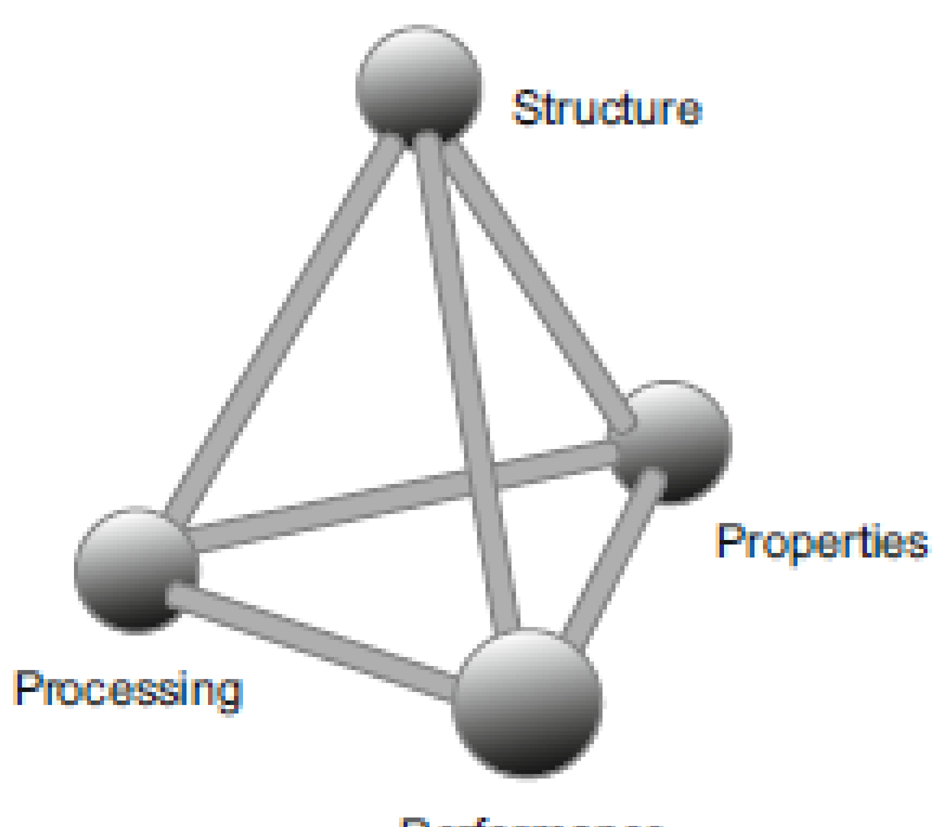
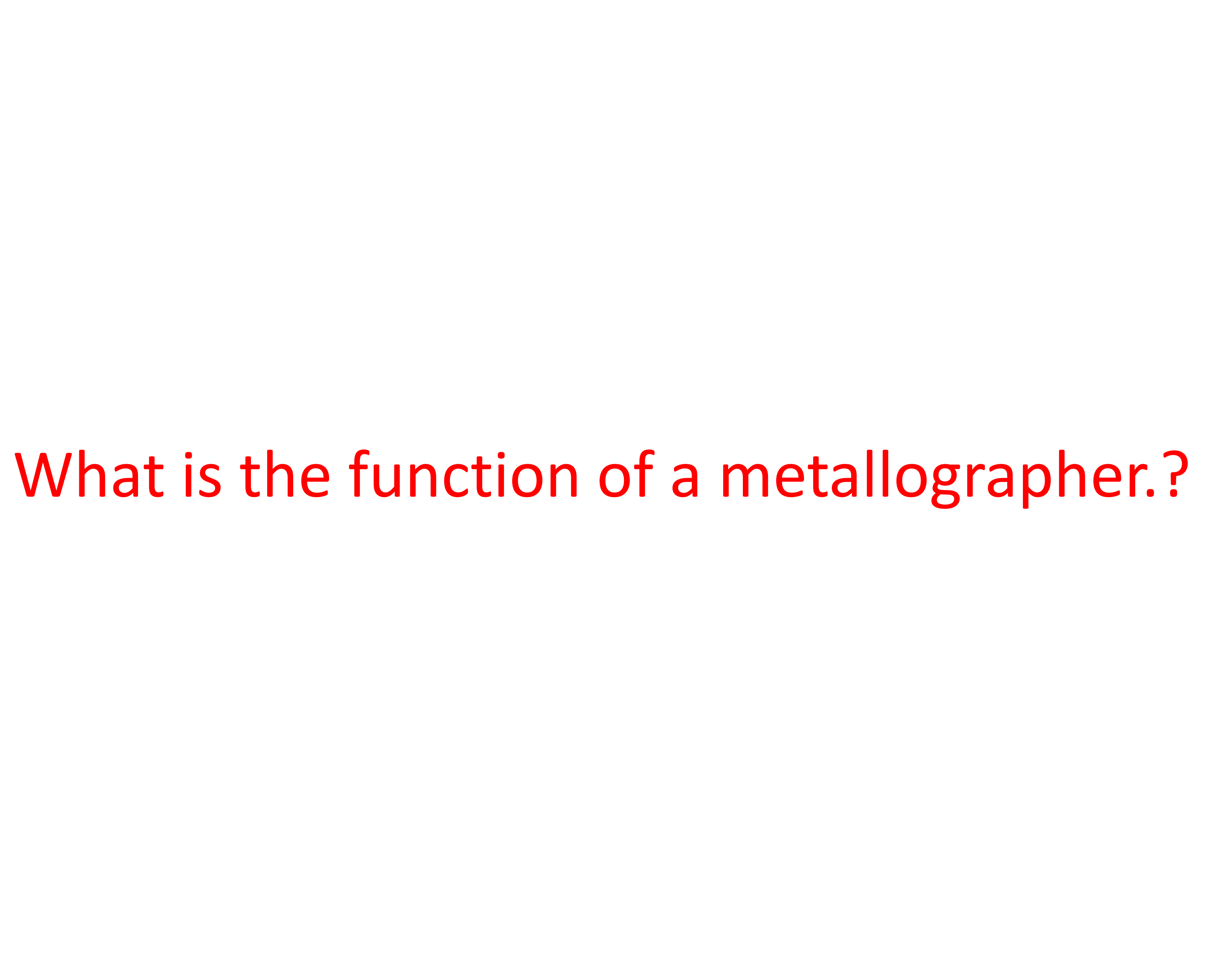
What is Metallography.?

- **Metallography**
  It is a branch of materials science which relates to the constitution and structure, and their relation to the properties, of metals and alloys.

- METALLOGRAPHY is the scientific discipline of examining and determining the constitution and the underlying structure of (or spatial relationships between) the constituents in metals, alloys and materials (sometimes called materialography).

- METALLOGRAPHY (or MATERIALOGRAPHY) is the characterization of structure of alloys (or materials) with optic microscope (mostly).


Structure
Properties
Processing
Performance


What is the function of a metallographer.?

- Every metallographer should know and understand how the various microstructures in alloys originate.
- This is because a key part of a metallographer's job is to interpret what he or she sees under the microscope and to make sound judgments and recommendations based on these observations. A metallograhper is not just an "metal polisher," as perceived by many people outside the profession.
- An effective metallographer needs to have a basic understanding of the fundamentals of ferrous and non-ferrous physical metallurgy. The basic tenet of ferrous and non-ferrous physical metallurgy is that the properties of alloys (for examples, steels and cast irons) are controlled by both microstructure and chemical composition. It is the metallographer's job to determine and characterize the microstructure, and it is the chemist's job to determine the composition.

- This course discusses, in an introductory way, some of the basic ferrous and non-ferrous physical metallurgy principles that are needed by the metallographer.

- The discussion focuses on the numerous microstructures that are generated as a result of the phase transformations that occur during both heat treatment (as in steels and Al, Cu, Zn wrought alloys) and solidification (as in cast irons and Al, Cu, Zn cast alloys).

- Next chapters show how numerous factors can influence microstructural development and how one can alter microstructure to attain desired properties.

Who is the founder of Metallography.?

(from the University of Sheffield, England, UK)

Henry Clifton Sorby (1826–1908), geologist, petrographer, mineralogist, and founder of metallography. (and materials science)

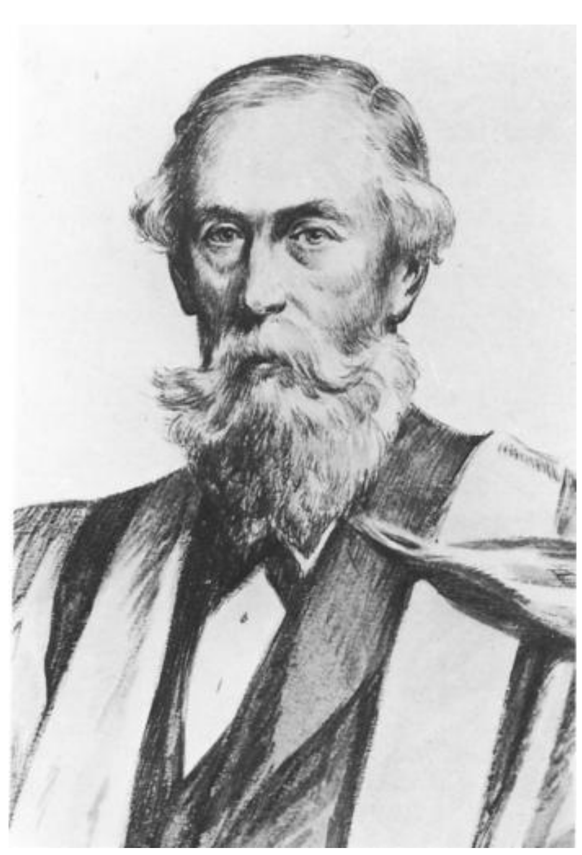
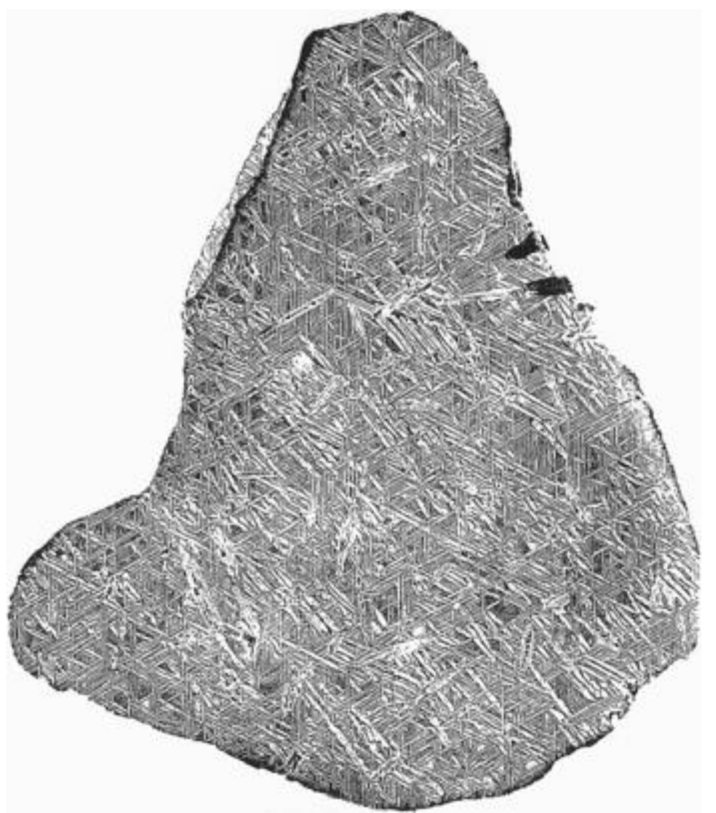


Fig. 4.1 Henry Clifton Sorby, the father of metallography

Macrograph of the Elbogen iron meteorite prepared in 1808 by Widmanstätten and Schreibers using heavy etching in nitric acid. After rinsing in water and drying, printer's ink was rolled on the etched surface, and the sample was pressed onto a piece of paper.



Jose Ramon Vilella (1897–1971), distinguished metallographer who understood the need to faithfully prepare representative surfaces in metallographic examinations.

George F. Vander Voort

material selection → Properties → microstructure

microstructure ↔ alloying

microstructure ↔ processing

microstructure ↔ heat treatment

Problem:
broad range

Alloy and heat treatment selection

Properties of Materials
(9 different catagories)

- Electrical (electrical conductivity, dielectric constant…)
- Optical (index of refraction..)
- Magnetic (magnetic permeability..)
- Mechnanical (elastic modulus, strength, hardness..)
- Thermal (Heat capacity, thermal conductivity..)
- Corrosion (or Chemical or deteriorative) (…)
- Nuclear
- Physical
- Fabrication properties

Physical and Optical Properties

- Crystal structure
- Atomic weight
- Density
- Melting point
- Boiling point
- Vapor pressure
- Viscosity
- Porosity
- Permeability
- Reflectivity
- Transparency
- Optical properties
- Dimensional stability

Electrical properties

- Conductivity
- Resistivity
- Dielectric constant
- Superconducting temperature

Magnetic properties

- Magnetic susceptibility
- Magnetic permeability
- Coercive force
- Saturation magnetization
- Transformation (Curie) temperature
- Magnetostriction

Nuclear properties

- Half-life
- Stability

Mechanical properties

- Hardness
- Modulus of elasticity
- Compression
- Poisson's ratio
- Stress-strain curve
- Yield strength
- Compression
- Ultimate strength
- Tension
- Shear
- Bearing
- Fatigue properties
- Smooth
- Notched
- Corrosion fatigue
- Rolling contact
- Fretting
- Charpy transition temperature
- Fracture toughness (*KIc)*
- High-temperature behavior

Creep
Stress rupture
Damping properties
Wear properties
Galling
Abrasion
Erosion
Cavitation
Spalling
Ballistic impact

Thermal properties

- Conductivity
- Specific heat
- Coefficient of thermal expansion
- Latent heat of fusion
- Emissivity
- Absorptivity
- Fire resistance

Chemical properties

- Position in electromotive series
- Galvanic corrosion
- Corrosion and degradation
- Atmospheric
- Fresh water
- Salt water
- Acids
- Hot gases
- Ultraviolet
- Oxidation
- Thermal stability
- Biological stability
- Stress corrosion
- Hydrogen embrittlement

Fabrication properties

- Castability
- Heat treatability
- Hardenability
- Formability
- Machinability
- Weldability

