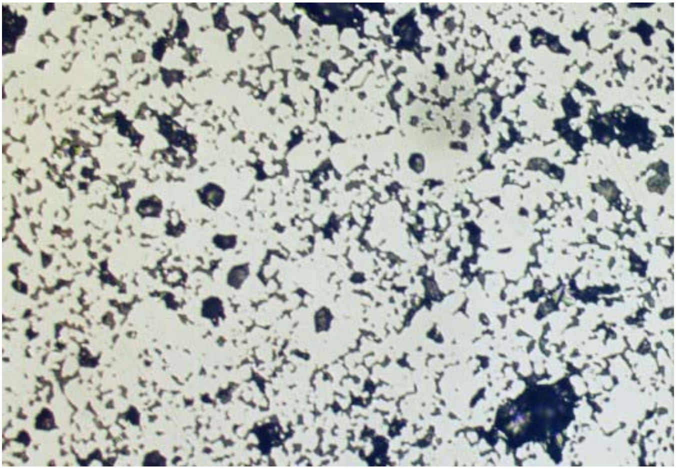


Figure 11-37 Ni-Zn ferrite, 400X (B.F.), as polished.

Table XLV. Selected Magnetic Ferrite Etchants

Name/Type	Application	Composition	Conditions
Ni-Fe (Microetchant) (76)	For nickel-zinc ferrite and nickel-ferrite magnetic alloys	15 ml lactic acid, 15 ml HNO_3, 5 ml HF	Use at 65-80°C for 20 minutes
Ferrite etchant (Microetchant) (76)	For ferrite and garnets magnetic alloys	10 ml HF, 10 ml HNO_3, 20 ml water	Use at 60-90°C for a few seconds to 30 minutes
Garnet (76)	For garnet magnetic alloys	H_2SO_4 (concentrated)	Immerse up to 30 minutes at 115°C; use with care; **a face shield is recommended**

11.5 CLASS 5 - MEDIUM HARD, DUCTILE METALS

Examples include stainless steel, soft and medium hard steels.

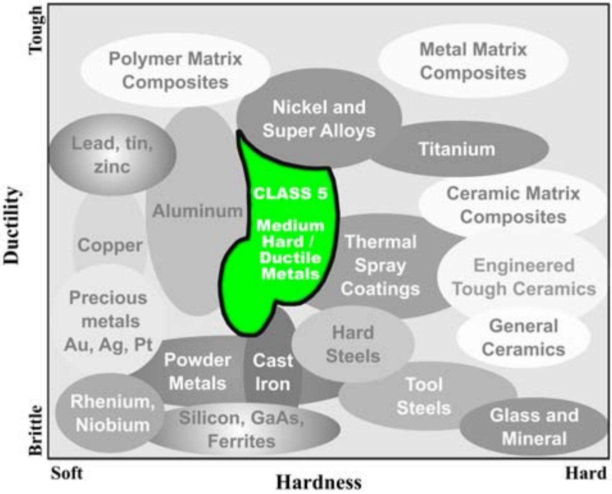


Figure 11-38 Class 5 - Medium hard, ductile metals.

Table XLVI. Preparation guidelines for Class 5 Materials

Preparation Step	Basic Recommendation
Rough Grinding	320 (P360) grit SiC paper 400 (P800) grit SiC paper 600 (P1200) grit SiC paper 800 (P2400) grit SiC paper 1200 P4000)grit SiC paper
Rough Polishing	1 micron diamond on a woven polishing pad
Final Polishing	Polycrystalline alumina on a woven polishing pad

11.5.1 Soft to Medium Hard Steels

Description:
Steels are ferrous alloys of iron containing relatively low concentrations of carbon (<2%). Steels also have a wide range of properties due to their ability to be heat treated and annealed. Depending upon the carbon concentration and other alloying elements, the microstructure of steel can be modified by heating, quenching and stress relief (annealing). Common microstructures include pearlite and ferrite for slow cooled low carbon steels and martensite for fast cooled high carbon steels. Perlite and ferrite are relatively soft and ductile, whereas, martensite is hard and brittle.

Preparation Challenge:
Metallographic specimen preparation is relatively straight forward.

SECTIONING
MAXCUT abrasive blade (MAX-D or MAX-I series)

MOUNTING
Phenolic, epoxy or diallyl phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/base)	Time
240 (P220) grit SiC paper 360 (P500) grit SiC paper 600 (P1200) grit SiC paper 800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	200/200 rpm	Until plane 1 minute 1 minute 1 minute 1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

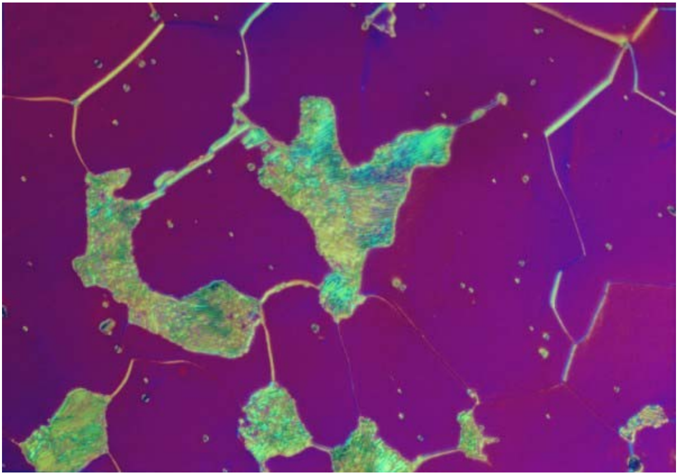


Figure 11-39a 1018 Low Carbon Steel, furnace cooled, 1000X (polarized light) Etchant 2% Nital. Structure is ferrite and pearlite.

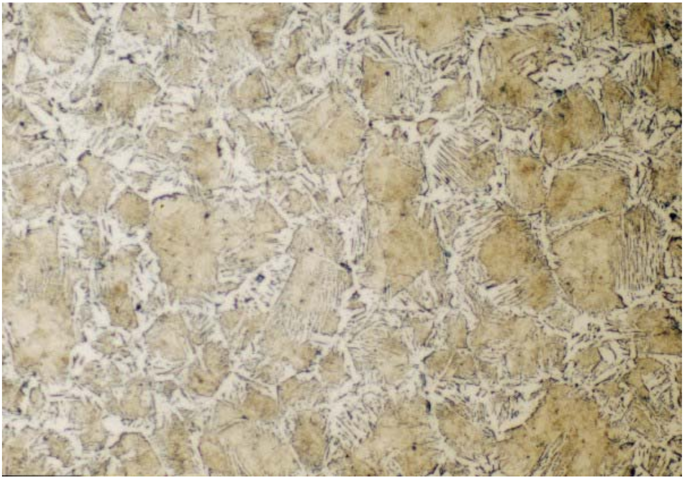


Figure 11-39b 1018 Low Carbon Steel, quenched, 400X (B.F.), Etchant 2% Nital. Structure is Bainite.

11.5.2 Steel Welds

Description:
Welding is a very common way of joining two pieces of steel together. Cross sectioning a weld is the best way to examine the quality of the weld and to either qualify a welder or to calibrate automated welding machines. The main dimensional features which are important in welding include throat height or weld penetration, leg length or how far the weld extends along each piece of metal, undercut, etc.

Preparation Challenge:
Metallographic specimen preparation is relatively straight forward. In addition, etching the specimen can significantly improve the welds visual characteristics.

SECTIONING
MAXCUT abrasive blade (MAX-D or MAX-I series)

MOUNTING
Phenolic, epoxy or diallyl phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
240 (P220) grit SiC paper 360 (P500) grit SiC paper 600 (P1200) grit SiC paper 800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	200/200 rpm	Until plane 1 minute 1 minute 1 minute 1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

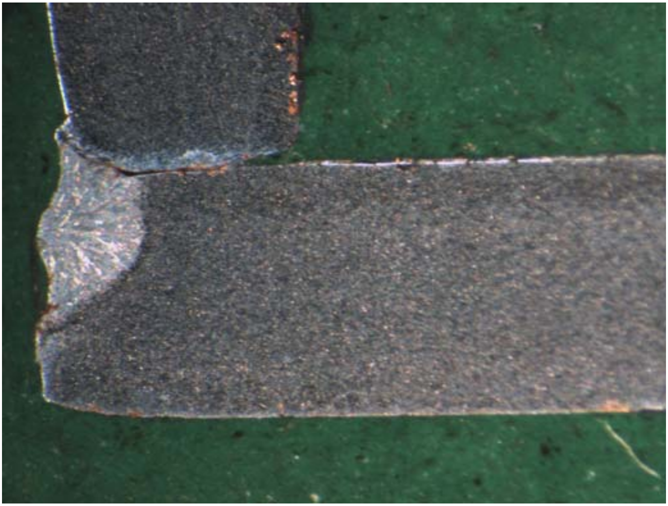


Figure 11-40a Steel weld, 50X, Etchant 2% Nital.

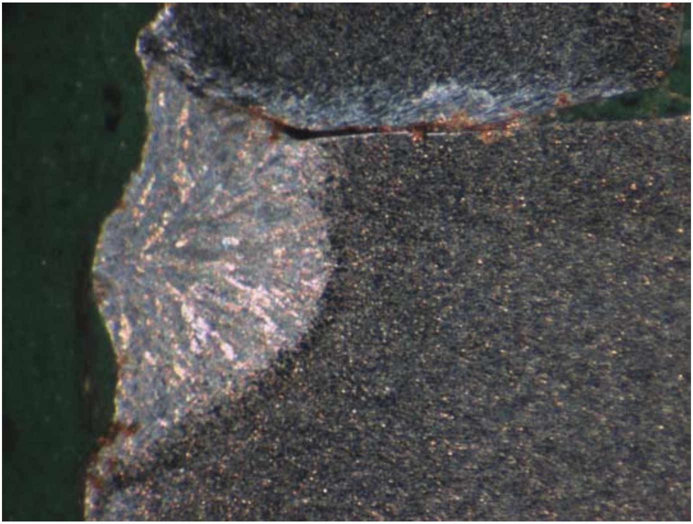


Figure 11-40b Steel weld, 200X, Etchant 2% Nital.

Table XLVII. Selected Etchant - Soft to Medium Hardness Steels

Name/Type	Application	Composition	Conditions
Nital (Microetchant) (75)	Most common etchant for pure iron, low carbon steels, alloy steels, and gray cast iron; segregation can cause uneven attack	100 ml ethanol or methanol (95%), 1-10 ml nitric acid	CAUTION: **EXPLOSIVE** - do not exceed 10 % solution
Picral (Microetchant) (75)	Generally used for iron and heat treated steels; pearlite, martensite and bainite; uniform etching, even with segregations; Fe_3C stained light yellow	100 ml ethanol (96%), 2-4 gm picric acid	CAUTION: **TOXI**C - etch for a few seconds to minutes
Beraha's tint etch (Microetchant) (77)	Reveals substructure, martensite grain boundaries, flow lines; tint etch for irons, steels, and tool steels; colors ferrite, martensite, bainite, pearlite; carbide, nitride, and phosphide are unaffected	5-10 ml HCl, 1000 ml water	Before use, add 1 gm potassium metabisulfite per 100 ml solution; good for a few hours; agitate strongly during etching, then hold motionless until surface is colored, 10-60 seconds total time
Modified Murakami's reagent (80)	Selective carbide etchant; cementite darkened, pearlite brown	1-4 gm $K_3Fe(CN)_6$, 10 gm KOH (or 7 gm NaOH), 100 ml DI water	Etch up to 15 seconds in boiling solution; use fresh!

Table XLVII (conti.) Etchants-Soft to Medium Hardness Steels

Name/Type	Application	Composition	Conditions
Amyl nital (Microetchant) (79)	For pearlite steels, low concentrations for galvanized steels; sometimes more sensitive than Nital	0.5-5 ml HNO3, 100 ml amyl alcohol	Use in fume hood; ***do not store***
Goerens' amyl picral (Microetchant) (79)	Recommended for etching very fine pearlite	100 ml amyl alcohol, 5 gm picric acid	Use in fume hood; ***do not store***
Color etchant (Microetchant) (80)	Colors pearlite and hardened structures of unalloyed steels; ferrite colored brown-black (dark red-dark violet); carbides, phosphides and nitrides remain white	50 ml cold-saturated (in distilled water) sodium thiosulfate solution, 1gm potassium metabisulfite	Immersion at room temperature for 40-120 seconds
Vilella's reagent (Microetchant) (81)	Recommended for annealed structures or those containing pearlite or bainite; does not reveal ferrite grain boundaries in annealed specimens	1 gm picric acid, 5 ml HCl, 100 ml ethanol	Use by immersion
Superpicral (Microetchant) (81)	Recommended for annealed structures or those containing pearlite or bainite; does not reveal ferrite grain boundaries in annealed specimens	10 gm picric acid, 100 ml ethanol	Must be heated to dissolve picric acid; use by immersion, up to 1 minute or more

11.5.3 Stainless Steel

Description:
Stainless steels have high concentrations of chromium (>12%) and are generally relatively soft compared to heat treated steels. This makes stainless steel more susceptible to smearing.

Preparation Challenge:
Metallographic specimen preparation is relatively straight forward; however, etching can be more difficult due to the corrosion resistance of stainless steels.