Comparison of some properties

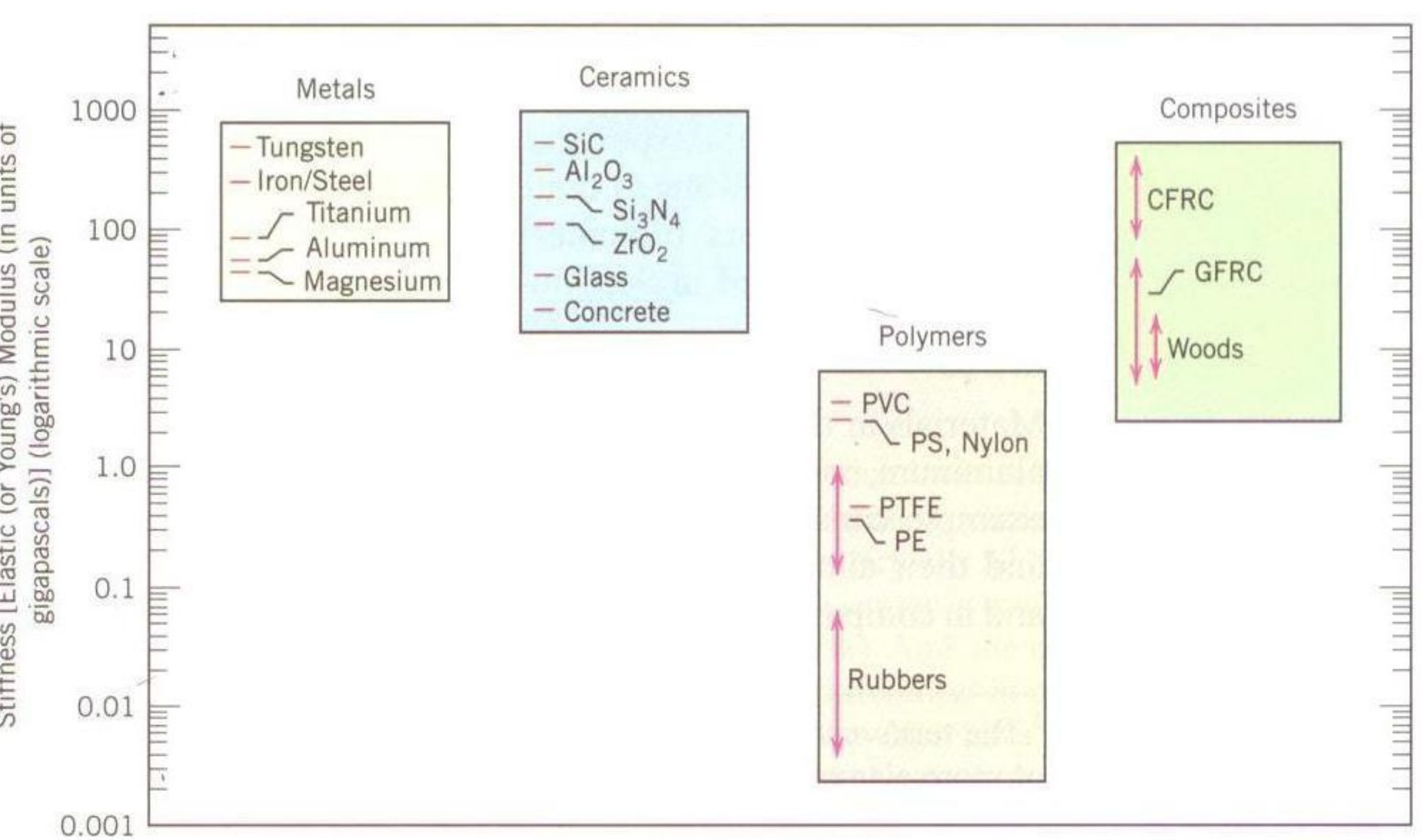


Figure 1.4 Bar-chart of room-temperature stiffness (i.e., elastic modulus) values for various metals, ceramics, polymers, and composite materials.

Comparison of some properties

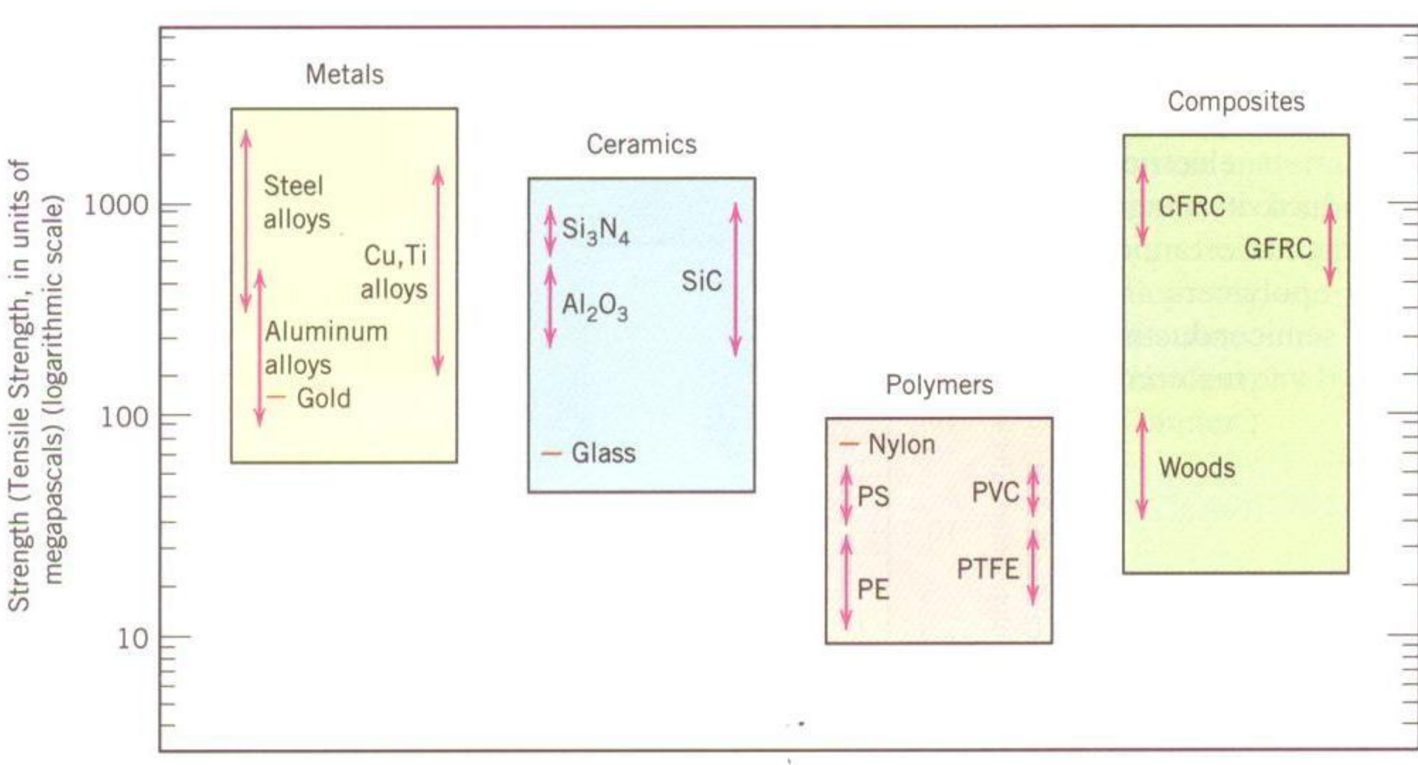


Figure 1.5 Bar-chart of room-temperature strength (i.e., tensile strength) values for various metals, ceramics, polymers, and composite materials.

Comparison of some properties

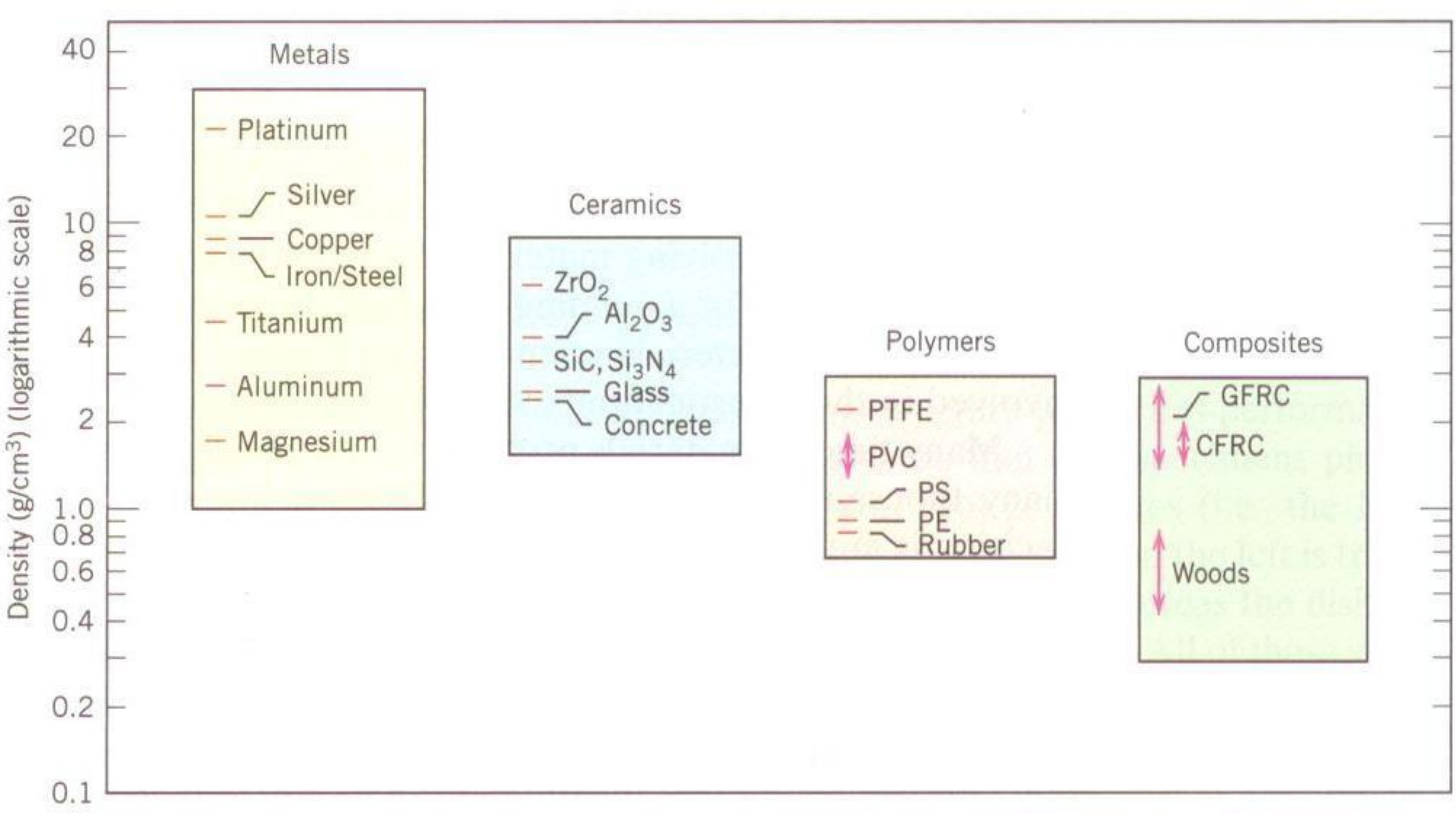


Figure 1.3 Bar-chart of room-temperature density values for various metals, ceramics, polymers, and composite materials.

Comparison of some properties

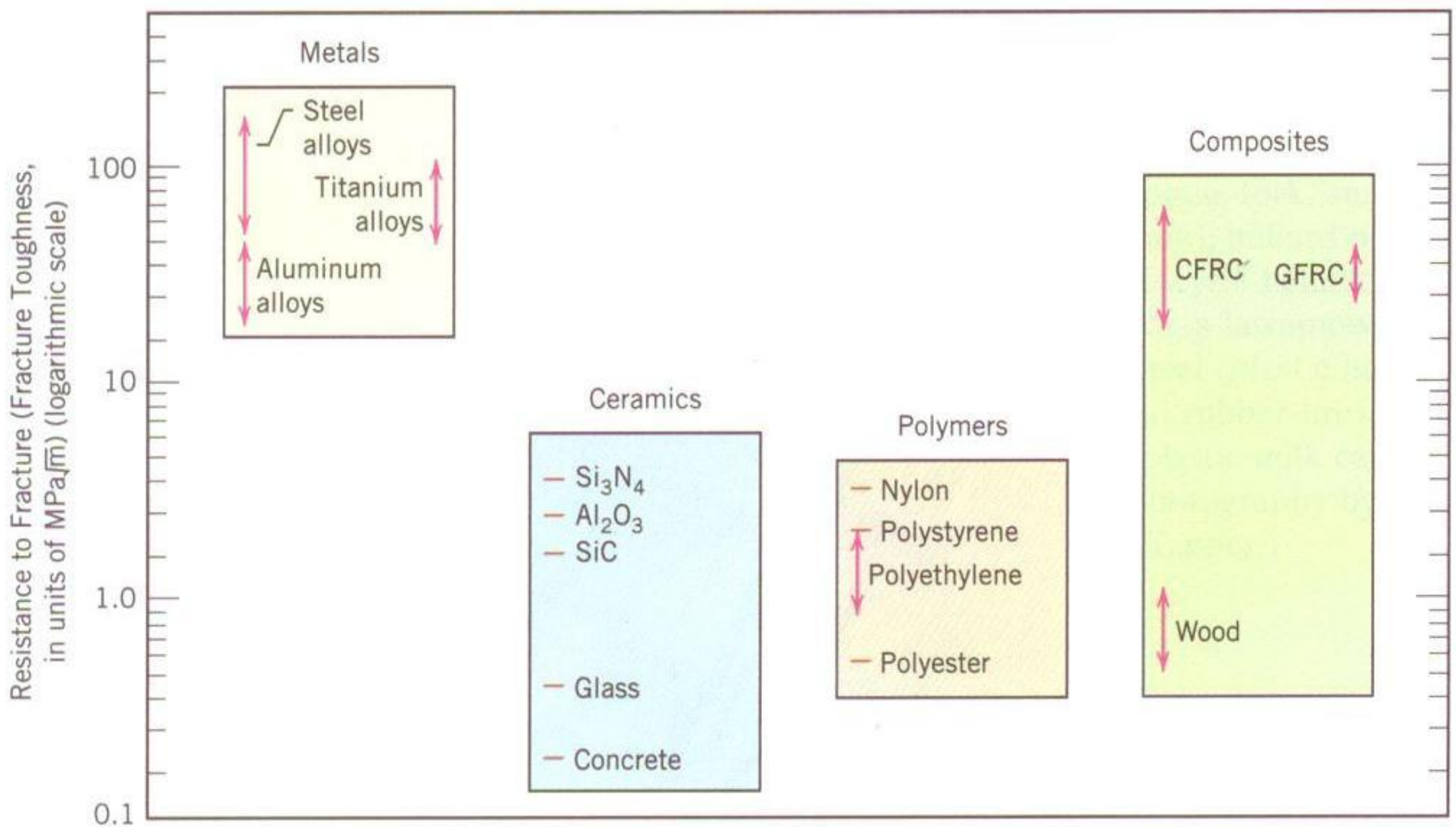
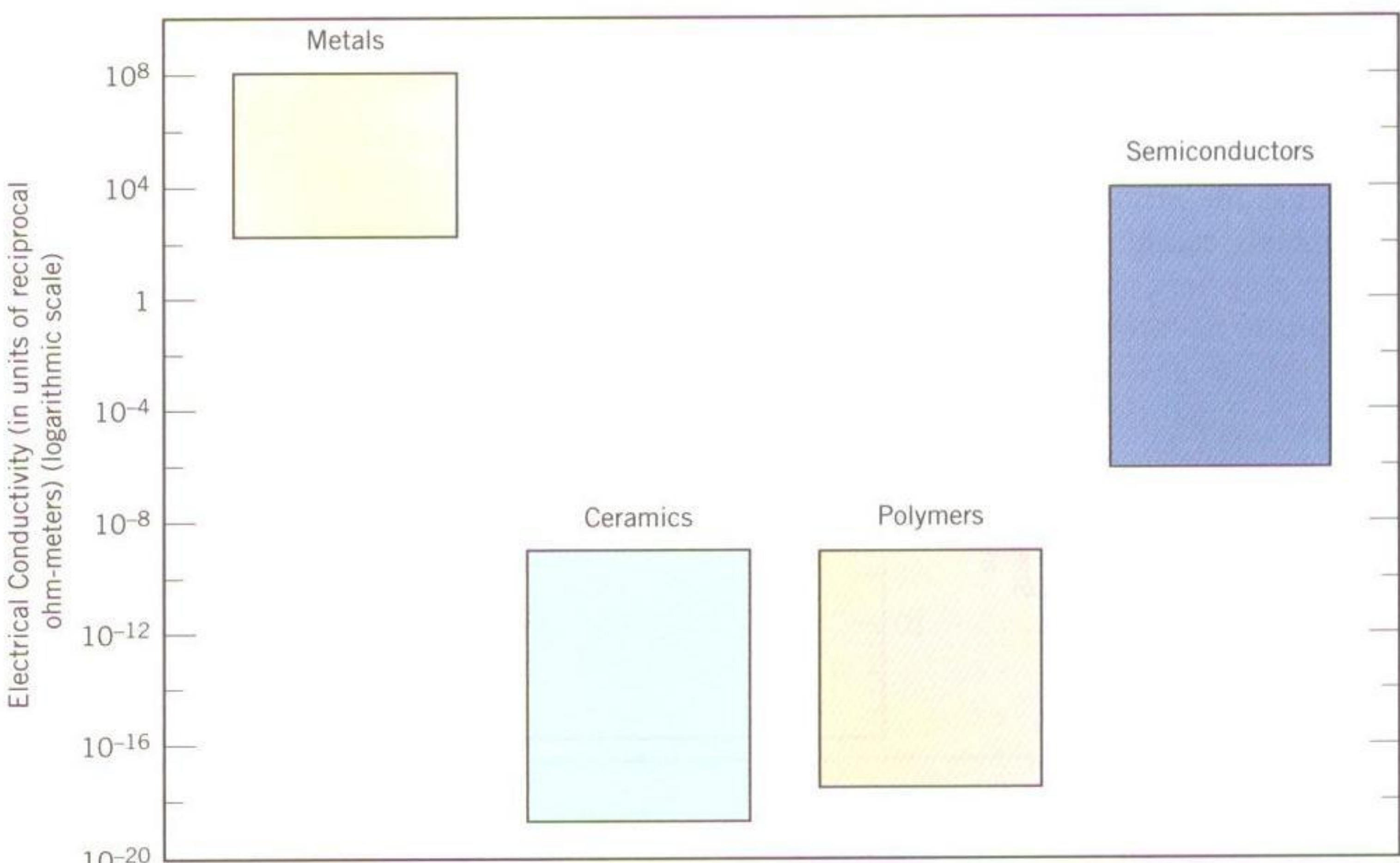


Figure 1.6 Bar-chart of room-temperature resistance to fracture (i.e., fracture toughness) for various metals, ceramics, polymers, and composite materials. (Reprinted from *Engineering Materials 1: An Introduction to Properties, Applications and Design,* third edition, M. F. Ashby and D. R. H. Jones, pages 177 and 178, Copyright 2005, with permission from Elsevier.)

Comparison of some properties

Figure 1.7
Bar-chart of room-temperature electrical conductivity ranges or metals, ceramics, polymers, and semiconducting materials.



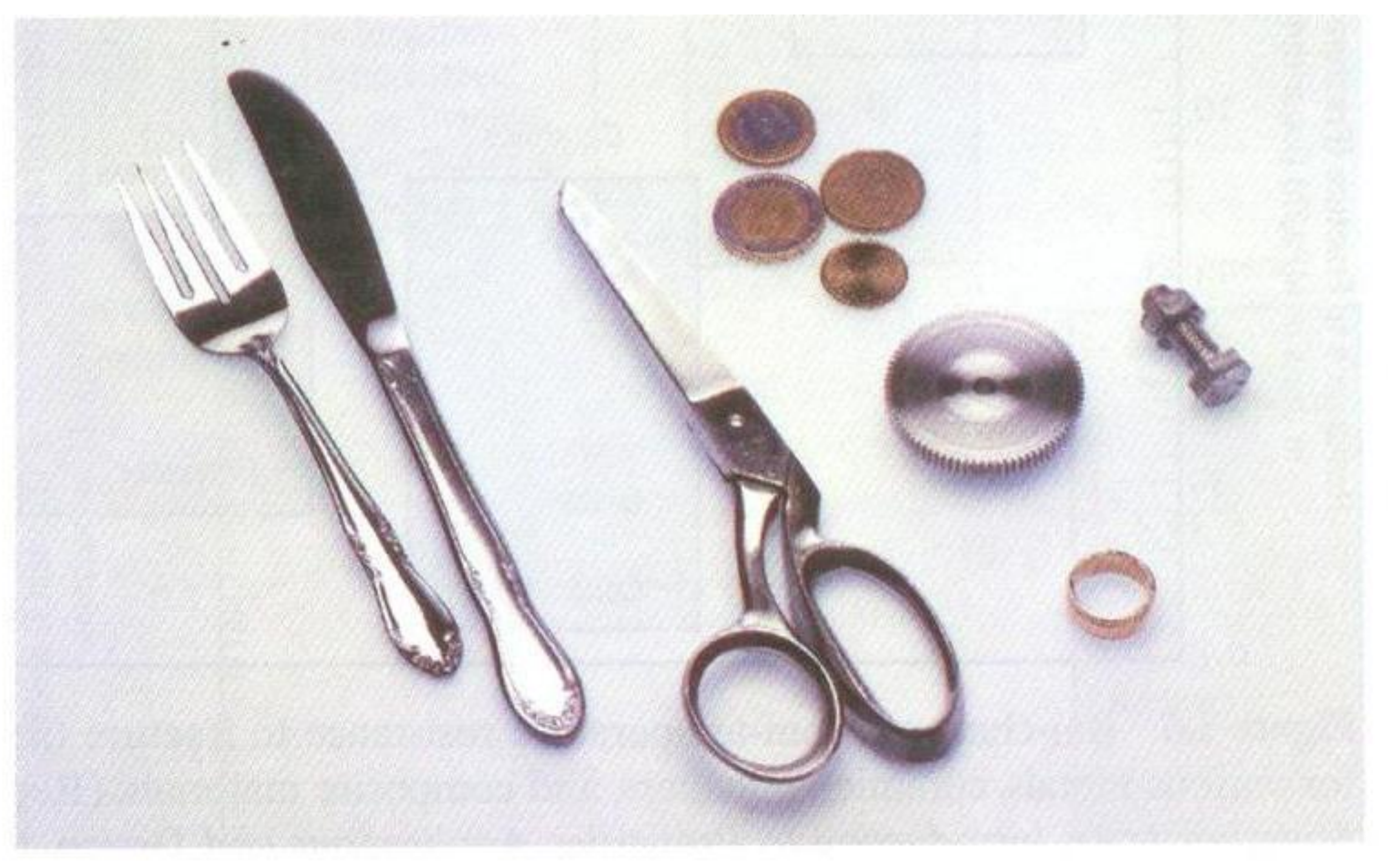
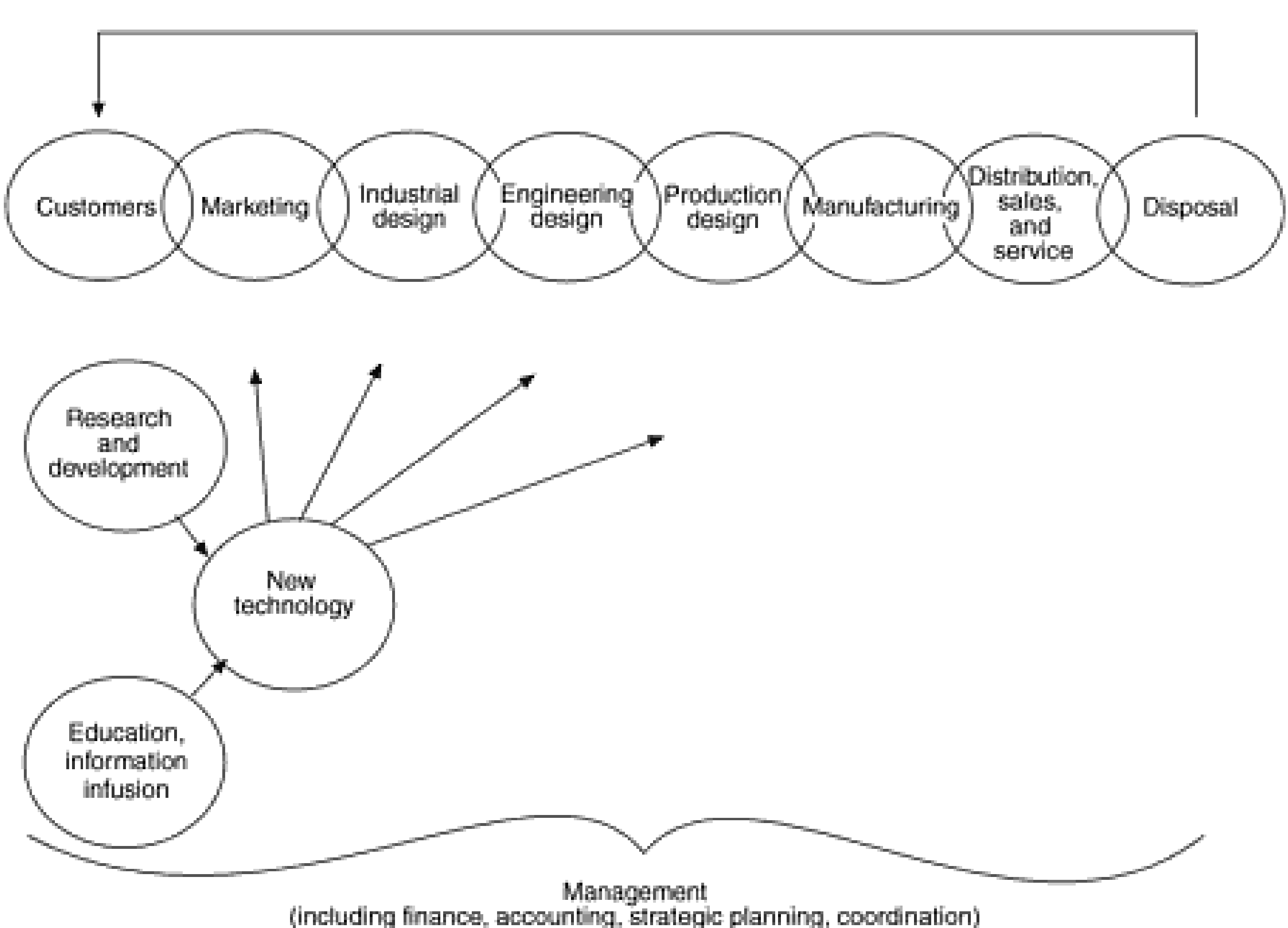
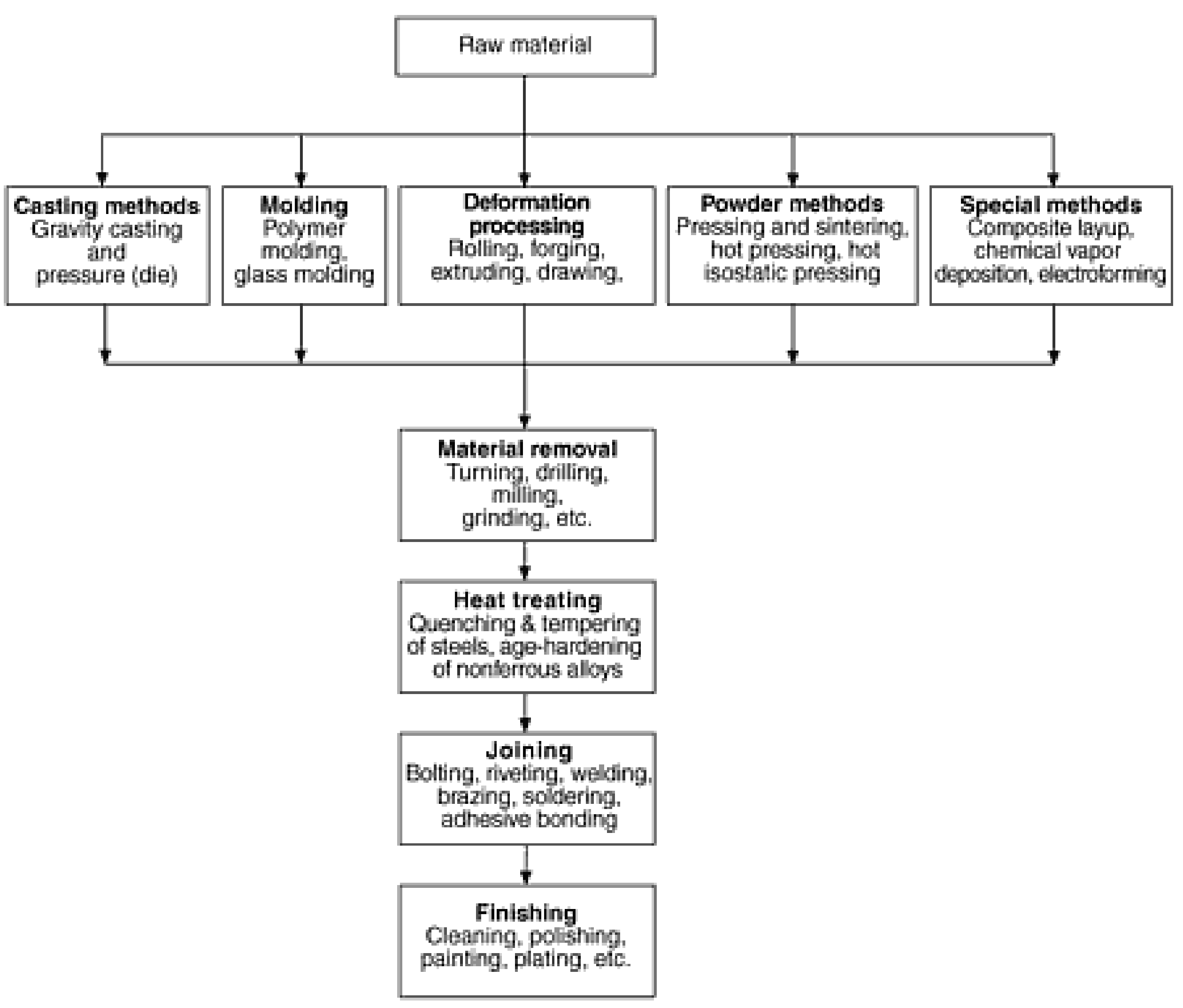


Figure 1.8 Familiar objects that are made of metals and metal alloys: (from left to right) silverware (fork and knife), scissors, coins, a gear, a wedding ring, and a nut and bolt. (Photograpy by S. Tanner.)