SECTIONING
MAXCUT abrasive blade (MAX-E or MAX-I series)

MOUNTING
Phenolic, epoxy or diallyl phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
240 (P220) grit SiC paper 360 (P500) grit SiC paper 600 (P1200) grit SiC paper 800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	200/200 rpm	Until plane 1 minute 1 minute 1 minute 1 minute
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

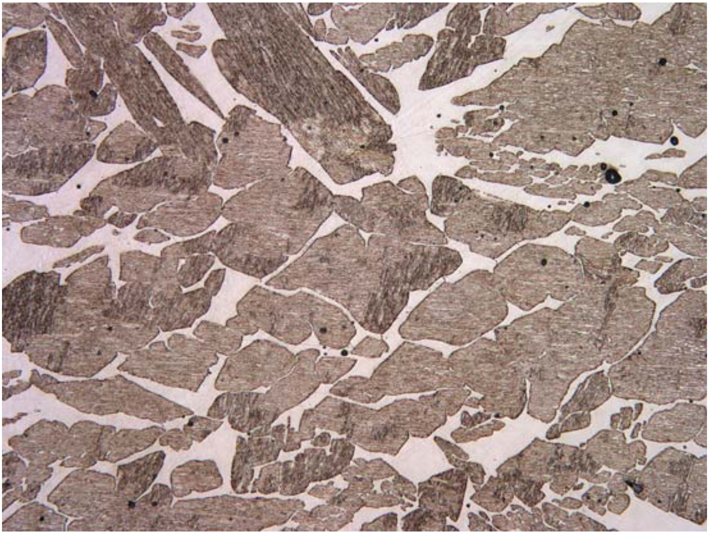


Figure 11-41a 431 Stainless Steel, 200X (BF), Etchant Modified Murakami's.

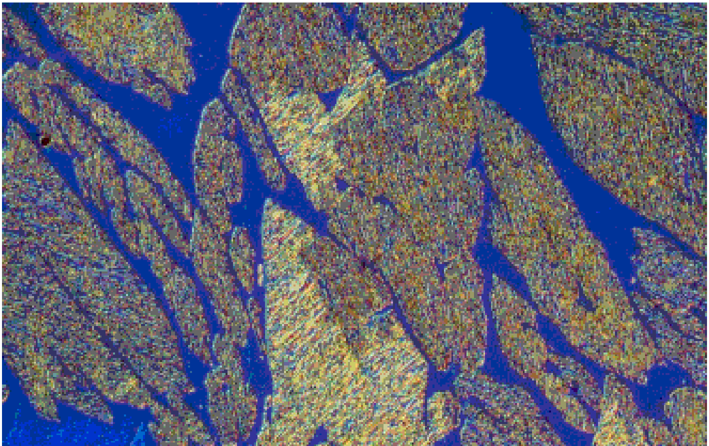


Figure 11-41b 431 Stainless Steel, 400X (DIC), Etchant Modified Murakami's.

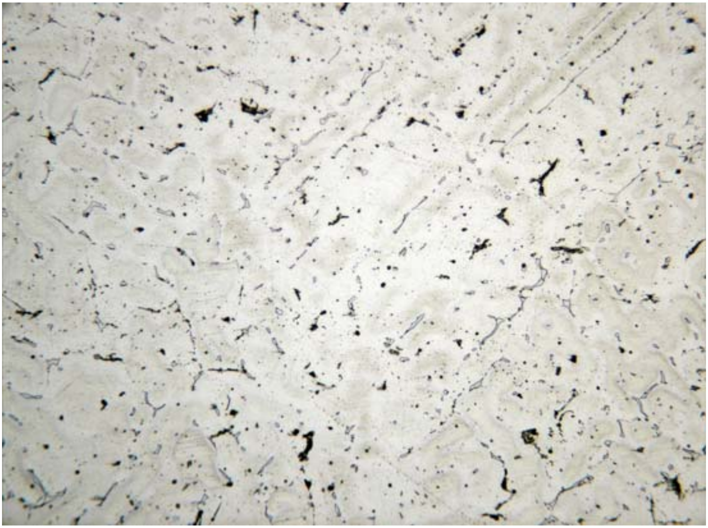


Figure 11-41c 300 Series Stainless Steel, 200X (BF), Etchant Oxalic Acid.

Figure 11-41d 400 Series Stainless Steel, 200X (BF), Etchant Viella's.

Table XLVIII. Selected Stainless Steel Etchants

Name/Type	Application	Composition	Conditions
Stainless Steel (Macroetchant) (82)	Wrought stainless steel	50 ml HCl, 25 ml saturated $CuSO_4$ in water	Etch at 75°C; immerse until the desired degree of contrast is obtained
Stainless Steel (Macroetchant) (82)	Wrought stainless steel	10-40 ml HNO_3, 3-10 ml 48% HF, 25-50 ml DI water	Etch at 70-80°C; immerse until the desired degree of contrast is obtained
Stainless Steel (Macroetchant) (82)	Wrought stainless steel	1 part HCl, 1 part DI water	Etch at 70-80°C for 15-45 minutes; desmut by dipping in warm 20% aqueous HNO_3 solution to produce a bright surface
Lepito's No. 1 etch (Macroetchant) (82)	Wrought stainless steel	**Solution a**: 5 gm $(NH_4)_2S_2O_8$, 50 ml H_2O, **Solution b**: 250 gm $FeCl_3$, 100 ml HCl, **Solution c**: 30 ml HNO3	Combine (a) and (b), then add (c); immerse specimen at room temperature; use fresh!
Marble's reagent (Macroetchant) (82)	Wrought stainless steel	50 ml HCl, 10 gm $CuSO_4$, 50 ml DI water	General purpose macroetch.

11.6 CLASS 6 - TOUGH, HARD NONFERROUS METALS

Examples include titanium, Inconel, Ni-Cr alloys, superalloys, nickel and cobalt.

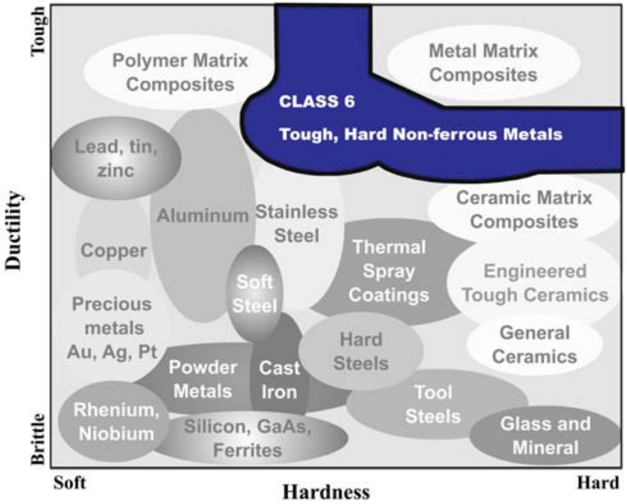


Figure 11-42 Class 6 - Tough, hard nonferrous metals.

Table XLIX. Preparation Guidelines for Class 6 Materials

Preparation Step	Basic Recommendation
Rough Grinding	240 (P220) grit SiC paper 360 (P500) grit SiC paper 600 (P1200) grit SiC paper
Rough Polishing	2-3 diamond polishing steps on woven polishing pads
Final Polishing	Polycrystalline alumina on napped polishing pad

11.6.1 Superalloys

Description:
Superalloys are high-performance alloys which exhibit excellent mechanical strength and creep resistance at high temperatures, good surface stability, and corrosion and oxidation resistance. The base elements of Superalloys are nickel, cobalt, or nickel-iron metals.

Preparation Challenge:
Metallographic specimen preparation is relatively straight forward.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Phenolic, epoxy or diallyl phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
240 (P220) grit SiC paper 360 (P500) grit SiC paper 600 (P1200) grit SiC paper	Water	5-10 lbs	200/200 rpm	Until plane 1 minute 1 minute
9 micron DIAMAT diamond on POLYPAD polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
6 micron DIAMAT diamond on TEXPAN polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

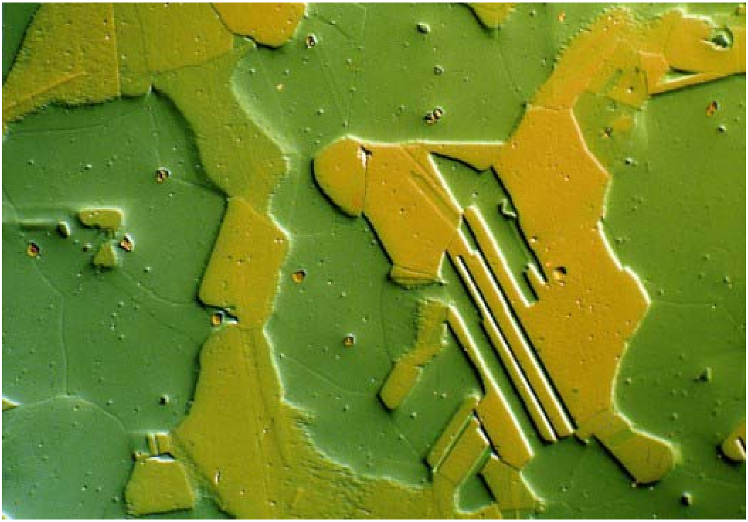


Figure 11-43 Nimonic 90 Superalloy, 400X (DIC).

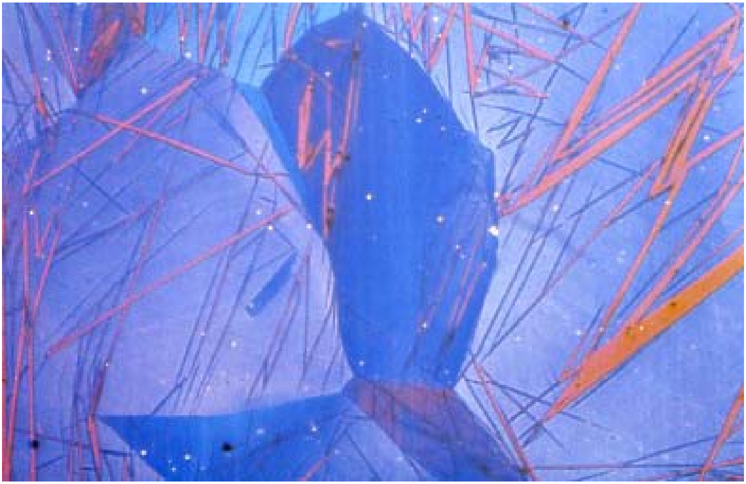


Figure 11-44 Fe-Ni-Co-Al Alloy, 400X (Polarized Light).

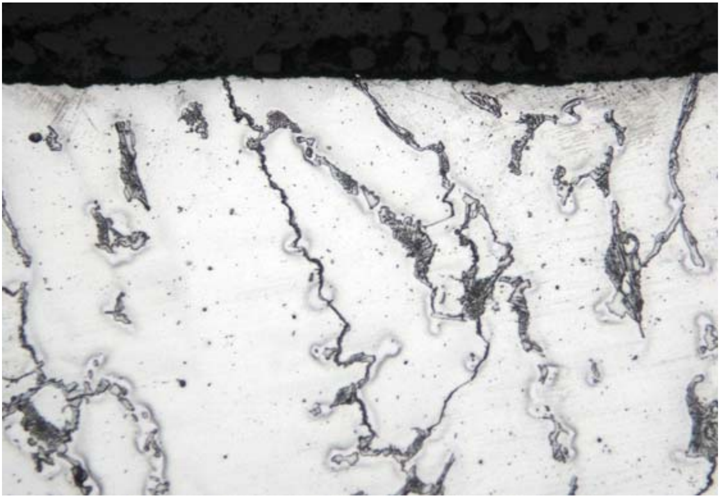


Figure 11-45 Cast cobalt alloy, 200X (BF).

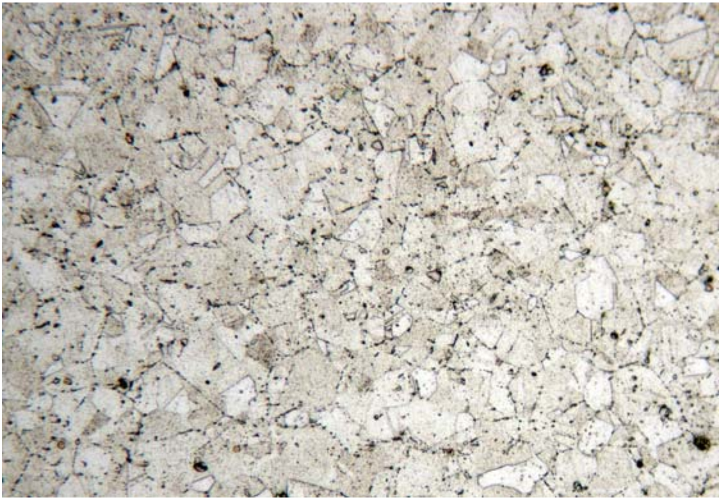


Figure 11-46 Wrought nickel, 200X (BF), Etchant HCl and H_2O_2.