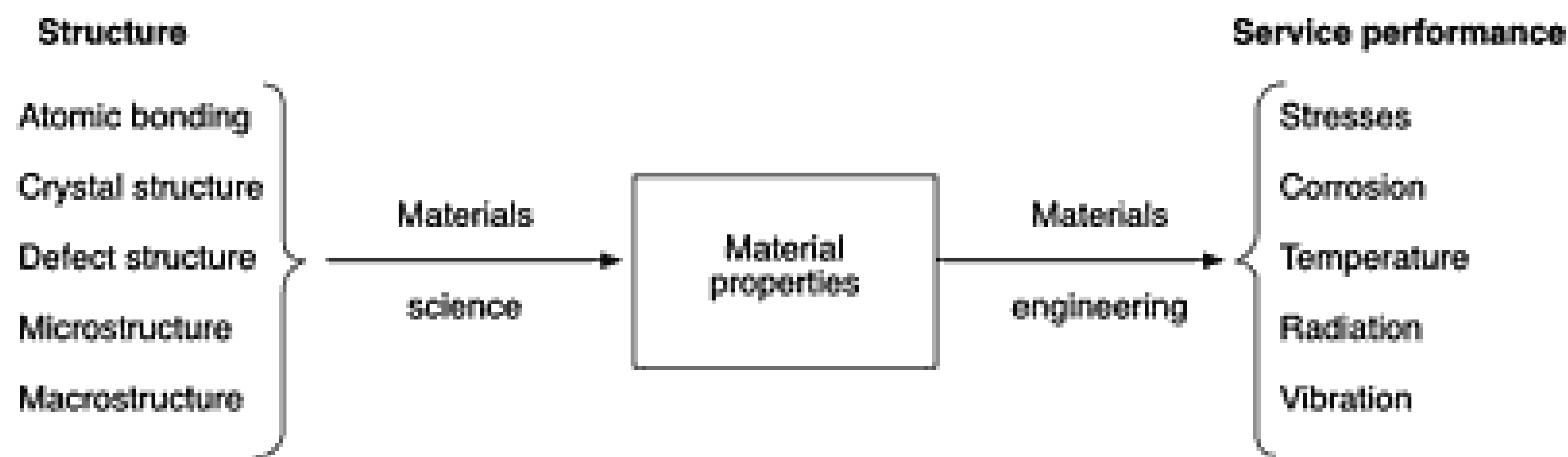
Engineering design as a part of the product realization process



The nine classes of manufacturing processes. The first row contains the primary forming (shaping) processes. The processes in the lower vertical column are the secondary forming and finishing processes.



The role played by material properties in the selection of materials



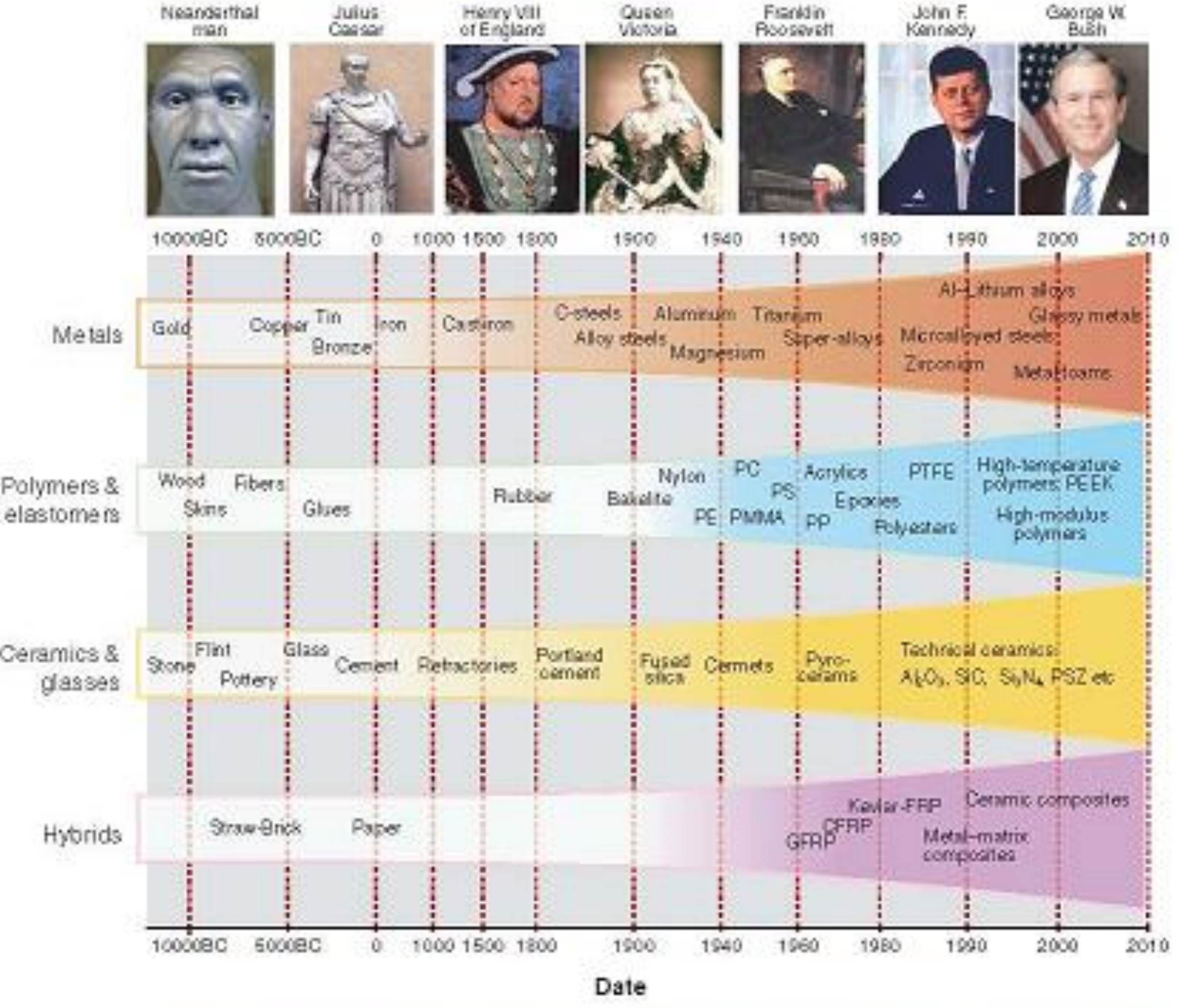
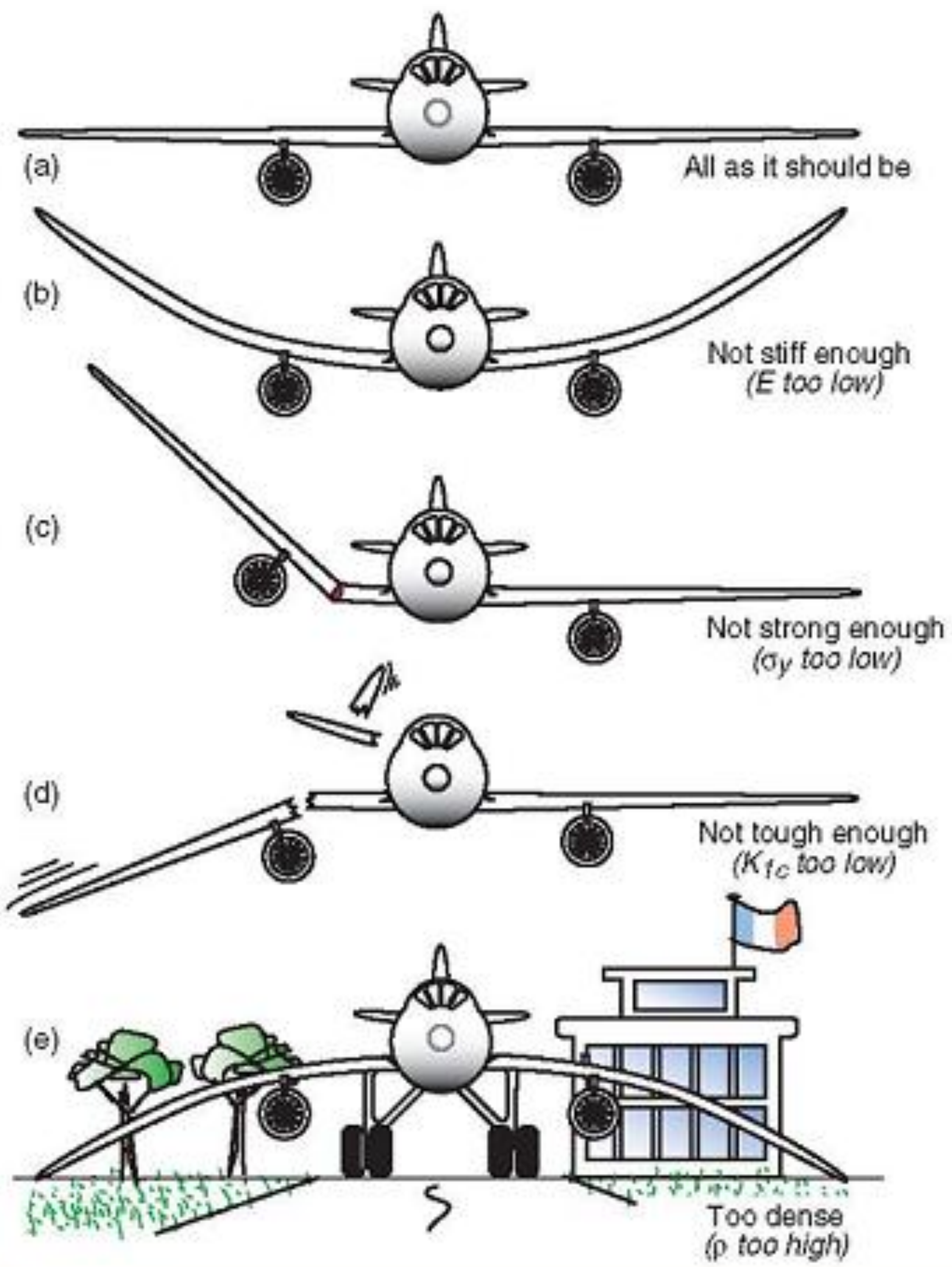
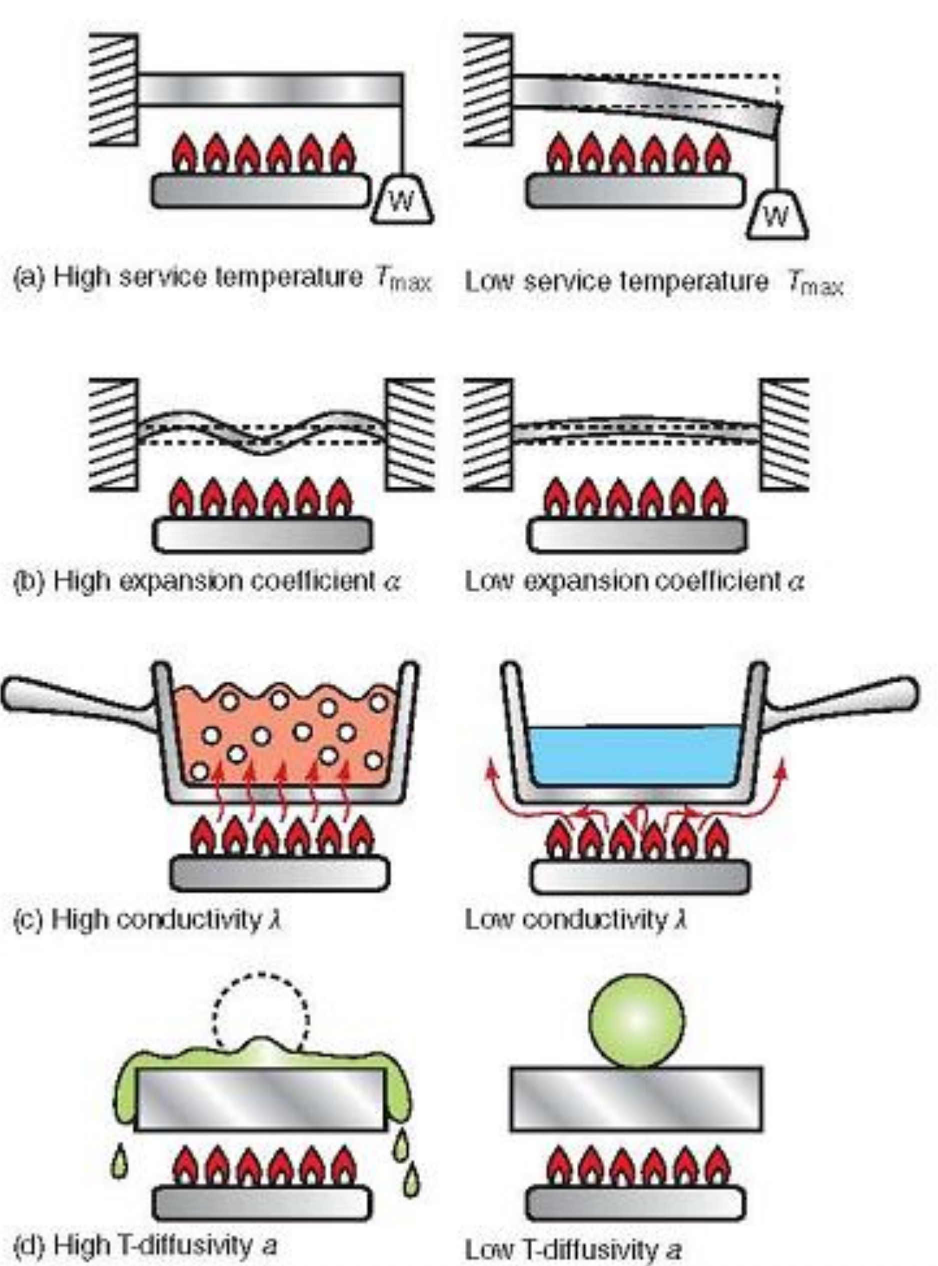