Table L. Selected Etchants for Superalloys

Name/Type	Application	Composition	Conditions
Marble's reagent (Macroetchant) (83)	For Co high-temperature alloys	50 ml HCl, 10 gm $CuSO_4$, 50 ml DI water	Immerse or swab sample for up to 1 minute; can add a few drops of H_2SO_4 to increase reaction
Cobalt Superalloys (Microetchant) (84)	Popular etch for cobalt-base superalloys	100 ml HCl, 5 ml 30% H_2O_2	1-10 seconds; mix fresh; can use up to 20% H_2O_2
Iron Superalloys (Macroetchant) (85)	For iron-nickel and nickel base alloys	50 ml saturated aqueous $CuSO_4$, 50 ml HCl	Swab or immerse at room temperature
Murakami's reagent (Microetchant) (86)	For iron-base and nickel-base superalloys	10 gm $K_3Fe(CN)_6$, 10 KOH, 100 ml DI water	Use hot (75°C) to darken epsilon phase; use at room temperature to darken carbides
Aqua regia (Microetchant) (86)	For iron-base and nickel-base superalloys; outlines carbides, reveals grain boundaries	20 ml HNO_3, 60 ml HCl	Immerse or swab 5-60 seconds; use in fume hood, ***do not store***

Table L (conti.). Selected Etchants for Superalloys

Name/Type	Application	Composition	Conditions
Iron Superalloys (Macroetchant) (86)	For iron-nickel and nickel based alloys	5 ml H_2SO_4, 3 ml HNO_3, 92 ml HCl	Add H_2SO_4 to HCl, stir, allow to cool, add HNO_3; swab 10-30 seconds; use in fume hood; do not store; discard when solution turns orange
Waterless Kalling's reagent (Microetchant) (86)	For iron-base and nickel-base superalloys	5 gm $CuCl_2$, 100 ml HCl, 100 ml ethanol	Immerse or swab fora few minutes
Glyceregias (Microetchant) (86)	Very popular general etch for iron-base and nickel-base superalloys; gamma prime in relief	3 parts glycerol, 2-3 parts HCl, 1 part HNO_3	Mix fresh, do not store; use by immersion or swabbing 5-60 seconds; discard when solution turns orange
Beraha's tint etch (Mircroetchant) (87)	Tint etching of superalloys; colors carbides and gamma prime, matrix unaffected	1-3 ml selenic acid, 20-30 ml HCl, 100 ml ethanol	Immerse sample 1-4 minutes at 20°C
Ni-Cr-Fe (Macroetchant) (88)	Macroetchant for Ni and Ni-base alloys; Ni-Cu alloys; Ni-Cr-Fe alloys; grain size in superalloys	50 ml DI water, 50 ml ethanol, 50 ml HCl, 10 gm $CuSO_4$	Etch for seconds to minutes

Table L (conti.). Selected Etchants for Superalloys

Name/Type	Application	Composition	Conditions
Inconel (Macroetchant) (88)	Inconel-type alloys on NiCr and Ni-Fe-Cr basis; Ni-Nb, Ni-Ta, Ni-Si, Ni-Co-Cr alloys.	20-30 ml DI water, 0-20 ml HNO_3, 20 ml HCl, 10 ml H_2O_2 (30%), (concentration variable)	Etch seconds to minutes; use fresh only!
Nickel Superalloys (Macroetchant) (85)	For nickel-base superalloys	200 gm $FeCl_3$, 200 ml HCl, 1000 ml DI H_2O	Etch up to 90 minutes at 100°C
Nickel Superalloy (Macroetchant) (88)	Macroetchant for superalloys	125 ml Aqueous solution of $FeCl_3$, 600 ml HCl, 18.5 ml HNO_3	Etch for 5-10 minutes; boiling
Nickel Superalloy Microetchant) (86)	For hard to etch solution-treated nickel-base alloys	10 ml HNO_3, 10 ml acetic acid, 15 ml HCl, 2-5 drops glycerol	Use fresh, ***do not store***; use by immersion or swabbing; 5-60 seconds; discard when solution turns orange
Nickel Superalloy Microetchant) (86)	For nickel-base superalloys	30 ml lactic acid, 20 ml HCl, 10 ml HNO_3	Etch seconds to minutes

11.6.2 Titanium and Titanium Alloys (Conventional Polishing)

Description:
Titanium alloys are very useful because they have a good strength-to-weight ratio. This makes them ideal for use in areas ranging from aerospace to sports equipment.

Preparation Challenge:

Metallographic preparation of titanium and titanium alloys is fairly straight forward by conventional metallographic techniques.

SECTIONING

MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING

Phenolic, epoxy or diallyl phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
240 (P220)grit SiC paper 360 (P500) grit SiC paper 600 (P1200) grit SiC paper 800 (P2400) grit SiC paper 1200 (P4000) grit SiC paper	Water	5-10 lbs	200/200 rpm	Until plane 1 minute 1 minute 1 minute 1 minute
6 micron DIAMAT diamond on TEXPAN polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	3 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on ATLANTIS polishing pad		5-10 lbs	100/100 rpm	1 minute

Figure 11-47 Ti_6Al_4V Titanium Alloy, 400X (DIC), Etchant Kroll’s Reagent.

Figure 11-48 Cast Titanium, 200X (BF), Etchant Kroll’s Reagent.

Figure 11-49 Wrought Titanium, 200X (BF), Etchant Ammonium Bifluoride.

Figure 11-50 Alpha-rich Case Wrought Titanium, 200X (BF).

11.6.3 Titanium Alloy - Attack Polishing

(contributed by Jim Hallquist Medtronic Inc.)

Description:
Titanium is a very wear and corrosion resistant material, thus it has found some useful applications in the medical industry ranging from hip implants to pacemaker casings.

Preparation Challenge:
Specimen preparation of titanium is relatively straight forward; however, attack polishing has been sucessfully used to enhance microstructural features.

SECTIONING
MAXCUT abrasive blade (MAX-C or MAX-I series)

MOUNTING
Phenolic, epoxy or diallyl phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
180 grit SiC paper 240 (P220) grit SiC paper 320 (P360) grit SiC paper	water	5-10 lbs	200/200 rpm	Until plane 1 minute 1 minute
9 micron DIAMAT diamond on TEXPAN polishing pad		5-10 lbs	200/200 rpm	10 minutes
100 ml SIAMAT colloidal silica with 20 ml H_2O_2 and 10 ml Kroll's reagent		5-10 lbs	100/100 rpm	10 minutes

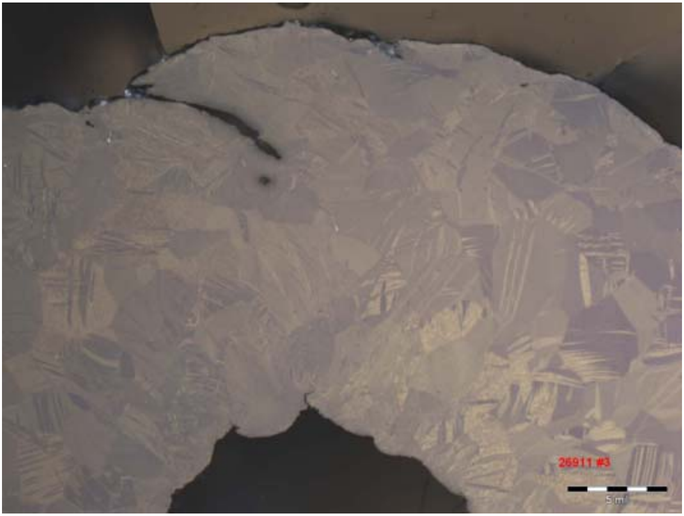


Figure 11-51 Titanium alloy, 500X, as attack polished with H_2O_2 /Kroll's reagent (photo courtesy of Medtronic Inc).

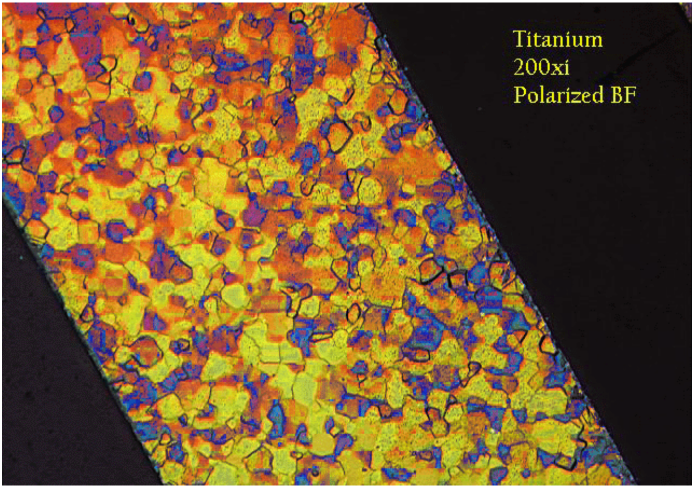


Figure 11-52 Titanium alloy, 200X (polarized light), as attack polished with H_2O_2 /Kroll's reagent (photo courtesy of Medtronic Inc).

Table LI. Selected Microetchants for Titanium Alloys

Name/Type	Application	Composition	Conditions
Keller's reagent (Microetchant) (89)	Keller's reagent; for etching Ti alloys	1 ml HF, 3 ml HCl, 5 ml HNO_3, 190 ml DI water	Immerse sample 3-10 seconds
Kroll's reagent (Microetchant) (90)	Kroll's reagent for Ti alloys; very good etch	1-3 ml HF, 2-6 ml HNO_3, 100 ml DI water	Swab sample 3-10 seconds or immerse sample 10-30 seconds
R-etch (Microetchant) (89)	R-etch, for Ti-Al and Ti-Al-Zr alloys	25 ml HF, 18.5 gm benzalkonium chloride, 35 ml methanol, 40 ml glycerin	Swab sample up to 20 seconds, or immerse sample up to 90 seconds with agitation
Smith (Microetchant) (91)	For etching beta-Ti alloys heat-treated below beta transus; reveals grain boundaries	60 ml propionic acid, 20 ml HNO_3, 10 ml HF	Swab sample
Gallaugher (Microetchant) (91)	For etching Ti-Cu alloys	60 ml lactic acid, 20 ml HNO_3, 10 ml HF, 10 ml DI water	Immerse sample 3-10 seconds

Table LI (conti.). Selected Microetchants for Titanium Alloys

Name/Type	Application	Composition	Conditions
Gurevich (Microetchant) (91)	For Ti alloys; reveals beta phase	10 ml HF, 10 ml HNO_3, 10 ml H_2SO_4, 1 gm succinic acid	Heat to 40-50°C, immerse sample 20-180 seconds
Ogden and Holden (Microetchant) (91)	For etching Ti alloys; differentiates phases in alpha-beta alloys	1-4 ml HCl, 1 ml H_2SO_4, 95-98 ml DI water	Use boiling, 3-10 minutes
Weck's tint etch (Microetchant) (91)	Weck's tint etch for Ti and alloys; alpha grains and twins colored according to orientation	5 gm ammonium bifluroide, 100 ml DI water	Immerse sample at 20°C for a few seconds
Beraha's tint etch (Microetchant) (91)	For as-cast Ti alloys; colors alpha matrix blue or green, TiC yellow or dark brown	2-3 gm sodium molybdate, 5 ml HCl, 1-2 gm ammonium bifluoride, 100 ml DI water	Immerse sample at 20°C until surface is colored
Stain removal (Microetchant) (92)	Removes etchant stains for most titanium and titanium alloys	1 ml HF, 2 ml HNO_3, 50 ml H_2O_2, 47 ml DI water	Immerse sample for several seconds

Table LII. Selected Macroetchants for Titanium Alloys

Name/Type	Application	Composition	Conditions
Titanium (Macroetchant) (93)	Macroetchant for Ti and Ti-base alloys; Ti-Al-Mo alloys	50 ml DI water, 40 ml HNO_3, 10 ml HF (40%)	Etch 5-8 minutes at 60-80°C (content of HF may be decreased)
Titanium (Macroetchant) (94)	Reveals flow lines and defects	15 ml HNO_3, 10 ml HF, 75 ml DI water	Etch for about 2 minutes
Titanium (Macroetchant) (95)	General purpose etch for alpha + beta alloys	50 ml HCl, 50 ml DI water	Etch for about 2 minutes
Titanium (Macroetchant) (93)	Macroetchant for Ti	30 ml DI water, 10 ml HF (40%), 60 ml H_2O_2 (30%)	Etch by swabbing until desired contrast is obtained
Titanium welds (Macroetchant) (96)	Macroetchant for welded seams	200 ml DI water, 10 ml HF (40%), 10 gm $Fe(NO_3)_3$-$9H_2O$	Etch seconds to minutes at 50-60°C

11.7 CLASS 7 - THERMAL SPRAY MATERIALS

Examples include powder spray coating, ceramic coating, intermetallic coatings.

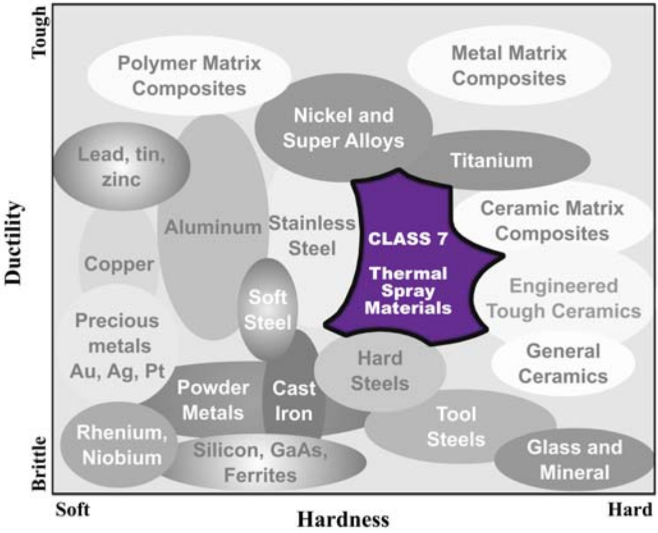


Figure 11-53 Thermal spray materials.

Table LIII. Preparation Guidelines for Class 7 Materials

Preparation Step	Basic Recommendation
Rough Grinding	Diamond on CERMESH metal mesh cloth
Rough Polishing	Composite diamond disks
Final Polishing	Polycrystalline alumina on a napped polishing pad

Thermal Spray Coatings.

Thermal spraying techniques are coating processes in which melted (or heated) materials are sprayed onto a surface. Thermal spraying can provide thick coatings (approximate thickness range is 20 microns to several mm), over a large area. Coating materials available for thermal spraying include metals, alloys, ceramics, plastics and composites. They are fed in powder or wire form, heated to a molten or semi-molten state and accelerated towards the substrates in the form of micron-size particles. Combustion or electrical arc discharge is usually used as the source of energy for thermal spraying. The coating quality is usually assessed by measuring its porosity, oxide content, macro and microhardness, bond strength and surface roughness.

Metallographic specimen preparation of thermal spray coatings is affected by the specimen’s composition and deposition conditions. Microstructural features of interest include: porosity, flow, thickness and inclusions. Proper metallurgical preparation takes into account that the microstructure may be porous and perhaps somewhat brittle as a result of inadequate processing.

The first step is to minimize damage with proper cutting.

The recommended initial grinding step is with either 15 or 30 micron diamond suspensions on a metal mesh cloth. This step is very important and must minimize damage or grain pull-out of a poorly deposited material. The next rough polishing stage must eliminate any remaining surface and subsurface deformation to the specimen surface. Polycrystalline diamond suspensions used in conjunction with a low-napped polishing pad result in minimal edge rounding and relief for the rough polishing stage. For the final polish, 0.05 micron Nanometer polycrystalline alumina provides an excellent surface finish.

11.7.1 Thermal Spray Coatings

Preparation Challenge:
Metallographic preparation of thermal spray coatings can be tricky; however, minimizing damage and understanding the chemical and mechanical properties of the coating and substrate will greatly improve successful specimen preparation.

SECTIONING
Precision wafering with either diamond or CBN blades

MOUNTING
Castable epoxies or acrylics

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Spedd (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth	DIALUBE Extender	5-10 lbs	100/100 rpm	Until plane
9 micron DIAMAT diamond on SIRIUS composite disk		5-10 lbs	100/100 rpm	3 minutes
3 micron DIAMAT diamond on ORION composite disk		5-10 lbs	100/100 rpm	3 minutes
1 micron DIAMAT diamond on ATLANTIS polishing pad	DIALUBE Purple Extender	5-10 lbs	100/100 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

Figure 11-54 Nickel-chrome thermal spray coating, 1000X (B.F.), as polished.

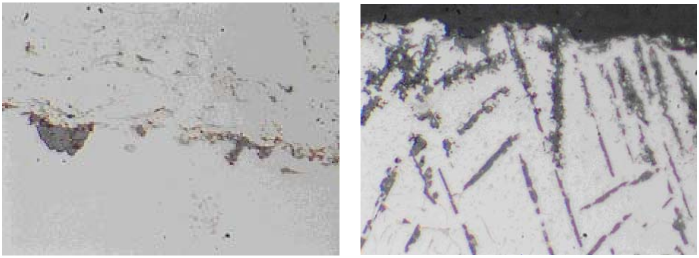


Figure 11-55 Zirconia coating (left), Superalloy (right) (B.F.), as polished.

11.8 CLASS 8 - HARDENED STEELS

Examples include case hardened steels, tool steels, and through-hardened steels.

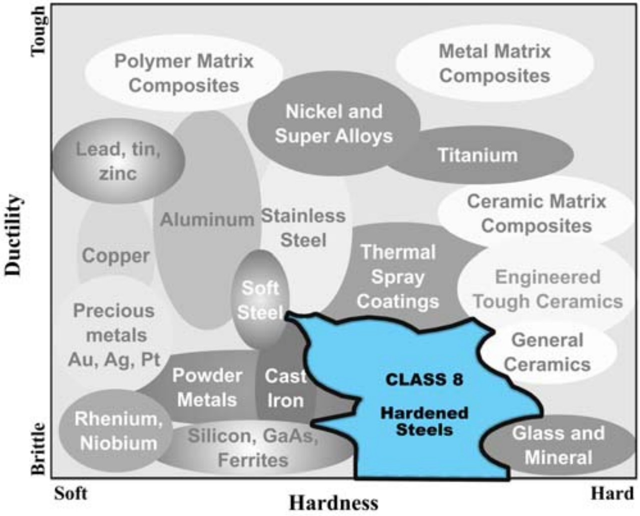


Figure 11-56 Class 8 - hardened steels.

Table LIV. Preparation Guidelines for Class 8 Materials

Preparation Step	Basic Recommendation
Rough Grinding	120 micron diamond disk
Rough Polishing	Compostie grinding disks
Final Polishing	Polycrystalline alumina on napped polishing pad

11.8.1 Tool Steels

Description:
Iron and steels play an important role in the world of structural and mechanical metals. Steel, in particular, is very useful because its hardness, wearability and toughness can be altered significantly by heat treating and annealing processes. Tool steels have high hardness (Rc>60) and generally contain alloying metals such as vanadium, molybdenum and manganese.

SECTIONING
MAXCUT abrasive blade (MAX-E or MAX-I series)

MOUNTING
Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
120 micron diamond grinding disk	Water	5-10 lbs	200/200 rpm	Until plane
9 micron DIAMAT diamond on SIRIUS composite disk	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	3 minutes
3 micron DIAMAT diamond on ORION composite disk	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	3 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

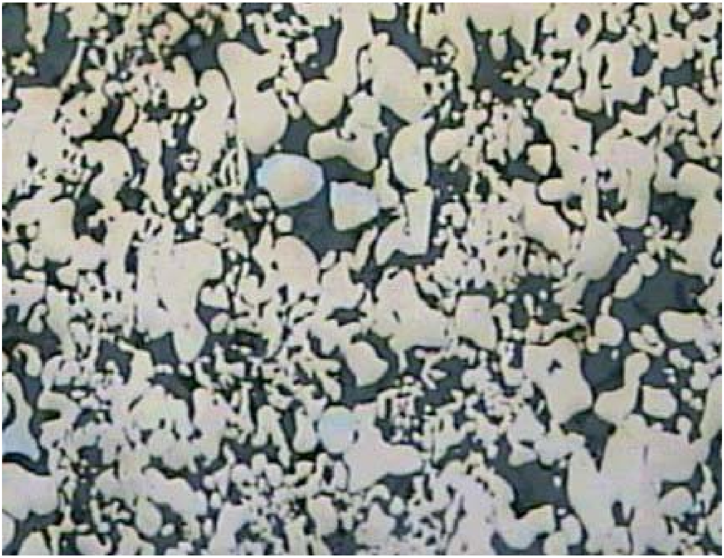


Figure 10-57 44 MnV F6 steel, 500X (DIC), Etchant 3 seconds Nital.

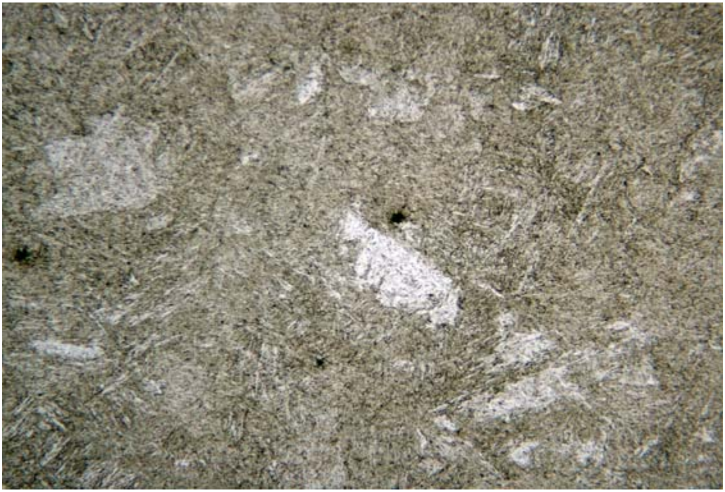


Figure 11-58 High alloy tool steel, 200X (BF), Etchant Picral.

11.8.2 Nitrided Steel

Description:
Nitriding is a heat treating process used for producing a very hard case (Vickers 1100) and is typically used to produce hard, strong, tough steels. The process involves heating the steel to 500-540°C (930-1000°F) in an atmosphere of ammonia gas for about 50 hours. No further quenching or heat treatment is required. The case depth is about 0.4 mm. Nitrided surfaces can also improve corrosion resistance.

SECTIONING
MAXCUT abrasive blade (MAX-E or MAX-I series)

MOUNTING
Epoxy or Diallyl Phthalate compression mounting resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
120 micron diamond grinding disk	Water	5-10 lbs	200/200 rpm	Until plane
9 micron DIAMAT diamond on SIRIUS composite disk	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	3 minutes
3 micron DIAMAT diamond on ORION composite disk	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	3 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

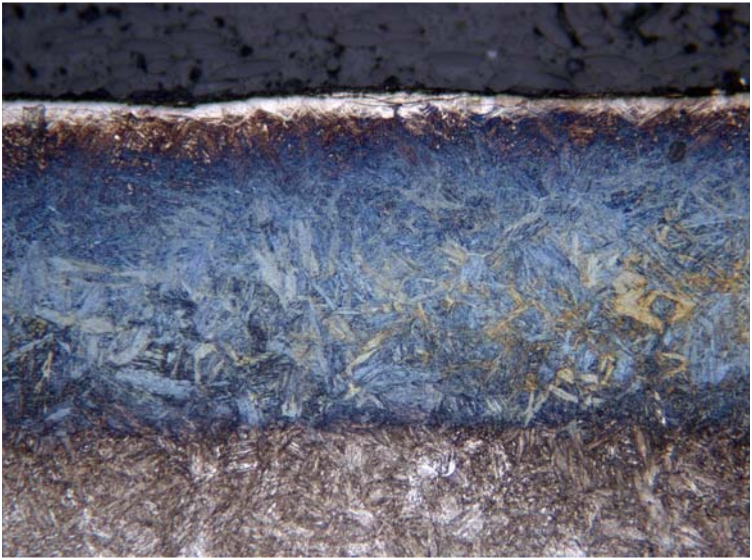


Figure 10-59 Nitrided steel, 200X (BF), Etchant Picral

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Table LV. Selected Etchants for Tool Steels

Name/Type	Application	Composition	Conditions
Nital (Microetchant) (86)	Reveals ferrite grain boundaries and ferrite-carbide interfaces in annealed sample; preferred etchant for martensite; reveals prior-austenite grain boundaries in as-quenched and lightly tempered high alloy steels	1-10 ml HNO_3, 90-99 ml methanol or ethanol	Most commonly used etchant; do not store solutions with more than 3% HNO_3 in ethanol; use by immersion
Vilella's reagent (Microetchant) (86)	Recommended for annealed structures or those containing pearlite or bainite; does not reveal ferrite grain boundaries in annealed specimens	1 gm picric acid, 5 ml HCl, 100 ml ethanol	Use by immersion
Picral (Microetchant) (86)	Recommended for annealed structures or those containing pearlite or bainite; does not reveal ferrite grain boundaries in annealed specimens	4 gm picric acid, 100 ml ethanol	Use by immersion; 10-20 drops of zephiran chloride increases response
Superpicral (Microetchant) (86)	Recommended for annealed structures or those containing pearlite or bainite; does not reveal ferrite grain boundaries in annealed specimens	10 gm picric acid, 100 ml ethanol	Must be heated to dissolve picric acid; use by immersion, up to 1 minute or more; a few more drops of HCl may be added to increase etch rate

Table LV (conti.). Selected Etchants for Tool Steels

Name/Type	Application	Composition	Conditions
Vilella's reagent (Microetchant) (96)	Recommended for annealed structures or those containing pearlite or bainite; does not reveal ferrite grain boundaries in annealed specimens	1 gm picric acid, 5 ml HCl, 100 ml ethanol	Use by immersion
Murakami's reagent (Microetchant) (96)	Cold-darkens chromium carbides and tungstides, cementite not attacked. Hot etchant attacks cementite	10 gm $K_3Fe(CN)_6$, 10 gm KOH or 7 gm NaOH, 100 ml DI water	Immerse etch with fresh solution, hot or cold, up to 10 minutes
Greosbeck's reagent (Microetchant) (96)	Fe_2MoC and M_6C outlined and colored (blue and brown, respectively), Mo_2C colored brown, $(Fe,Cr)_{23}C_6$ attacked but $(Fe,Mo)_{23}C_6$ not attacked	4 gm $KMnO_4$, 4 gm NaOH, 100 ml DI water	Immerse at 20°C
Tool Steels (Microetchant) (96)	Fe_2MoC, Mo_2C and M_6C outlined (later also colored)	10 ml H_2O_2 (30%), 20 ml 10% aqueous NaOH	Immerse 10 seconds at 20°C
Tool Steels (Microetchant) (96)	Mo_2C and M_7C_3 attacked, M_6C outlined and colored brown	4 gm NaOH, 100 ml saturated aqueous $KMnO_4$	Immerse at 20°C

11.9 CLASS 9 - METAL MATRIX COMPOSITES

Examples include ceramic fibers in metal matrix and ceramic particles in metal matrix.

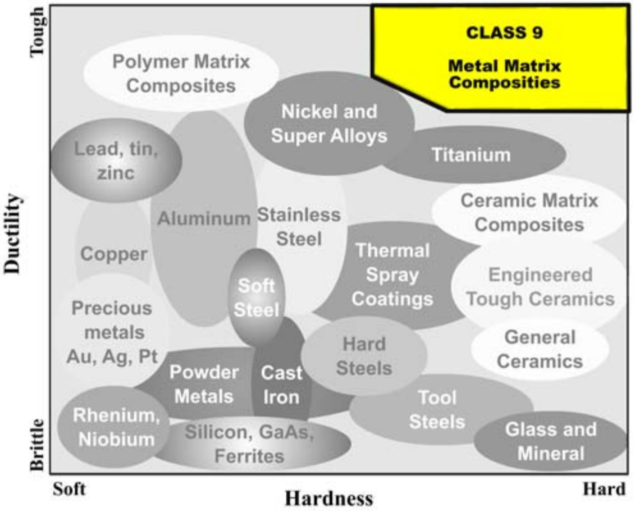

Figure 11-60 Class 9 - Metal Matrix composites.