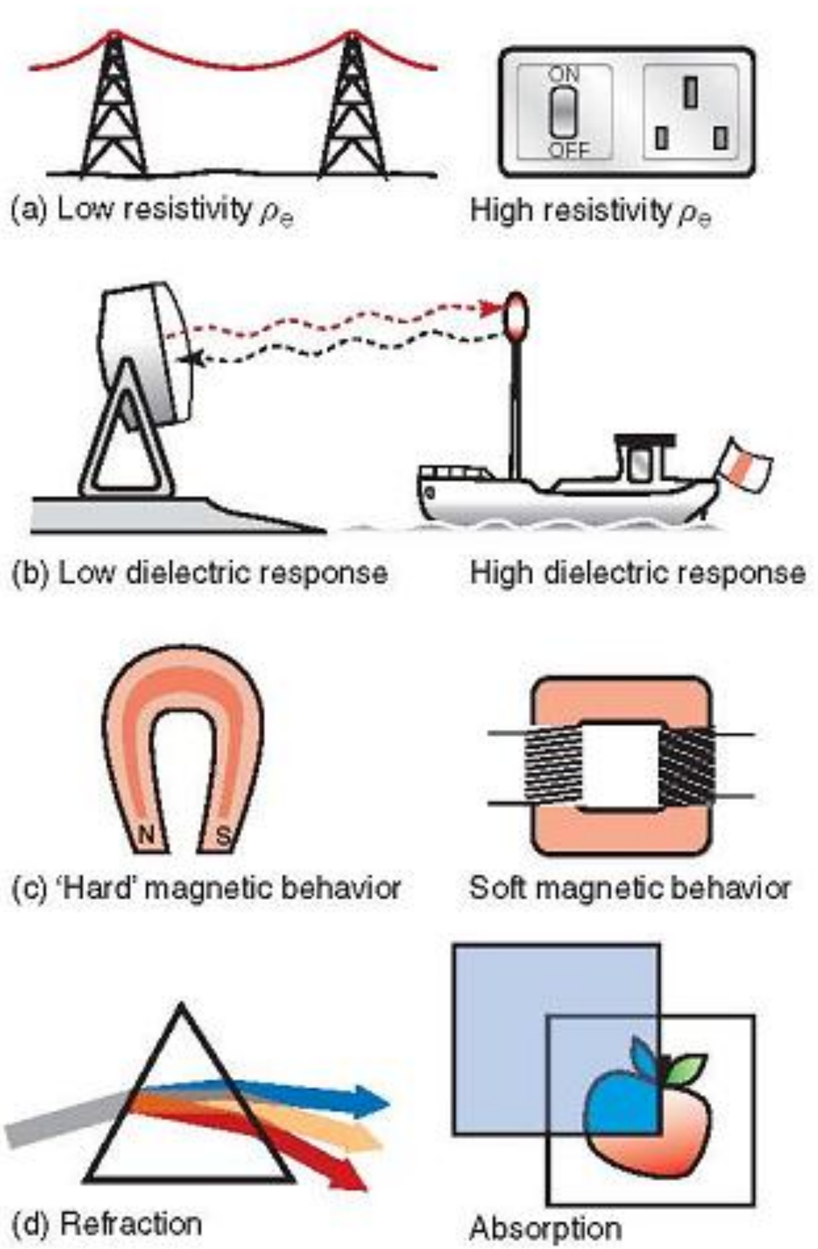
Figure 1.1 The development of materials over time. The materials of pre-history, on the left, all occur naturally; the challenge for the engineers of that era was one of shaping them. The development of thermochemistry and (later) of polymer chemistry enabled man-made materials, shown in the colored zones. Three—stone, bronze and iron—were of such importance that the era of their dominance is named after them.



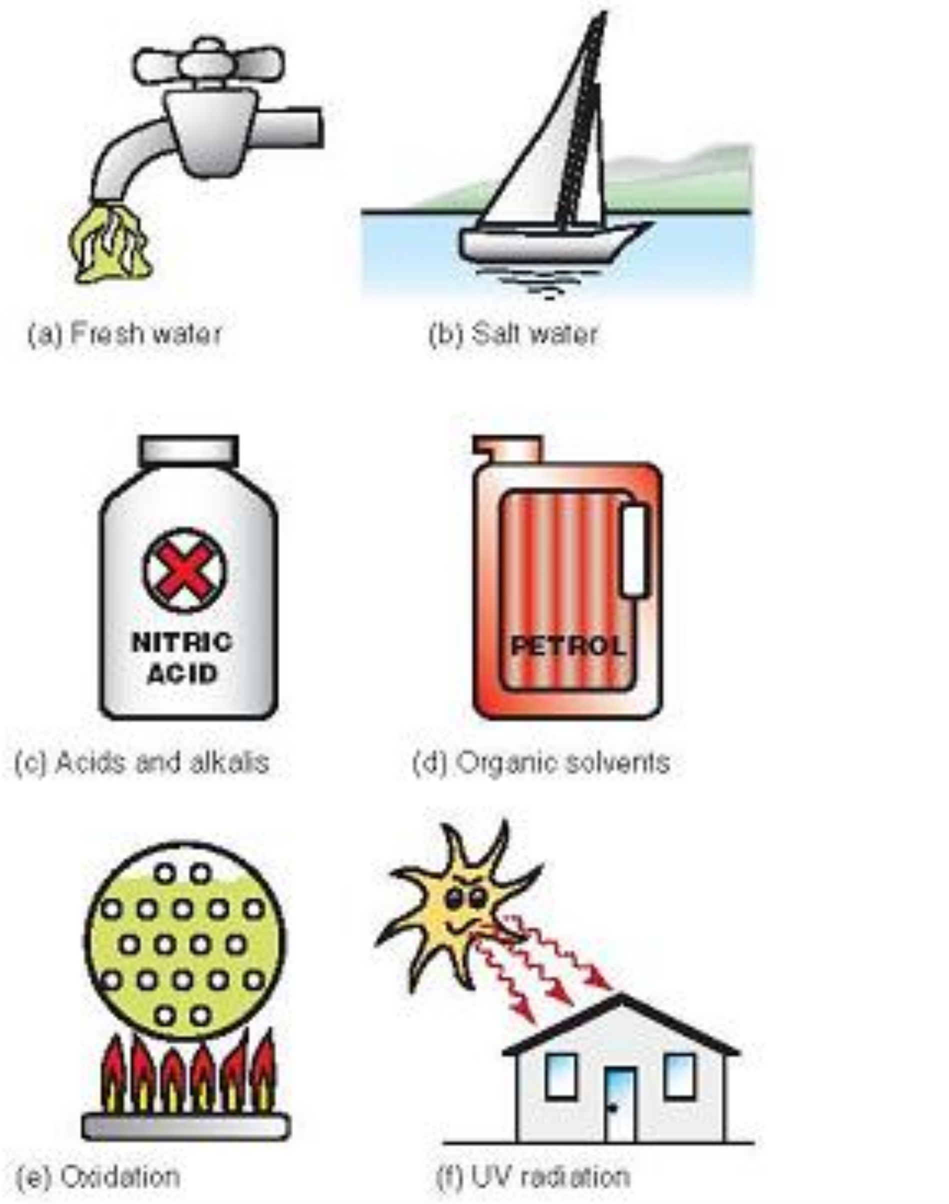
2 Mechanical properties.



Thermal properties.



Electrical, magnetic and optical properties.



Chemical properties: resistance to water, acids, alkalis, organic solvents, oxidation and radiation.

What is Microstructure.?

- **What is Microstructure**.?
  The structure of a suitable prepared specimen as revealed by a microscope.
- 1. Microstructure
- 2. Macrostructure

Dimensions of structural feature (m)

10^{-14} 10^{-12} 10^{-10} 10^{-8} 10^{-6} 10^{-4} 10^{-2}

Subatomic particles

Atom/ion diameters

Unit cell edge lengths

Dislocations (width)

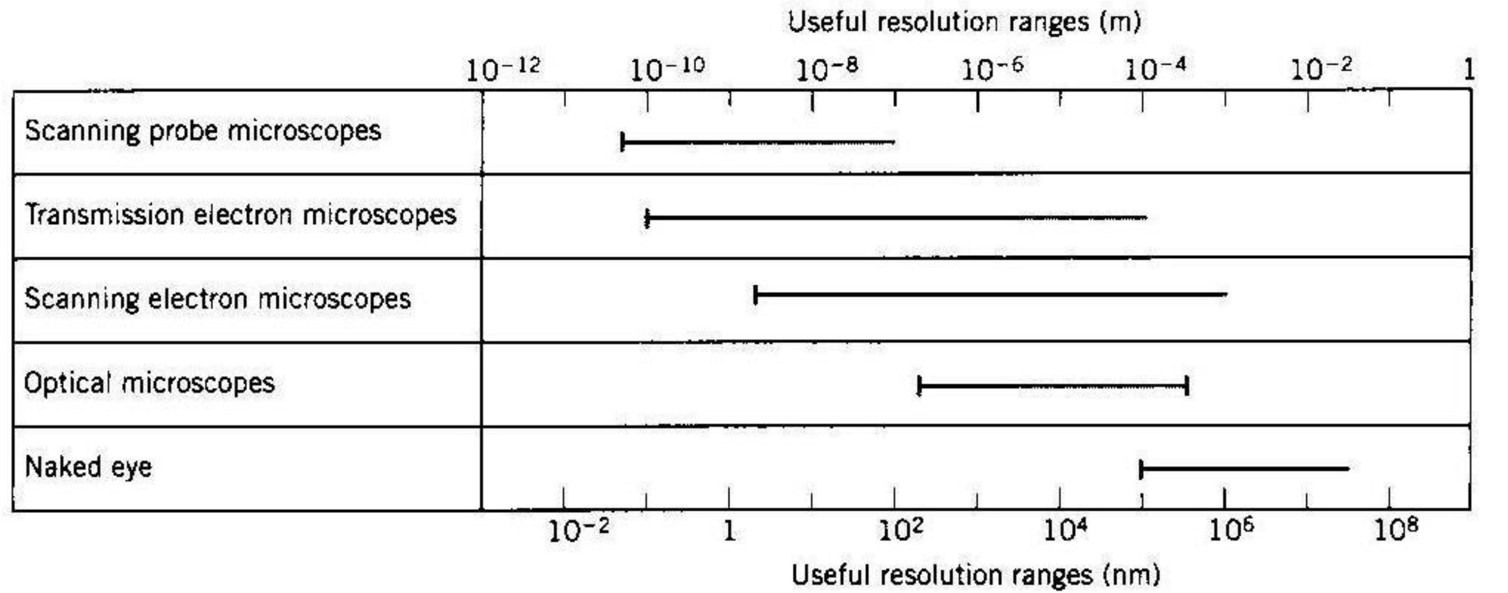
Second phase particles

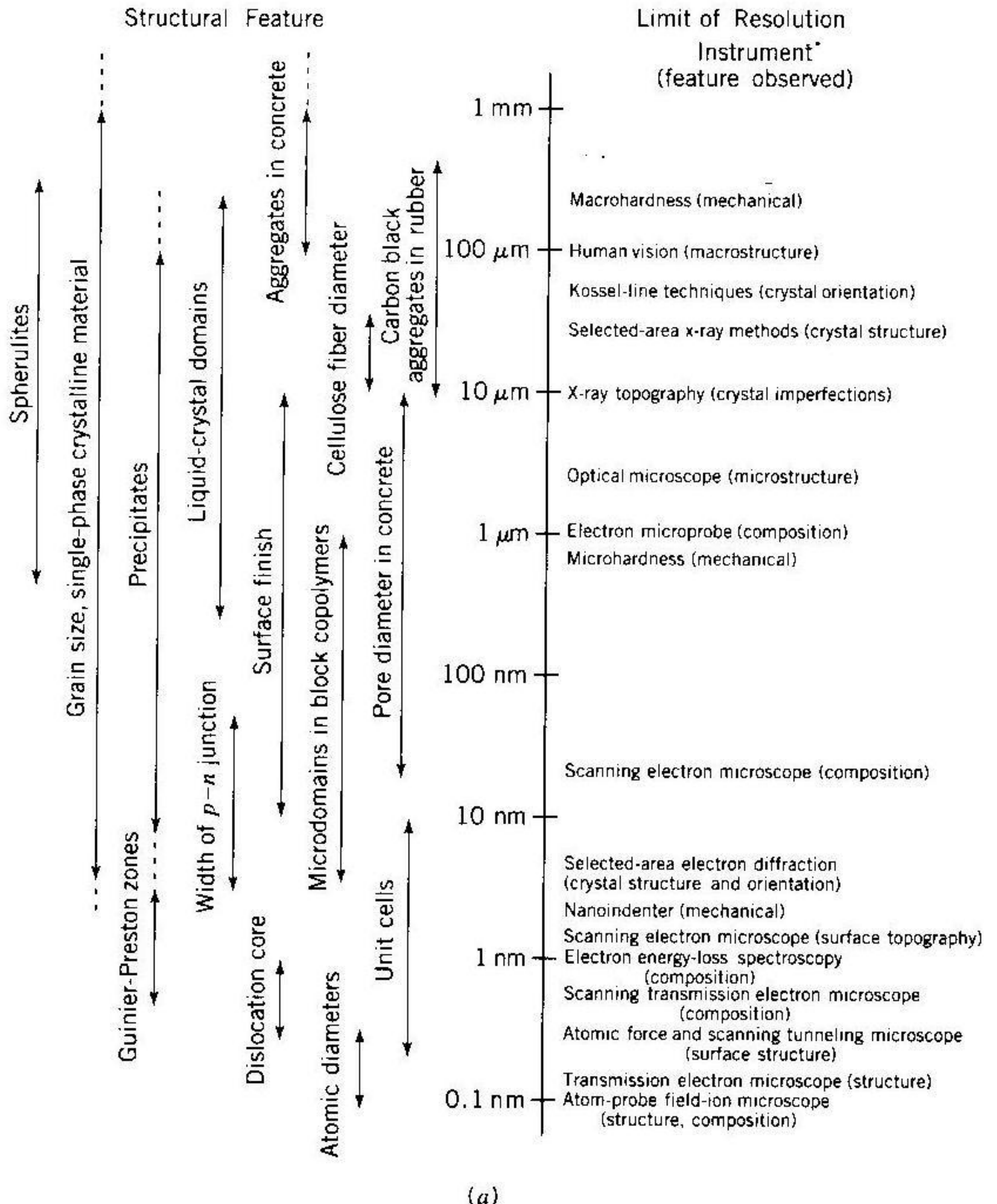
Grains

Macrostructural features (porosity, voids, cracks)

10^{-6} 10^{-4} 10^{-2} 1 10^{2} 10^{4} 10^{6} 10^{8}

Dimensions of structural feature (nm)





(*a*)

Figure 6.1 (*a*) Size scale relating structural features of materials to experimental techniques for structural observation (after Allen and Bever, 1986, p. 4700).

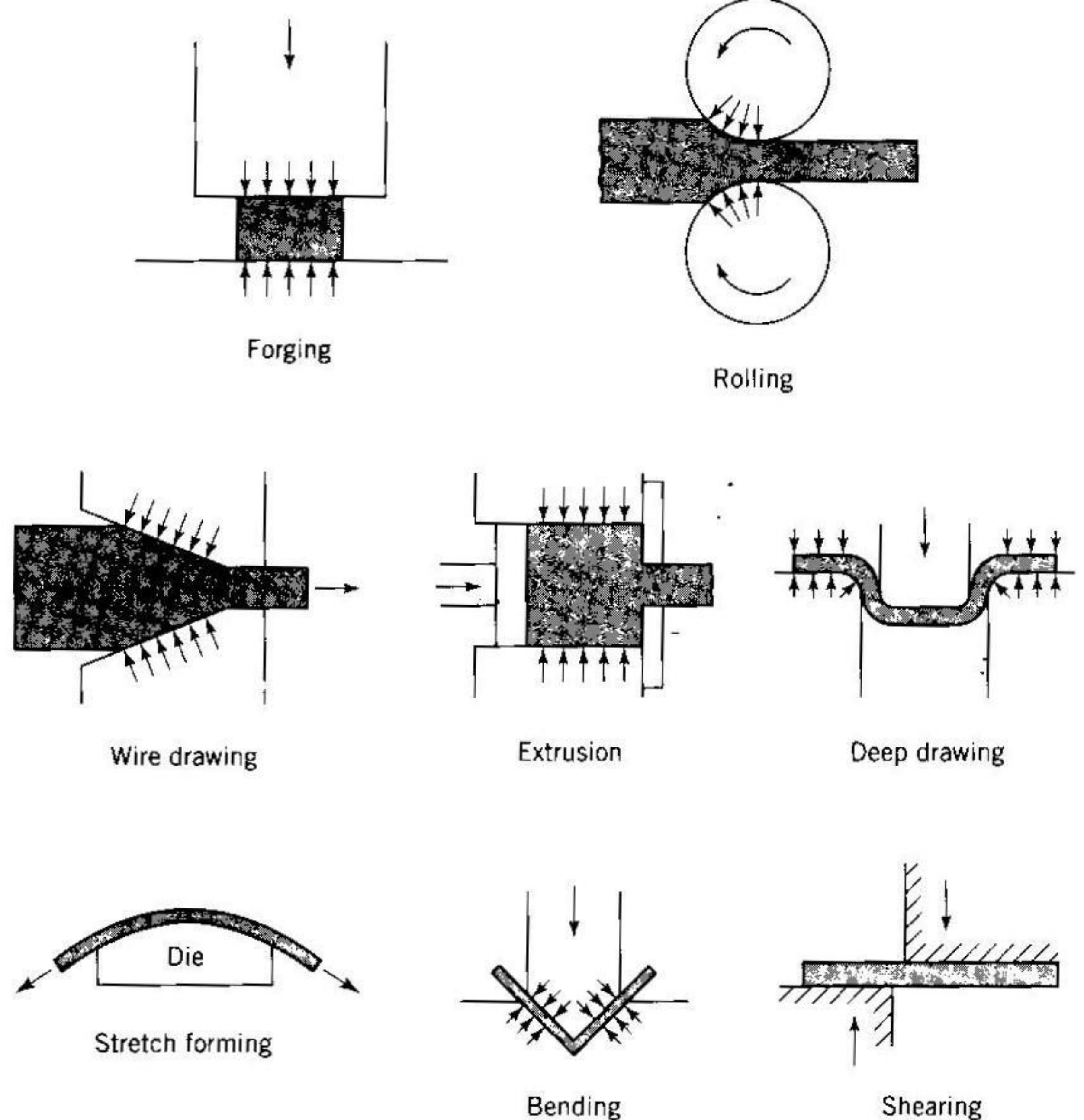


Figure 6.8 Illustration of deformation processing operations. Deforming material is indicated by shading (Dieter, 1986, p. 504).

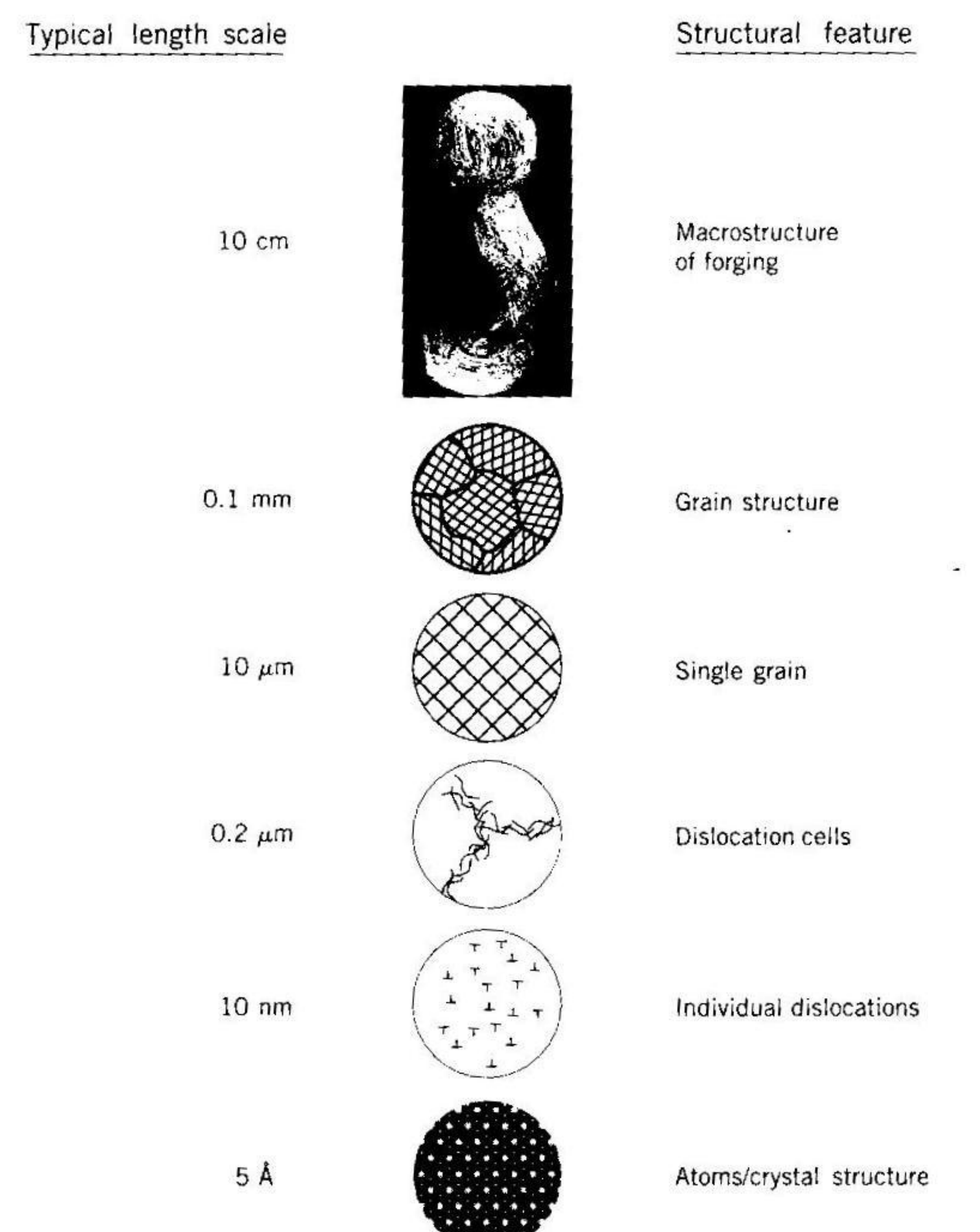


Figure 6.2 Hierarchical structure in metal forging (American Society for Materials, 1988, Vol. 14, p. 367).

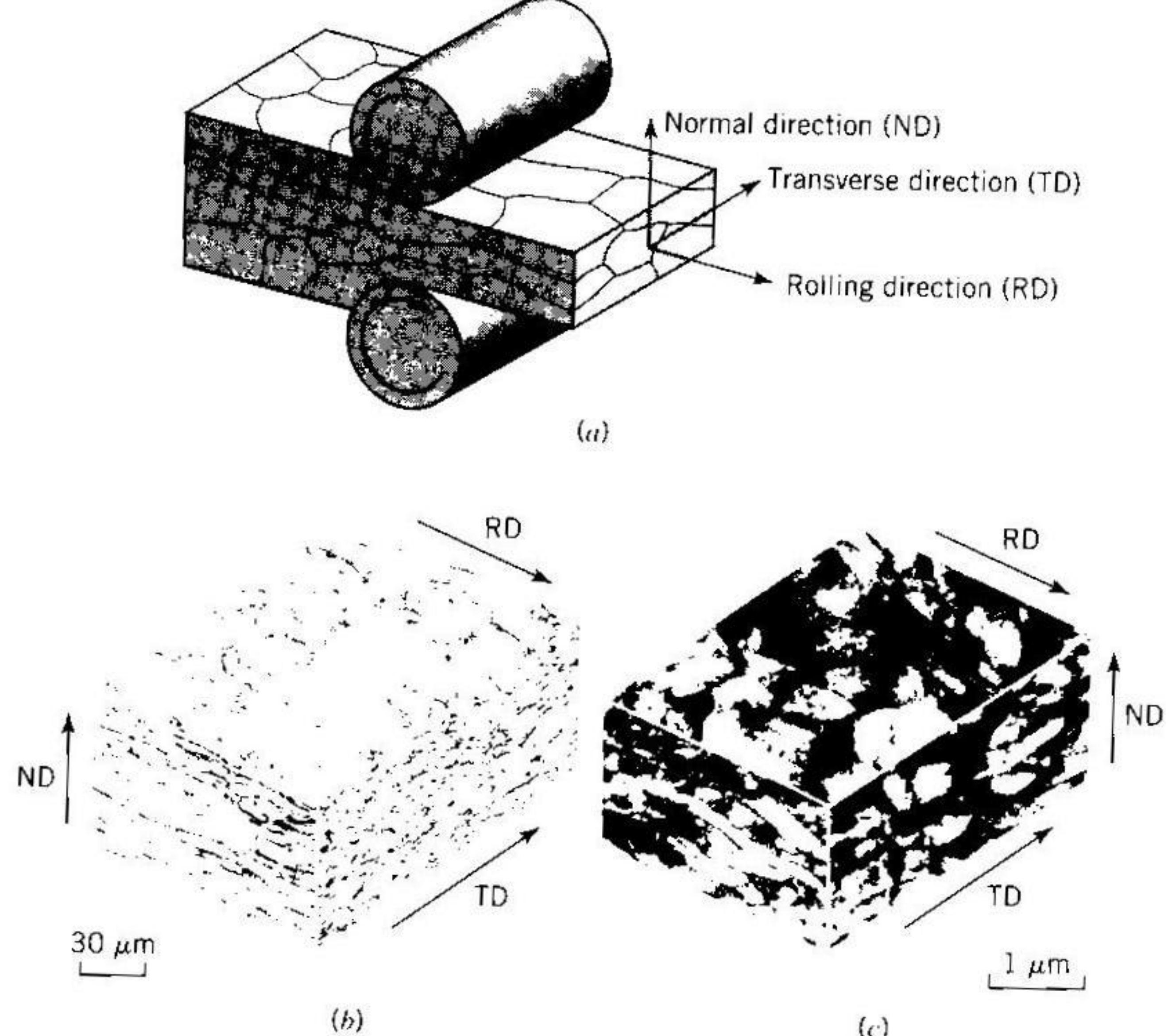
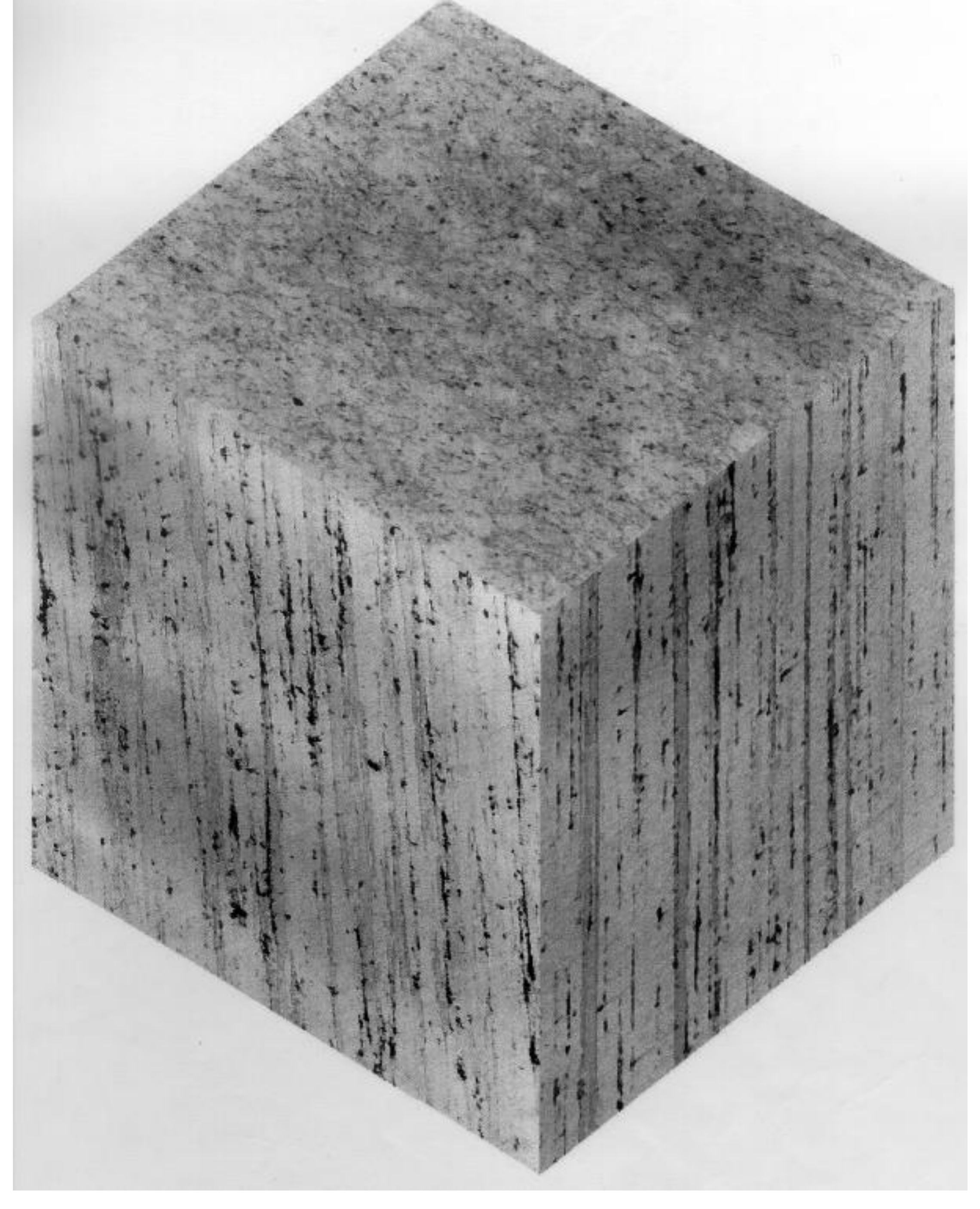
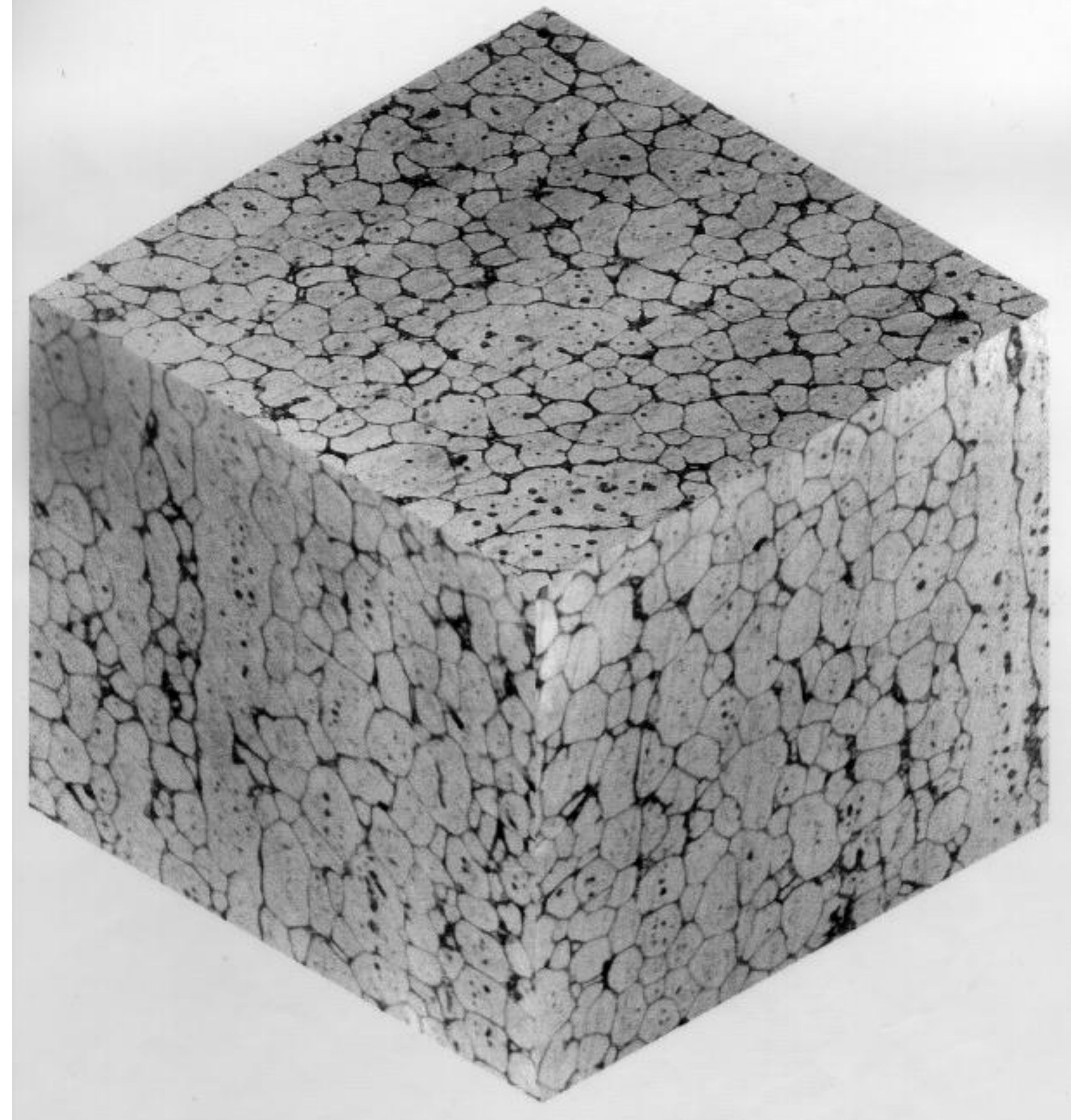