Table LVI. Preparation Guidelines for Class 9 Materials

Preparation Step	Basic Recommendation
Rough Grinding	Diamond on CERMESH metal mesh cloth
Rough Polishing	Diamond and colloidal silica on woven polishing pads
Final Polishing	Colloidal silica on low-napped urethane polishing pads

11.9.1 Metal Matrix Composites

Description:
Metal matrix composite applications have been found in high performance sporting goods and high performance military applications. Their primary application combines the hardness and energy absorbing characteristics of the ceramic with the bonding and ductility characteristics of the metal.

Preparation Challenge:
Hard particles in a metal matrix can be difficult to microstructurally prepare because of particle pull-out, as well as, excessive polishing relief between the hard particles and the softer matrix.

SECTIONING
MAXCUT abrasive blade (MAX-I or Diamond cut-off blade)

MOUNTING
Castable mounting with epoxies and acrylics

ETCHING
Use the recommended etchant for the metal matrix component.

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
9 micron DIAMAT diamond on SIRIUS composite disk		5-10 lbs	200/200 rpm	3 minutes
3 micron DIAMAT diamond on ATLANTIS polishing pad	SIAMAT colloidal silica	5-10 lbs	200/200 rpm	2 minutes
0.05 micron Nanometer alumina on TRICOTE polishing pad		5-10 lbs	100/100 rpm	1 minute

Figure 11-61a ZrB_2 particles in a Titanium Matrix, 1000X (DIC), as polished.

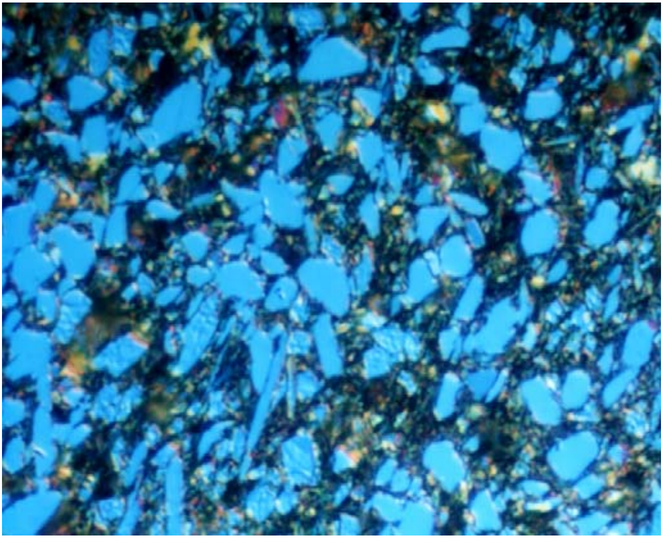

Figure 11-61b ZrB_2 particles in a Titanium Matrix, 1000X (DIC), Etchant Kroll’s.

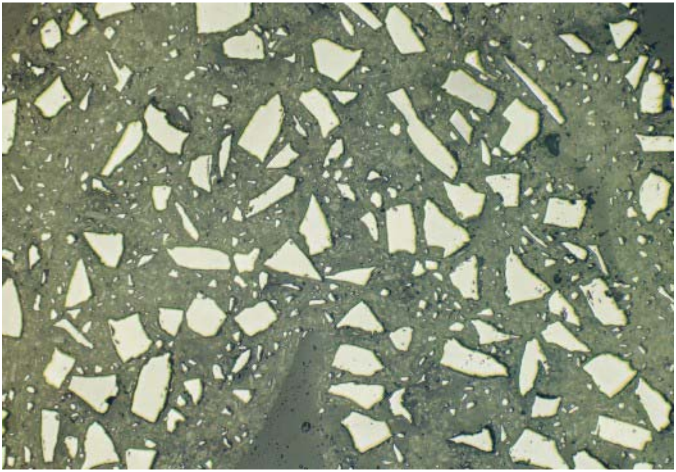

Figure 11-62 SiC particles in an Aluminum Matrix, 400X (DIC), as polished.

11.9.2 Metal Matrix Composite - Metal Injection Molding (MIM)

(contributed by Jim Hallquist Medtronic Inc.)

Description:
Metal Injection Molding (MIM) is a manufacturing process which combines the technology of powder metallurgy and plastic injection molding in order to produce complex parts. When combined with ceramic particles, these parts also become very hard and wear resistant. Metal injection molded parts have very high densities (up to 98% density of wrought metal) and has a broad range of applications, including medical, dental, firearms, aerospace and automotive components.

Preparation Challenge:
Hard particles in a metal matrix can be difficult to microstructurally prepare because of particle pull-out, as well as excessive polishing relief between the hard particles and the softer matrix.

MOUNTING
Castable mounting with epoxies and acrylics

ETCHING
Use the recommended etchant for the metal matrix component.

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
180 grit SiC paper 240 (P220) grit SiC paper 320 (P360) grit SiC paper	water	5-10 lbs	200/200 rpm	Until plane 1 minute 1 minute
9 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	5-10 lbs	200/200 rpm	10 minutes
100 ml SIAMAT colloidal silica with 20 ml H_2O_2 and 10 ml Kroll's Reagent		5-10 lbs	100/100 rpm	10 minutes

Figure 11-63a TiC particles in a Ti-6Al-4V Matrix, 500X (polarized light), Attack polish with H_2O_2/ Kroll's reagent (photo courtesy of Medtronic).

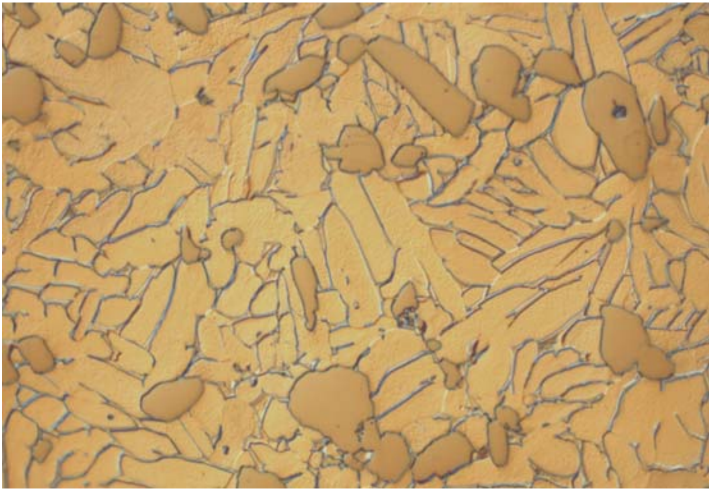

Figure 11-63b TiC particles in a Ti-6Al-4V Matrix, 1000X (DIC), Etchant, Kroll's reagent (photo courtesy of Medtronic).

11.10 CLASS 10 - ENGINEERED CERAMICS

Examples include silicon nitride, zirconia, SiSiC, silicon carbide, boron carbide, alumina, mullite, and ceramic matrix composites (CMC's).

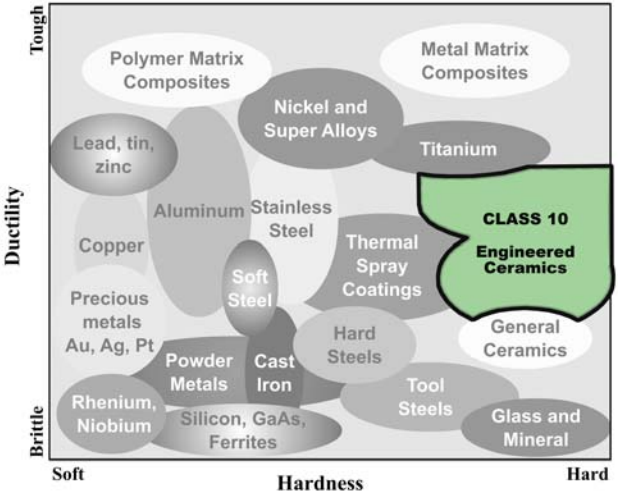

Figure 11-64 Class 10 - Engineered Ceramics.

Table LVII. Preparation Guidelines for Class 10 Materials

Preparation Step	Basic Recommendation
Rough Grinding	Diamond on CERMESH metal mesh cloth
Rough Polishing	Diamond and colloidal silica on woven polishing pads
Final Polishing	Colloidal silica on low-napped urethane polishing pads

11.10.1 Engineered Ceramics - ZrO_2, SiALON, Si_3N_4

Description:
Tough engineering ceramics, such as silicon nitride and zirconia, continue to find industrial applications where strength or wear resistance is required at relatively high operating temperatures.

Preparation Challenge:
Engineering ceramics are very hard; therefore, specimen preparation procedures generally use a CMP (chemical mechanical polishing) component to remove induced microstructural damage.

SECTIONING
Diamond wafering blades - medium grit/low concentration

MOUNTING
Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
SIAMAT colloidal silica on TEXPAN polishing pad		10 lbs	100/100 rpm	5 minutes

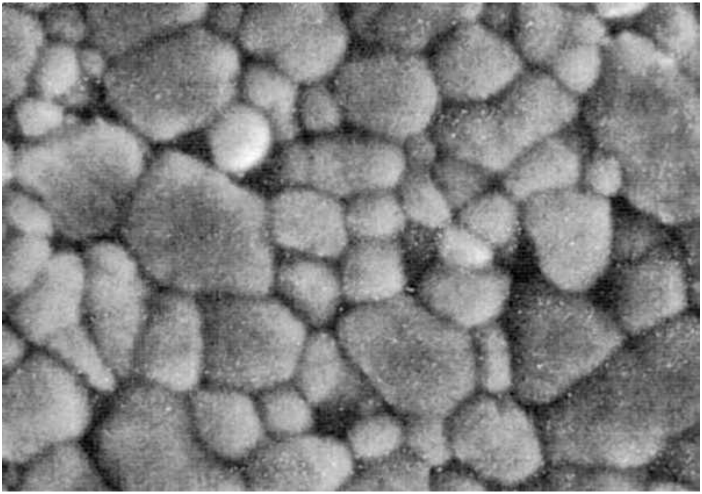

Figure 11-65 Yittria stabilized Zirconia, 15,000X, Thermally etched.

Figure 11-66 SiAlON ceramic, 20,000X, etched in molten KOH.

11.10.2 Engineered Ceramics - Alumina

Description:
Alumina ceramics are some of the most commonly used ceramics. Applications range from dielectric substrate materials to medical implants.

Preparation Challenge:
Alumina is very hard; therefore, specimen preparation procedures generally use a CMP (chemical mechanical polishing) component to remove induced microstructural damage.

SECTIONING
Diamond wafering blades - medium grit/low concentration

MOUNTING
Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
SIAMAT colloidal silica on TEXPAN polishing pad		10 lbs	100/100 rpm	5 minutes

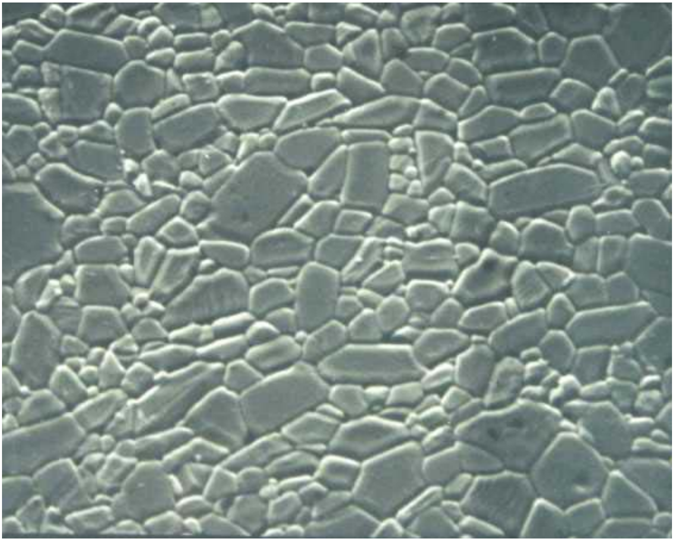

Figure 11-67 Alumina, 5,000X, Thermally etched.

11.10.3 Engineered Ceramics - ALON

Description:
Aluminium oxynitride (AlON) is a ceramic composed of aluminum, oxygen and nitrogen. It is a transparent ceramic that is harder than glass.

Preparation Challenge:
AlON is very hard and, therefore, specimen preparation procedures generally require a CMP (chemical mechanical polishing) component to remove induced microstructural damage. When examining the surface, it is recommended that a sputter coating be used to increase the reflectivity.

SECTIONING

Diamond wafering blades - medium grit/low concentration

MOUNTING

Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
SIAMAT colloidal silica on TEXPAN polishing pad		10 lbs	100/100 rpm	5 minutes

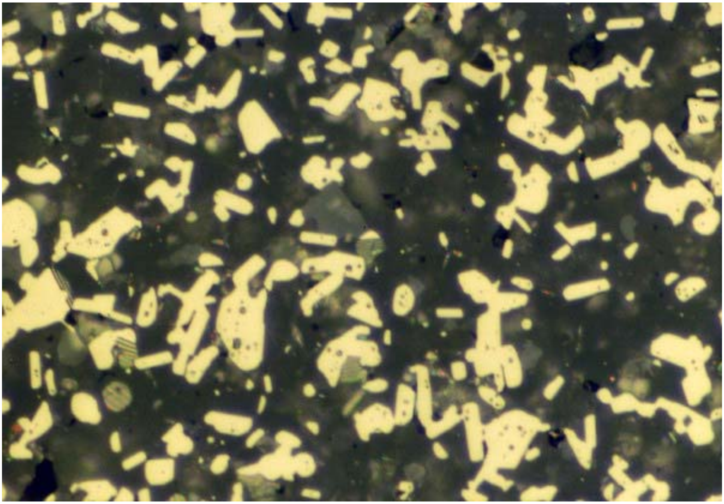

Figure 11-68a AlON ceramic, 1000X (BF), as polished.