Figure 6.10 (*a*) Grain shape anisotropies induced by rolling deformation. (*b*) Optical micrographs illustrating grain shape in low-carbon steel cold-rolled to 65% in thickness. (*c*) Transmission electron micrographs reveal shape anisotropy of dislocation cells in rolled steel. (American Society for Materials, 1973, Vol. 7, p. 220).





What is Microstructure.?

- **What is Microstructure**.?
  The structure of a suitable prepared specimen as revealed by a microscope.
- 1. Microstructure
- 2. Macrostructure
- **In order to investigate structure we should preparate samples**
- **How.?**

- Introduction
  Sectioning
  Cutting the Sample
  Abrasive Cutting
  Sawing
  Fracturing
  Mounting of Specimens
  Cold Sample Mounting
  Hot Sample Mounting
  Grinding
  Coarse Grinding
  Fine Grinding
  Preliminary Polishing
  Polishing
  Final Polishing
  Mechanical Polishing

- Etching
  Electrolytic Polishing
  Plasma Etching
  Sample Cleaning
  Specimen Storage

Sample Preparation Introduction

Preparation of metallographic specimens generally requires five major operations:

1. Sectioning
2. Mounting (which is optional)
3. Grinding
4. Polishing
5. Etching

A well-prepared metallographic specimen is:

- Represent sample.
- Sectioned, ground and polished so as to minimize disturbed or flowed surface metal caused by mechanical deformation, and thus to allow the true microstructure to be revealed by etching.
- Free from polishing scratches and pits and liquid staining.
- Polished so that inclusions are preserved intact.
- Flat enough to permit examination at high magnifacation.

Sectioning

- Many metallographic specimens are used for process control. Separate test pieces or coupons attached to castings or forgings should be designed so that a minimum of sectioning is required for producing metallographic specimens.

Sectioning

- Important uses of metallography other then process control include: examination of defects that appear in finished or partly finished products and studies of parts that have failed in service. Investigations for these purposes usually require that the specimen be broken from a large mass of material, and often involve more than one sectioning operation

Sectioning

- Many metallographic studies require more than one specimen.
- For example a study of deformation in wrought metals usually requires two sections - one perpendicular to, and the other parallel to, the major axis of direction of deformation.
- Failed parts may best be studied by selecting a specimen that intersects the origin of the failure, if the origin can be identified on the surface. Depending on the type of failure, it may be necessary to take several specimes from that area of the failure and from adjacent areas.

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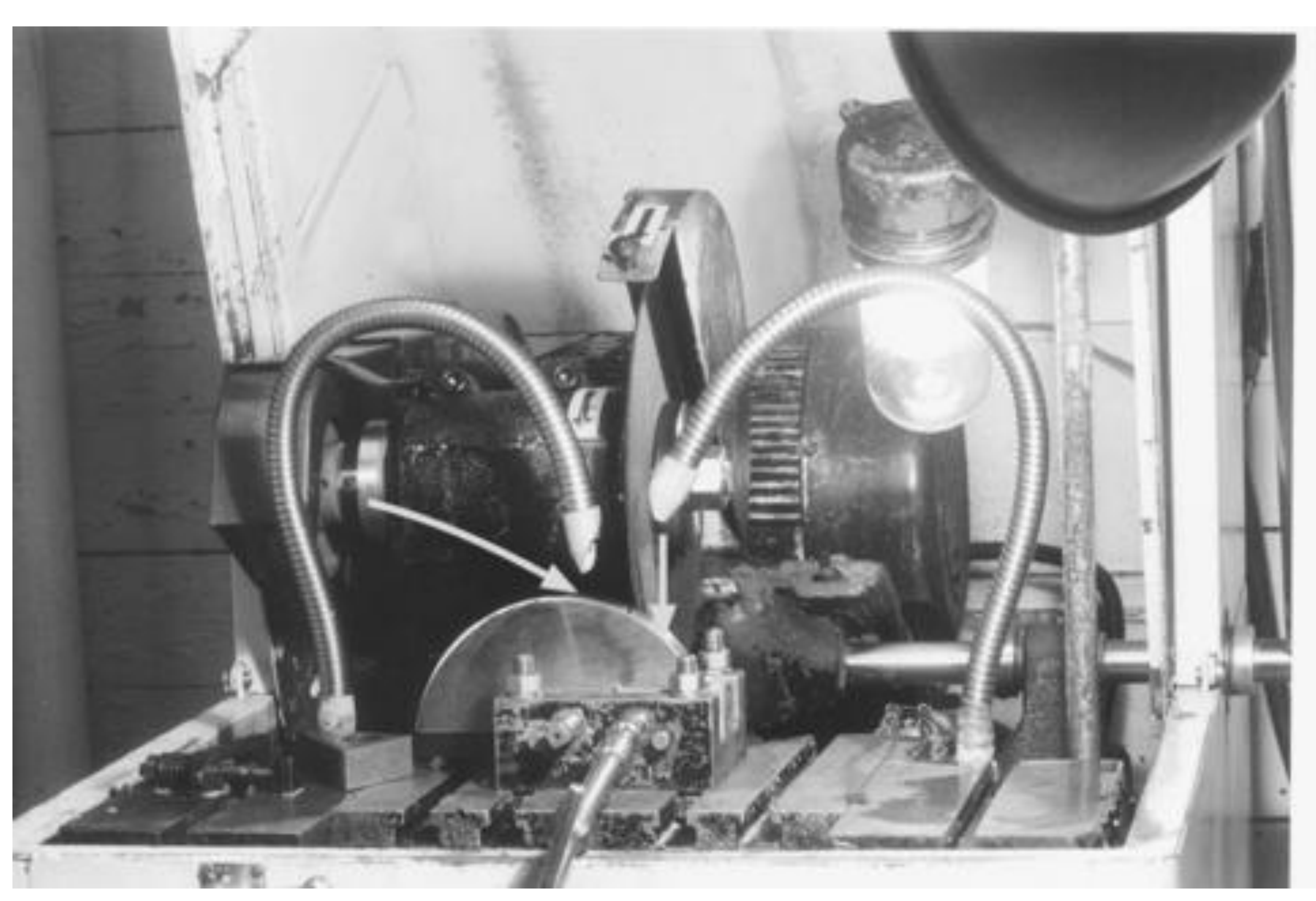
Cutting the Sample

- The first thing to remember when cutting samples, is to preserve the sample axes orientation. Cut the sample in such a manner that important sample directions, like the Rolling Direction, Transverse Direction and Sample Normal are not lost.
- The second thing to remember that the cutting process must not damage or change the sample as this would lead to erroneous results

Cutting the Sample

Fig. 2 Typical abrasive cutter. (Buehler Ltd.)



Cutting the Sample

- Avoid aggressive cutting methods that generate heat or cause deformation at the cut surface. Severe damage induced at this stage may extend so deep into the material that it is not removed by subsequent grinding and polishing.

- Heating caused during cutting may cause changes to the microstructure - phase transformations or precipitation/diffusions mechanisms may become active. Therefore heating must be avoided at all costs.

Cutting the Sample

- Abrasive cutters are common in the metallurgical industry and are suited to cutting larger sections of material.

- Often the sample is subjected to considerable force and heating, although if used with care, good cuts can be performed with minimal damage. Heating is the main problem, and using excessive force with an inappropriate wheel can cause a sample to locally glow red with heat.

Cutting the Sample

- Manufacturers of cutting equipment and cut-off wheels publish tables and diagrams to help to make the choice of wheel easier. The importance of observing the manufacturer's recommendations cannot be over-stressed. If over-heating of the sample occurs, it is usually due to using a wheel that is too 'hard' for the material being cut. The wheel does not wear properly which causes the abrasive to become blunt.

- Another possibility is that the abrasive becomes clogged. Friction then causes excessive heating and damage to the sample. Heating often results in altered surface structures, which subsequent grinding and polishing do not remove.

Cutting the Sample

- The selection of abrasive wheel is therefore important to avoid introducing unnecessary levels of damage when cutting materials.

Precision, Low Deformation Cutting Machines

- There are many examples of cutters on the market designed for precision and low damage cutting. Such machines normally employ boron nitride and diamond type cutting wheels, although other types may be available, depending on the machine.

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Abrasive-Wheel Cutting

- By far the most widely used sectioning devices in metallographic laboratories are abrasive cutoff machines. They range from small, thin-sectioning machines employing abrasive or diamond-rimmed wheels approximately 4 in. in diameter and a few mils thick to large floor-model machines employing abrasive or diamond-rimmed wheels up to 12 in. in diameter and 1/16 in. thick.
- An advanced design of automatic cutoff machine for laboratory use employs abrasive wheels 6 to 12 in. in diameter

Abrasive-Wheel Cutting

Abrasive-Wheel Cutting

- Abrasive-wheel cutting may produce deformation damage to a depth as great as 0.04 in. Deformation damage can be minimized by using thin cutoff wheels.
- A hard wheel is usually best for cutting soft stocks, whereas a soft wheel is preferred for cutting hard materials.
- A good general purpose cutoff wheel is a medium-hard silicon carbide abrasive wheel.

Abrasive-Wheel Cutting

- All abrasive-wheel sectioning should be done wet.
- An ample flow of water or water soluble oil coolant should be directed onto cut. Some laboratory cutoff machines provide for submerged wet cutting.
- Wet cutting will produce a smooth surface finish and, most important, will guard against excessive surface damage cused by overheating.