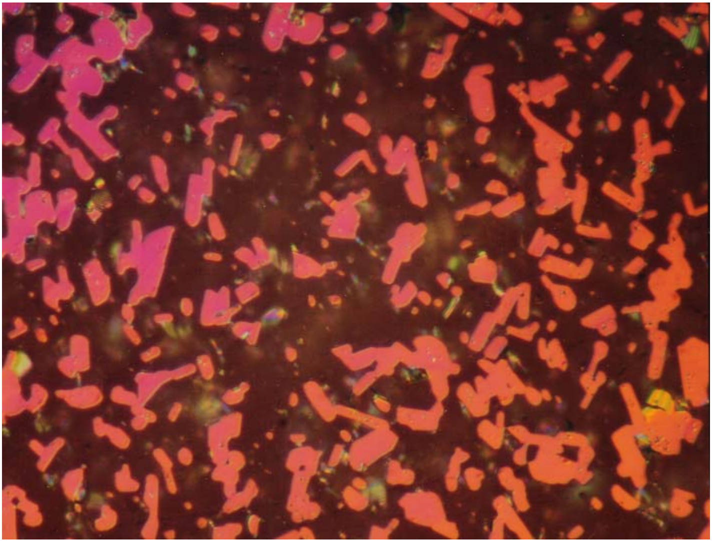

Figure 11-68b AlON ceramic, 1000X (DIC), as polished.

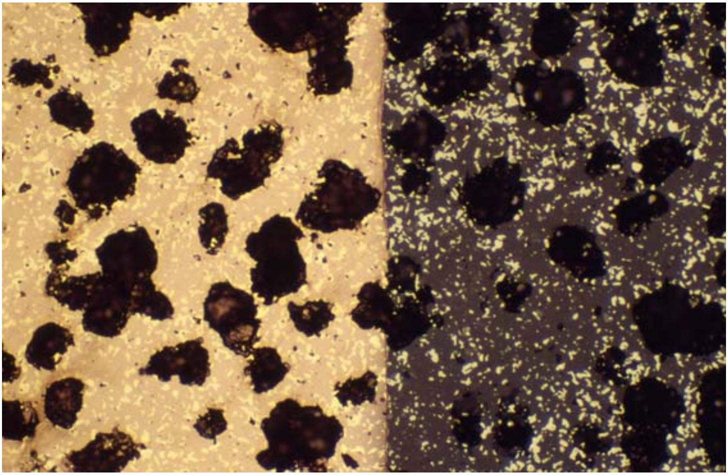

Figure 11-68c AlON ceramic, 200X (BF), as polished, half sputter coated for increased reflectivity and contrast (left).

11.10.4 Engineered Ceramics - SiSiC

Description:
SiSiC is a silicon / silicon carbide ceramic. SiSiC is also called a machinable ceramic since its silicon phase makes it a relatively easy material to machine.

Preparation Challenge:
SiSiC ceramics machine very easily because the silicon phase makes them very brittle. In order to get rid of the grinding damage, SiSiC polishing should include a CMP (chemical mechanical polish) to remove the previously induced microstructural damage.

SECTIONING
Diamond wafering blades - medium grit/low concentration

MOUNTING
Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
SIAMAT colloidal silica on TEXPAN polishing pad		10 lbs	100/100 rpm	5 minutes

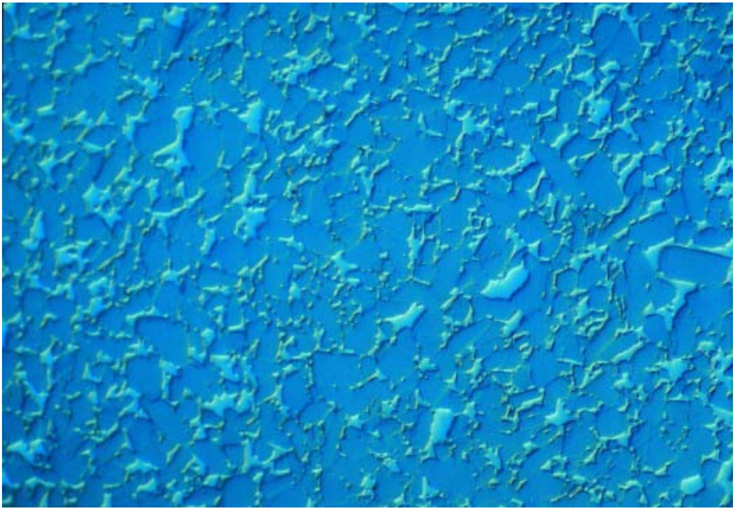

Figure 11-69 SiSiC, 200X (DIC), as polished.

Table LVIII. Selected Etchants for Borides

Name/Type	Application	Composition	Conditions
Borides - Chemical (Microetchant) (97)	Etchant for ZrB_2	10 ml DI water, 10 ml HF (40%), 10 ml HNO_3	Etch seconds to minutes
Borides - Chemical (Microetchant) (97)	Etchant for ZrB_2 and TiB_2	10 ml glycerol, 10 ml HNO_3, 10 ml HF (40%)	Etchant for ZrB_2 and TiB_2
Borides - Chemical (Microetchant) (97)	Etchant for ZrB_2 and TiB_2	30 ml lactic acid, 10 ml HNO_3, 10 ml HF (40%)	Etch seconds to minutes
Borides - Chemical (Microetchant) (97)	Etchant for CrB_2 and MoB_2	10 ml HCl, 10 ml HNO_3	Etch for 1-5 minutes at 40°C, vapor etching.
Borides - Chemical (Microetchant) (97)	Etchant for TiB_2	6 ml HCl, 2 ml HNO_3	Etch for 15 seconds
Borides - Chemical (Microetchant) (97)	Etchant for HfB_2-NbB_2 mixtures	6 ml HCl, 2 ml HNO_3, 1 ml HF (40%)	Etch for 9 seconds at 30-40°C

Table LIX. Selected Etchants for Carbides

Name/Type	Application	Composition	Conditions
Carbides - Thermal (Microetchant) (97)	Thermal etch SiC	Vacuum	2200°F (1200°C) in vacuum
Carbides - Molten Salt (Microetchant) (97)	Molten salt etch for SiC	Sodium tetraborate	Immerse sample for a few minutes
Carbides - Molten Salt (Microetchant) (97)	Molten salt etch for SiC	Sodium or potassium bicarbonate	Immerse sample for approximately 10 minutes
Carbides - Chemical (Microetchant) (97)	Chemical etching SiSiC	60 ml DI water, 3 gm NaOH, 30 gm potassium ferricyanide	Etch for 8-15 minutes; use boiling
Carbides - Plasma (Microetchant) (97)	Plasma etching SiC with 5-15% oxide solutions	CF_4 gas and O_2 gas (1:1)	Plasma etch for 3-3.5 minutes at 60-80 W

Table LX. Selected Etchants for Nitrides

Name/Type	Application	Composition	Conditions
Nitride - Chemical (Microetchant) (97)	For Si_3N_4	Concentrated HF	Immerse sample up to 15 minutes
Nitride - Chemical (Microetchant) (97)	For Si_3N_4	Concentrated H_3PO_4	Use boiling up to 15 minutes
Nitride -Molten Salt (Microetchant) 97)	For Si_3N_4	95.4 gm potassium carbonate, 12 gm sodium fluoride	Immerse sample 1-4 minutes
Nitride - Thermal (Microetchant) (97)	For Si_3N_4	2912°F (1600°C) in dry N_2	Use high purity N_2 for approximately 5 hours
Greskovich (Microetchant) (97)	For beta - silicon nitride	4 parts KOH, 4 parts NaOH, 1 part LiOH (By weight)	Use at 392°F (200°C) for 20 minutes
Nitride - Chemical (Microetchant) (97)	For etching TiN	10 ml DI water, 10 ml glacial acetic acid, 10 ml HNO3 acid	Etch for 40-60 minutes at 100°C

Table LX (conti.). Selected Etchants for Nitrides

Name/Type	Application	Composition	Conditions
Nitride - Plasma (Microetchant) (97)	For Si_3N_4	CF_4 gas and O_2 gas (2:1)	Etch for 3-5 minutes at 60-80 W
Nitride - Chemical (Microetchant) (97)	For Si_3N_4	12 gm potassium hydroxide, 12 gm sodium hydroxide, 3 gm lithium hydroxide	Etch for 20 minutes at 200°C
Nitride -Molten Salt (Microetchant) (97)	For Si_3N_4	Sodium hydroxide	Etch for 20 seconds to 3 minutes at 300-350°C, 2-3 samples per melt
Nitride - Chemical (Microetchant) (97)	For etching HfB_2 with TaB_2	Nitric acid	Etch for 1-2 minutes at 40-50°C
Nitride - Chemical (Microetchant) (97)	For etching ZrB_2	Nitric acid	for 8-10 minutes at 40-50°C
Nitride - Chemical (Microetchant) (97)	For etching TiB_2	10 ml DI water, 1 ml H_2SO_4	Etch for 15 seconds

Table LXI. Selected Etchants for Oxides

Name/Type	Application	Composition	Conditions
Oxide - Thermal (Microetchant) (97)	For etching Al_2O_3 with additives: CaO, MgO, SiO_2, Na_2O; grain boundary and glass phase etchant. glass phase melts, wets the surface, and solidifies again	Air	Etch 15 minutes at 1400°C
Oxide - Molten Salts (Microetchant) (97)	For grain boundary etching of Al_2O_3. Grain boundaries are uniformly revealed; small grains remain intact	Vanadium pentoxide	Etch for 1 minute at 900°C; use platinum crucible; preheat sample also to 900°C; rinse in diluted aqueous hydrochloric acid 5-10 minutes
Oxide - Chemical (Microetchant) (97)	For etching Al_2O_3; grain boundaries are not uniformly revealed; spinel and glass phases are dissolved, etchant can only be used for 2-3 samples	H_3PO_4	Etch for 5 seconds to 3 minutes at 250°C
Oxide - Chemical (Microetchant) (97)	For etching mullite (Al_2O_3-SiO_2), BeO, MgO, $Ca_2Zr_{(1-x)}O_y$	HF (40%)	Cool hydrofluoric acid to 0°C; swab with a cotton swab

Table LXI (conti.) Selected Etchants for Oxides

Name/Type	Application	Composition	Conditions
Oxide - Chemical (Microetchant) (97)	For etching $BaTiO_3$	75 ml DI water, 15 ml HCl, 10 ml HF	Etch 4 minutes.
Oxide - Chemical (Microetchant) (97)	For etching NiO	75 ml glacial acetic acid, 25 ml HNO_3, 1.5 ml HF	Etch 5 seconds to 5 minutes; use boiling
Oxide - Thermal (Microetchant) (97)	For etching ZrO_2-TZP (Y_2O_3, RE oxide, TiO_2), ZrO_2-PSZ (Y_2O_3, MgO, CaO), ZrO_2-CSZ (ZrO_2, MgO, CaO), ZrO_2-ZTC	Air	Etch minutes to 1 hour at 1300-1400°C
Oxide - Ion Beam (Microetchant) (97)	Ion beam etch for ZrO_2 with MgO, CaO	Ion Beam	Etch 30 minutes at 1-7 kV, 60-400 mA in an Ar/Kr atmosphere, beam angle: 5-60 degrees

11.10.5 Ceramic Matrix Composites (CMC's)

Description:
Ceramic Matrix Composites (CMC's) provide high temperature stability and enhance the toughness of ceramic materials.

Preparation Challenge:
The key to proper specimen preparation of hard/brittle ceramic matrix composite materials is to first section the sample with the appropriate diamond wafering blade, rough grind with as fine a semi-fixed abrasive as possible, followed by CMP polishing.

SECTIONING
Diamond wafering blades - medium grit/low concentration

MOUNTING
Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
9 micron DIAMAT diamond on POLYPAD polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	5 minutes
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
SIAMAT colloidal silica on BLACKCHEM 2 polishing pad		10 lbs	100/100 rpm	5 minutes

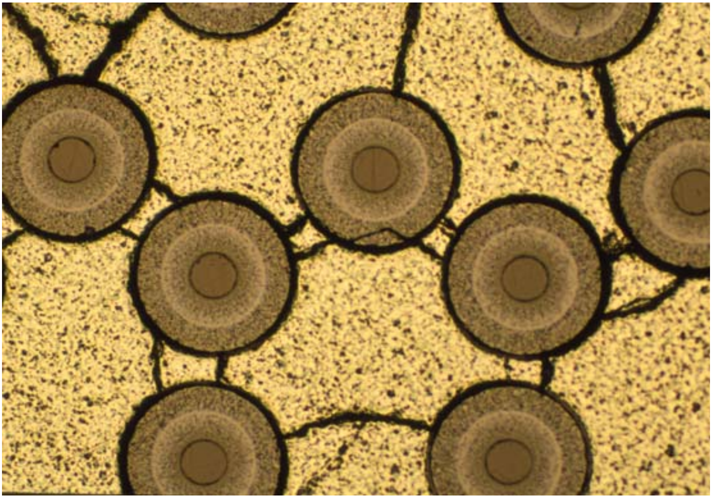

Figure 11-70a SiC fibers in a silicon nitride matrix, 100X (DIC), as polished condition (thermal expansion cracks).

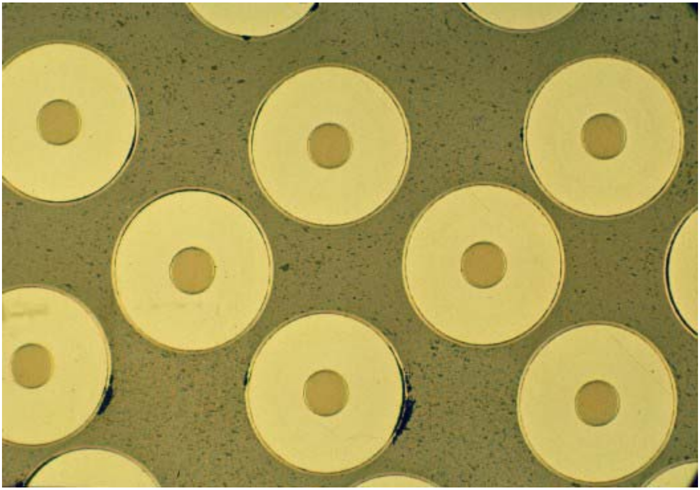

Figure 11-70b SiC fibers in a silicon nitride (5% mullite) matrix, 100X (DIC), as polished condition.

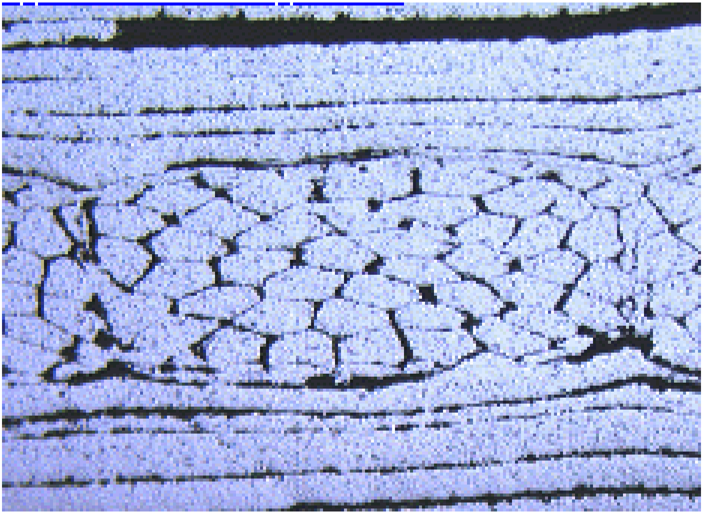

Figure 11-71 Hafnium diboride in a boron nitride matrix, 100X (DIC), as polished.

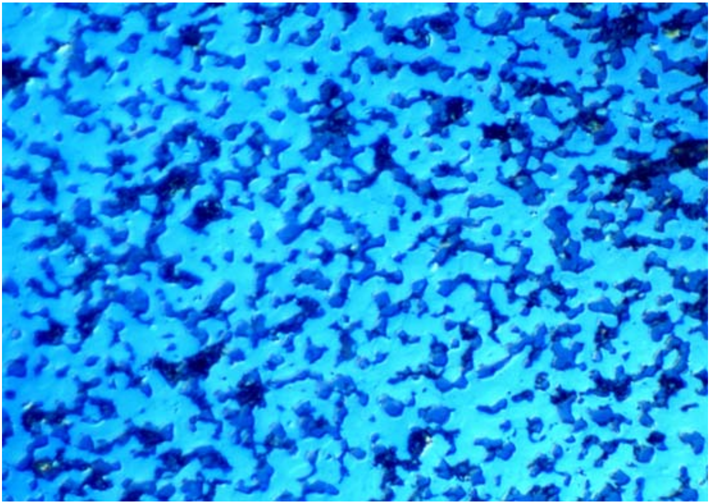

Figure 11-72 20% SiC - 80% ZrB_2 ceramic, 1000X (DIC), as polished.

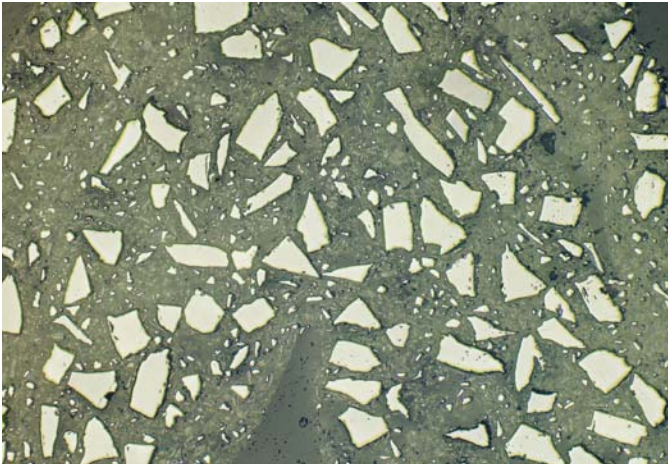

Figure 11-73 SiC particles in a Si_3N_4 matrix, 1000X (DIC).

11.10.06 CERMETS (Tungsten Carbide)

Description:
CERMET's are very wear resistant and tough materials.

Preparation Challenge:
The key to proper specimen preparation of hard / tough CERMET materials such as tungsten carbide is to grind with a semi-fixed abrasive (polycrystalline diamond on a metal mesh cloth), followed by combining diamond abrasive with a CMP (chemical mechanical polishing) abrasive such as SIAMAT colloidal silica.

SECTIONING
Diamond wafering blades - medium grit/low concentration

MOUNTING
Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
9 micron DIAMAT diamond on POLYPAD polishing pad	DIALUBE Purple Extender	5-10 lbs	200/200 rpm	5 minutes
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
SIAMAT colloidal silica on BLACKCHEM 2 polishing pad		10 lbs	100/100 rpm	5 minutes

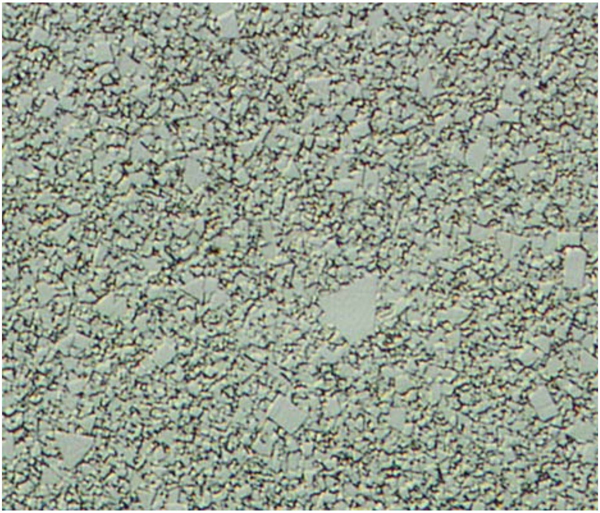

Figure 11-74 WC, Etched with 50% HCl, 50% HNO_3 in a ultrasonic cleaner for 30 minutes, 2000X (DIC).

11.11 CLASS 11 - VERY BRITTLE MATERIALS

Examples include glass, glass-ceramics and minerals.

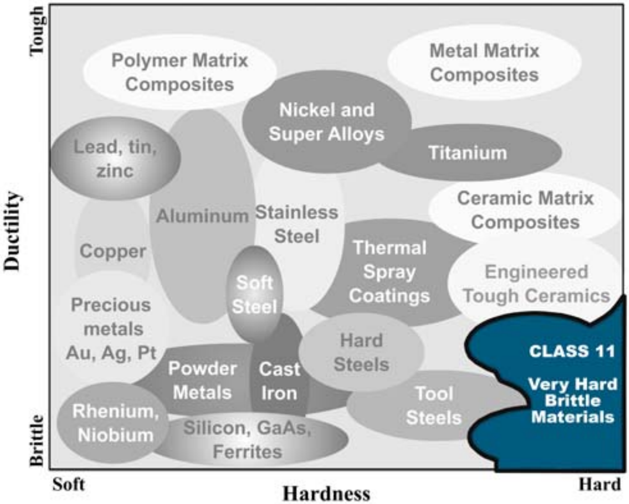

Figure 11-75 Class 11 - Very hard brittle materials.

Table LXII. Preparation Guidelines for Class 11 Materials

Preparation Step	Basic Recommendation
Rough Grinding	Diamond on CERMESH metal mesh cloth
Rough Polishing	Diamond on woven polishing pads
Final Polishing	Colloidal silica on porous urethane polishing pad

11.11.1 Glass and Hard Brittle Noncrystalline Materials (Slag)

Description:
Glasses are typically hard/brittle noncrystalline materials. The composition and properties of these materials can vary significantly.

Preparation Challenge:
Proper microstructural preparation of glass and hard brittle materials requires proper cutting or sectioning with fine grit diamond wafering blades (see figures 11-76a and 11-76b). If the glass is chipped or excessively cracked during sectioning it may be impossible to remove the damage by standard polishing operations..

SECTIONING
Diamond wafering blades - low grit/low concentration

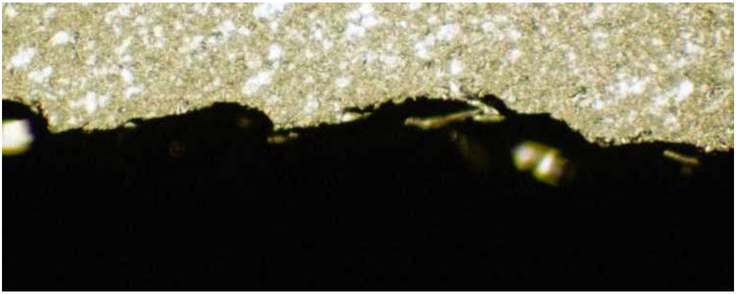

Figure 11-76a Glass sectioned with medium grit diamond blade.

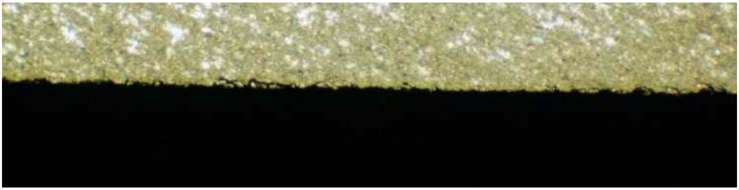

Figure 11-76b Glass sectioned with fine grit diamond blade.

MOUNTING
Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
SIAMAT colloidal silica on BLACKCHEM 2 polishing pad		10 lbs	100/100 rpm	5 minutes

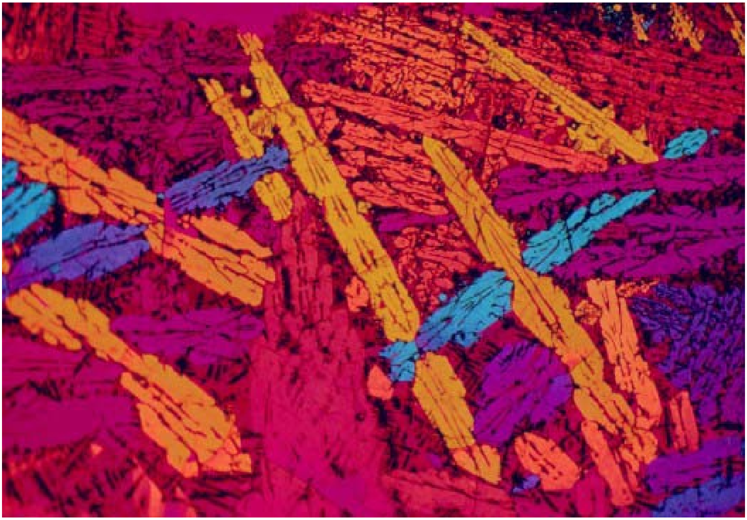

Figure 11-77 Slag, 100X (Polarized light), as polished.

11.11.2 Glass-Ceramics (Alumino-Silicate)

Description:
Glass ceramics are unique materials that have very small crystalline structures. The primary advantage of glass ceramics is that they produce very interesting thermochemical properties because they are impervious to thermal shock.

Preparation Challenge:
Proper microstructural preparation of these materials requires minimizing damage during cutting or sectioning. Therefore, the most important step in the preparation of glass ceramics is sectioning. If the glass ceramic is chipped or excessively cracked during sectioning, it may be impossible to remove this damage. Wafer sectioning with a fine grit diamond wafering blade is essential.

Planar grinding is accomplished with diamond on a metal mesh cloth to minimize cracking and subsurface damage. For rough polishing, the use of a low-napped polishing pad with polycrystalline diamond and colloidal silica eliminates most of the surface and subsurface damage. Final polishing is accomplished with a resilient porous urethane polishing pad such as BLACKCHEM 2 using SIAMAT colloidal silica.

SECTIONING
Diamond wafering blades - low grit/low concentration

MOUNTING
Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	200/200 rpm	Until plane
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	200/200 rpm	5 minutes
SIAMAT colloidal silica on BLACKCHEM 2 polishing pad		10 lbs	100/100 rpm	5 minutes

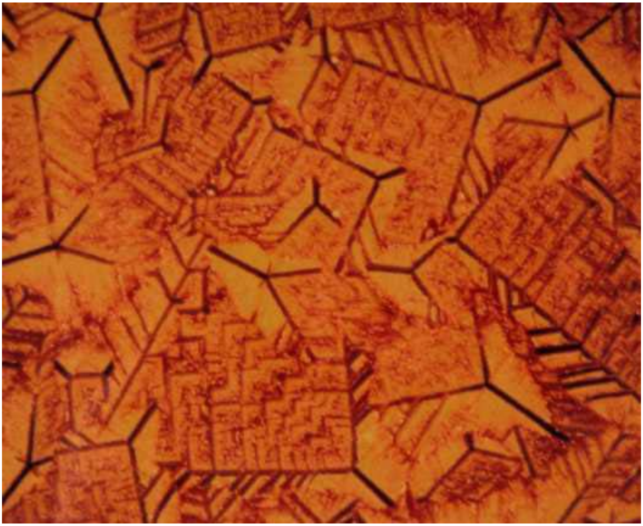

Figure 11-78 Alumino-silicate glass, 400X (Polarized light), as polished.

11.11.3 Mineral Specimens (Mining Concentrates)

Description:
The microstructural analysis of mining concentrates or tailings is useful for determining if the important minerals have been liberated from the gang material through grinding. In the examples shown in figures 11-79a and 11-79b, a concentrate of chalcopyrite and molybdenite are to be separated by a secondary flotation process. For this to be effective, the chalcopyrite and the molybdenite minerals need to be separated. If they are not, an additional or an extended grinding operation may be required.

Preparation Challenge:
Proper microstructural preparation of these materials must maintain the structure of the ore concentrate.

SECTIONING
Not required

MOUNTING
Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
320 (P360) grit SiC paper	Water	5-10 lbs	100/100 rpm	Until plane
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	100/100 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	100/100 rpm	5 minutes
SIAMAT colloidal silica on BLACKCHEM 2 polishing pad		10 lbs	100/100 rpm	5 minutes

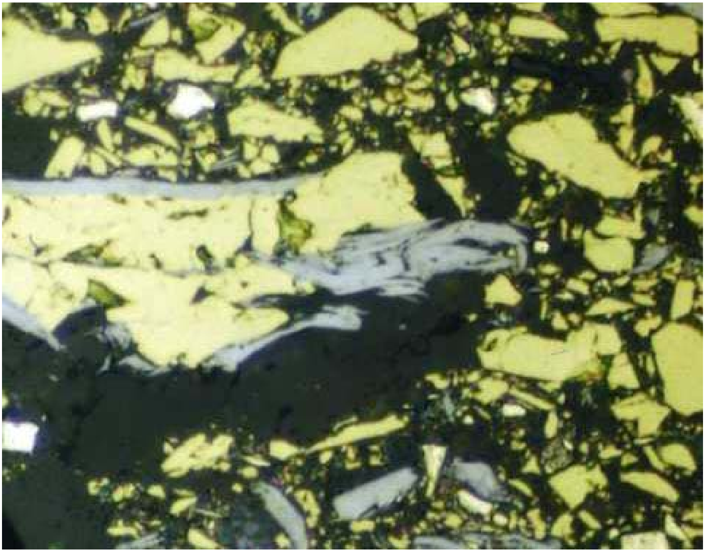

Figure 11-79a $CuFeS_2/MoS_2$ Mineral. Note that the two minerals have not been completely liberated from each other.

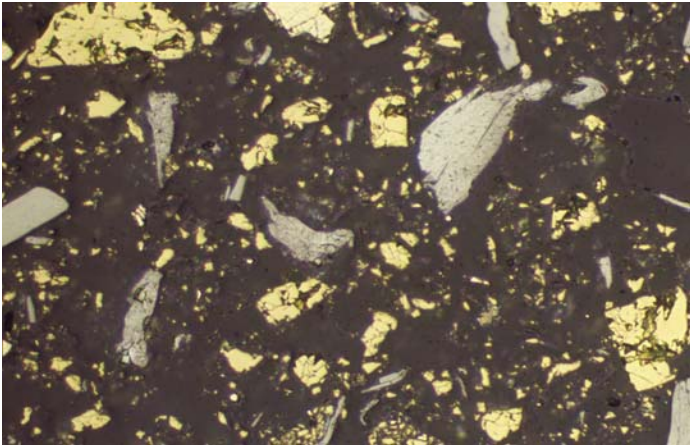

Figure 11-79b $CuFeS_2/MoS_2$ Mineral. Note the separation of the two minerals.

11.11.4 Minerals (Periclase)

Description:

Minerals range from pure elements to complex silicates.

Preparation Challenge:

Proper microstructural preparation requires minimizing fracturing and damage during cutting and initial grinding. The use of colloidal silica as a CMP polishing lubricant and abrasive significantly improves the surface finish for this class of materials.

SECTIONING

Diamond wafering blades - low grit / low concentration

MOUNTING

Castable mounting with epoxies or acrylic resins

POLISHING

Abrasive/Surface	Lubrication	Force/ sample	Speed (Head/Base)	Time
30 micron DIAMAT diamond on CERMESH metal mesh cloth		5-10 lbs	100/100 rpm	Until plane
6 micron DIAMAT diamond on TEXPAN polishing pad	SIAMAT colloidal silica	10 lbs	100/100 rpm	5 minutes
1 micron DIAMAT diamond on GOLDPAD polishing pad	SIAMAT colloidal silica	10 lbs	100/100 rpm	5 minutes
SIAMAT colloidal silica on BLACKCHEM 2 polishing pad		10 lbs	100/100 rpm	5 minutes

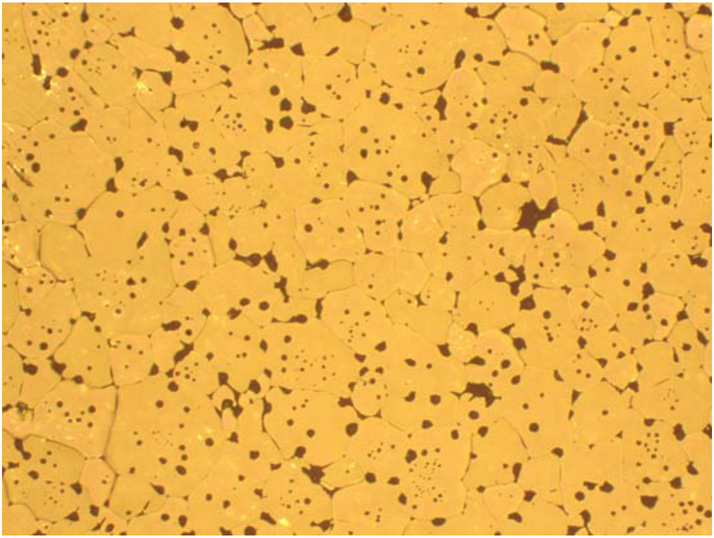

Figure 10-80a Periclase, 100X (B.F.) as polished.

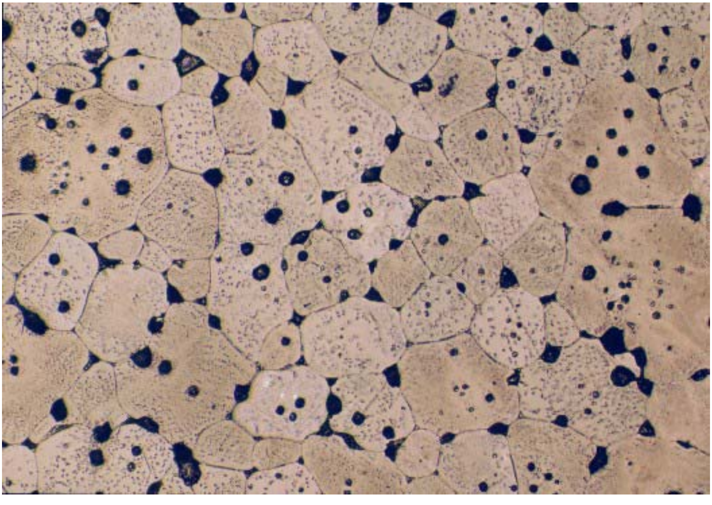

Figure 10-80b Periclase, 200X (B.F.) thermal etching at 1200° Celsius for 2 hours.

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APPENDIX B: SAFETY PROCEDURES

B.1 STORAGE

- Clearly label all containers

- HF, H_2SiF_6 and HBF_4 storage – these materials react with glass, therefore proper storage should be in polyethylene, polypropylene or similarly inert plastic containers (98).

- Strong alkaline solutions – these materials react with glass, therefore proper storage should be in polyethylene, polypropylene or similarly inert plastic containers (98).

- Phosphoric acid (H_3PO_4) – these materials react with glass, therefore proper storage should be in polyethylene, polypropylene or similarly inert plastic containers (98).

- Perchloric acid ($HClO_4$) – **do not** store high concentration of perchloric acid in plastic bottles (99).

B.2 DANGEROUS MIXTURES

Review all Material Safety Data Sheets (MSDS) before mixing

- *Perchloric acid* in concentrations exceeding 60% are highly **FLAMMABLE** and **EXPLOSIVE**. The danger is greatly increased by the presence of organic materials or metals that oxidize readily. Keep the temperature of the solution below 35°C (95°F) and, if necessary, use a coolant bath. Safety glasses are helpful; however, a safety shield is preferable.

- *Nital* (methanol/ethanol and HNO_3) can build up a gaseous reaction product and must be stored in a vented or pressure-relief container (98).

- *Mixtures of alcohol and hydrochloric acid* can react in various ways to produce aldehydes, fatty acids, explosive nitrogen compounds, etc. The likelihood of **EXPLOSION** increases with increasing molecule size. Hydrochloric acid content should not exceed 5% in ethanol or 35% in methanol. These mixtures should not be stored (99).

- *Mixtures of phosphoric acid* can result in the formation of ester, some of which are potent nerve **POISONS**. If absorbed through the skin or inhaled, severe personal injury may result (99).

- *Mixtures of methanol and sulfuric acid* may form dimethylene sulfate, an odorless, tasteless compound that may be **FATAL** if absorbed in sufficient quantities into the skin or respiratory tract. Even gas masks do not offer adequate protection. Sulfates of the higher alcohols, however, are not potentially dangerous poisons (99).

- *Mixtures of chromium (VI) oxide and organic materials* are **EXPLOSIVE**. Mix with care and do not store (99).

- *Lead and lead salts* are highly **TOXIC**, and the damage produced is cumulative. Care is also recommended when handling cadmium, thallium, nickel, mercury, and other heavy metals (99) to avoid toxicity.

- *Cyanide compounds (CN)* are highly dangerous because hydrocyanic acid (HCN) may easily form. These are fast-acting **POISONS** that can cause **DEATH**, even in relatively low concentrations.

- *Hydrofluoric acid* is a very strong skin and respiratory **POISON** that is hard to control. It should be handled with extreme care, because sores resulting from its attack on the skin do not readily heal. Hydrofluoric acid also attacks glass, and fumes from specimens etched in HF solution could easily damage front elements of microscope lenses. Specimens should be rinsed thoroughly and in some cases, placed in a vacuum desiccator for one to two hours before examination.

- *Picric acid anhydride* is an **EXPLOSIVE**.

- *Mixing oxidizing agents with reducing agents*. Mixing oxidizing agents, such as HNO_3, H_3SO_4, perchloric acid ($HClO_4$), CrO_3, salts of these acids, persulfates, Br_2 and H_2O_2, with reducing agents – for example, alcohols and other organic solvents, acetic acid, acetic anhydride and most organic compounds – requires special care (88). Mix slowly and stir continuously.

B.3 PERSONAL SAFETY

- Wear appropriate eye protection (safety goggles or safety glasses – see MSDS sheets).

- Wear approved rubber gloves and laboratory coats or aprons.

- Mix etchant and etch with adequate and appropriate ventilation. A fume hood is generally recommended.

- FIRST AID – Review MSDS sheets for specific medical instructions.

> SKIN – In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Get medical attention immediately. Wash clothing before reuse. Thoroughly clean shoes before reuse.
>
> EYES – Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.
>
> INHALATION – Remove to fresh air immediately. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention immediately.
>
> INGESTION: Refer to MSDS sheets for specific guidelines. Never give anything by mouth to an unconscious person. Get medical attention immediately.

B.4 MIXING GUIDELINES

- Acids and Bases: ALWAYS mix concentrated acids and bases into water to prevent excessive heat generation.

- Monitor temperature of mixture to prevent overheating.

- Mix in well ventilated area.

- Use recommended personal safety equipment.

B.5 DISPOSAL

When appropriate, dilute all concentrated chemicals prior to disposal. If regulations allow disposal to sewer, use a substantial amount of running water and slowly add etchant to flow. Continue to purge drain thoroughly with water. Follow all Local, State and Federal Disposal Guidelines.

B.6 DISCLAIMER

THE INFORMATION AND RECOMMENDATIONS SET FORTH HEREIN ARE TAKEN FROM SOURCES BELIEVED TO BE ACCURATE AS OF THE DATE HEREIN: HOWEVER, PACE TECHNOLOGIES MAKES NO WARRANTY, EXPRESS OR IMPLIED WITH RESPECT TO THE ACCURACY OF THE INFORMATION OR THE SUITABILITY OF THE RECOMMENDATIONS, AND ASSUMES NO LIABILITY TO ANY USER THEREOF.

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About the Author

Donald Zipperian was born in Butte, Montana, in 1959 and studied Metallurgical Engineering, Metallurgy and Materials Science at the University of Arizona. For more than 25 years he has worked in research and development of metallographic specimen preparation and grinding and polishing of advanced materials. He is the author and coauthor of many technical papers in the field of materials science and has been an Adjunct Professor at both the University of Arizona and Clemson University. Dr. Zipperian is currently the CTO (Chief Technical Officer) at PACE Technologies in Tucson, Arizona.

Prior to joining PACE Technologies, Dr. Zipperian was the General Manager for BUEHLER Ltd's Southwest Research Center where he developed many of the consumable products currently sold by BUEHLER and other metallographic suppliers today. Dr. Zipperian also managed SEAGATE Technologies surface finishing group in Minneapolis, Minnesota where he lead the development and implementation of advanced surface finishing techniques for producing nanometer finishes on the read-write heads used in computer hard drives.

Dr. Zipperian has also been on numerous ASM and ASTM committee's related to metallography.

Metallographic Equipment

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