Abrasive Blade Selection Guidelines Chart

Abrasive Blade Selection Guidelines Chart

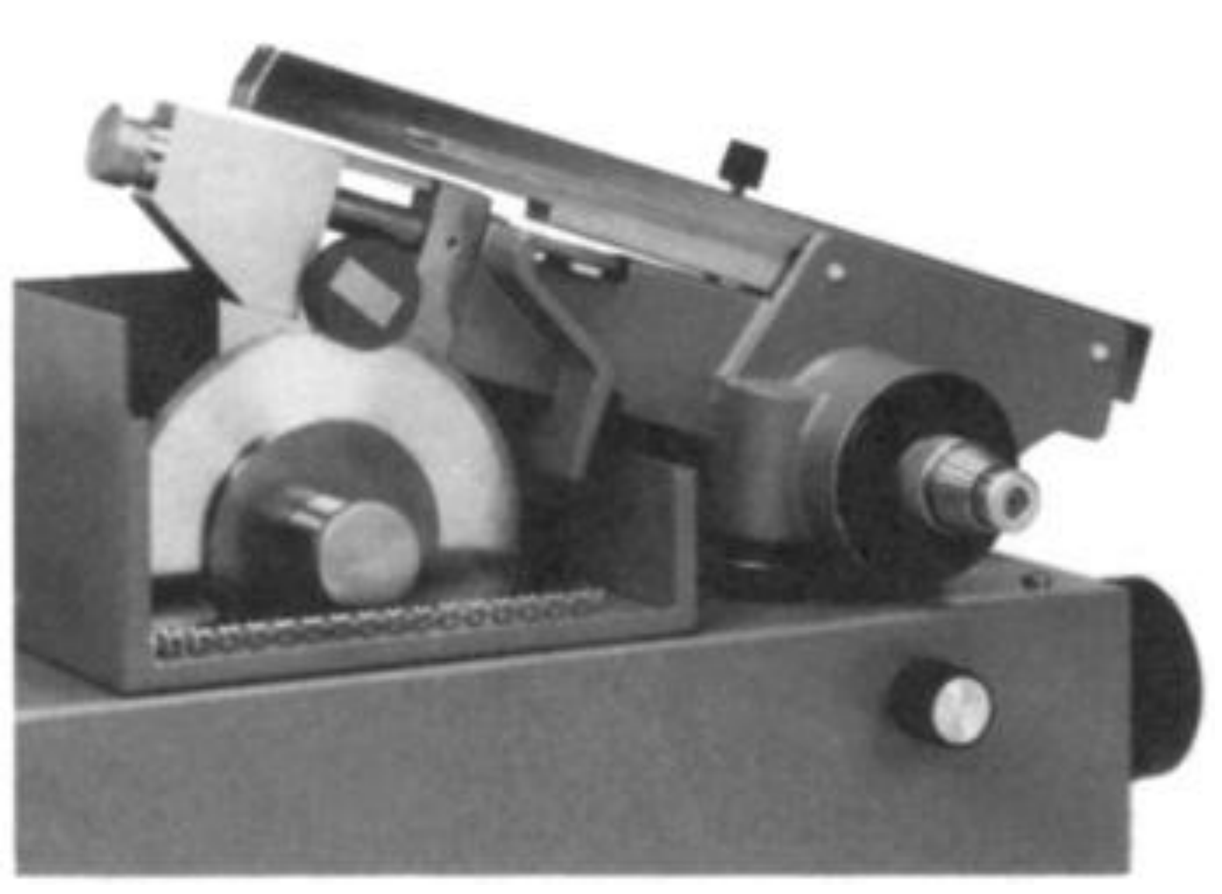
Materials (alloys)	Classification	Abrasive/Bond
Aluminum, brass, zinc, etc.	Soft non-ferrous	SiC/Rolled rubber
Heat treated alloys	Hard non-ferrous	Alumina/Rubber resin
< Rc 45 steel	Soft ferrous	Alumina/Rubber resin
> Rc 45 steel	Hard ferrous	Alumina/Rubber resin
Superalloys	High Ni-Cr alloys	SiC/Rolled rubber

Diamond Wafer Blade Selection Guidelines

Diamond Wafer Blade Selection Guidelines

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	500	250	Fine/low

Typical low-speed diamond saw



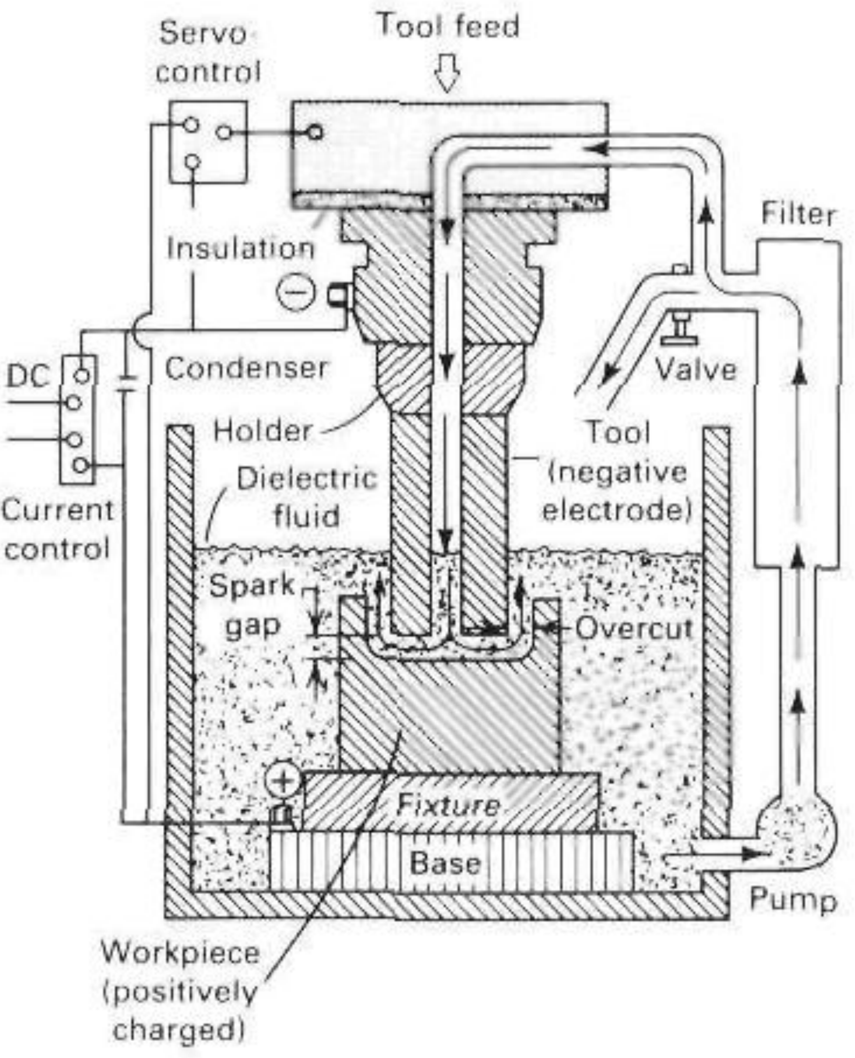
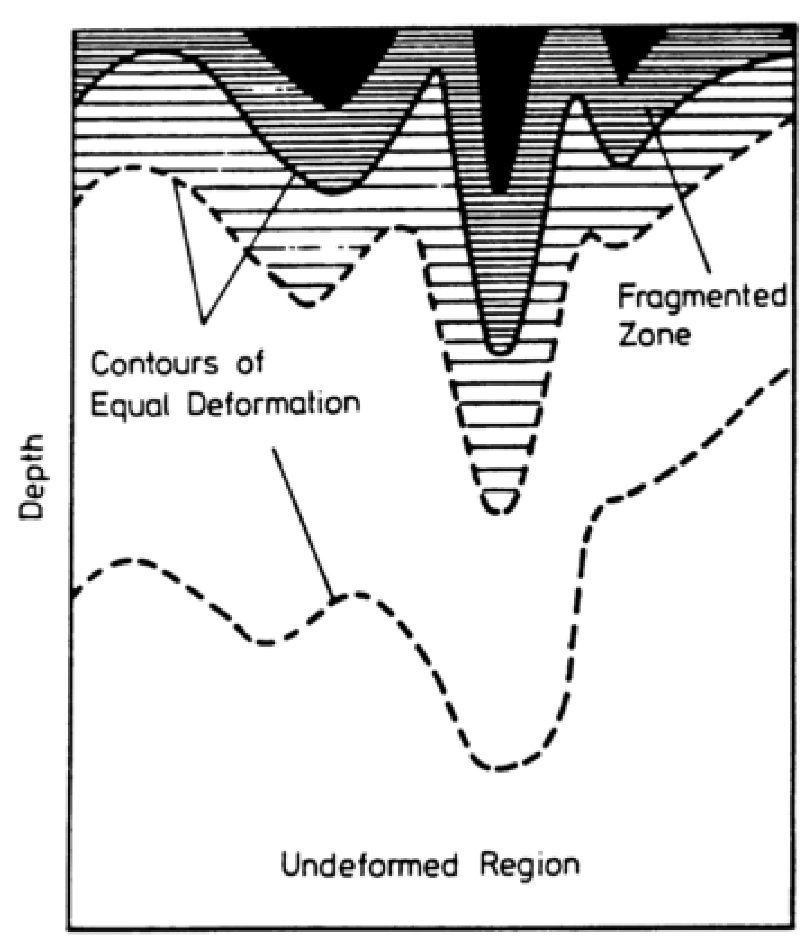
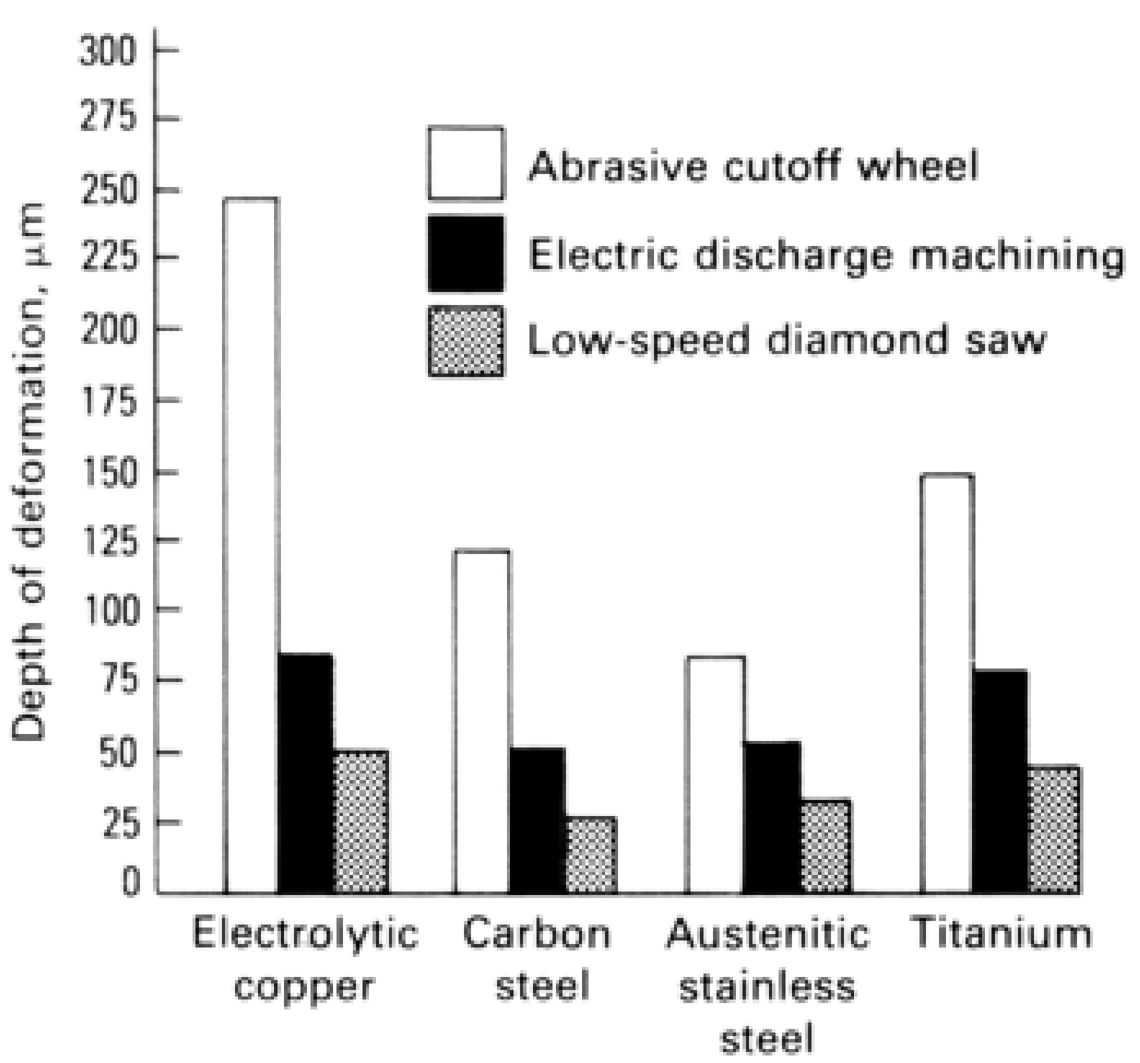


Fig. 10 Typical setup for electric discharge machining

Sketch showing depth of grinding scratches below the surface of a specimen



Depth of deformation in different metals due to cutting method.



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Sawing

- Sawing is perhaps the oldest method of sectioning in the metallographic laboratory. The method is still used today as manual hacksawing, power hacksawing, or band haksawing.
- Surface damage with sawing is primarily mechanical deformation; usually relatively little damage results from frictional heat.

Sawing

- Saw blades are generally made of hardened steel and are used to cut only materials softer than saw blade. Oil or water-soluble oil should be used as a cutting fluid to avoid premature wear of the saw teeth, as well as to minimize frictional heat, which may soften the saw teeth or alter the microstructure of the specimen below the cut surface.

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Fracturing

- Fracture surfaces may be obtained by breaking specimen with blows of a hammer or steady application of pressure. Controlled fractures may be produced by impact testing or tension testing, and the location of the fracture may be controlled by nicking or notching the material.

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Mounting of Specimens

- Mount Size and Shape
- Mounting Methods
- 1. Clamp Mounting
- 2. Compression (Hot) Mounting
- 3. Cold Mounting
- 4. Conductive Mounting

Mounting of Specimens

- The primary purpose of mounting specimens is for convenience in handling specimens of difficult shapes or sizes during the subsequent steps of preparation and examination.
- A secondary purpose is to protect and preserve extreme edges or surfaces defects during preparation.
- Specimens also may require mounting to accommodate various types of automatic devices used in laboratories or to facilitate placement on the microscope stage.

Mounting of Specimens

- An added benefit of mounting is the ease with which a mounted specimen can be identified by name, alloy number, or laboratory code number for storage by scribing the surface of the mount without damage to the specimen.

Mounting of Specimens

- Small specimens generally require mounting so that the specimen is supported in a stable medium for grinding and polishing. The medium chosen can be either a cold curing resin or a hot mounting compound.

- Characteristics of the mounting material include:

- Good abrasion characteristics and sufficient hardness such that the edges of the sample are protected, i.e., the rate at which abrasion takes place should be even across the face of the mount and the specimen.

Mounting of Specimens

- Stable and adherent to sample.
- This is important. If the mounting material has poor adhesion or high shrinkage, gaps may open up between the mounting material and the sample surface.
- When this happens, it is very difficult to prevent cross-contamination of one abrasive to another, causing heavy scratching in the finished section.
- Also any friable surface layers (oxide layers etc.) should be held adhered to the surface and not pulled off.

Mounting of Specimens

- Proper curing - insufficient time and temperature can lead to partially cured specimen mounts. Under these conditions the properties of the mounting material are not properly developed and the material may be loose and powdery.

- Generally, if the material is improperly cured, the hardness and abrasion characteristics are poor and the material is adversely affected by etches and solvents.

- Further, the characteristics under vacuum are very poor with out-gassing a major problem. If the mounting stage is suspected to be at fault, it is best to break the sample out and start again.

Mounting of Specimens

- Stable in vacuum - no out-gassing or vapour to cause contamination. This is particularly important for high magnification work, long map acquisition times and microscopes with high vacuum requirement.

Mounting of Specimens

- The mounting operation accomplishes three important functions
- (1) it protects the specimen edge and maintains the integrity of a materials surface features
- (2) fills voids in porous materials and
- (3) improves handling of irregular shaped samples, especially for automated specimen preparation.
- without damage to the specimen.

Mounting of Specimens

- The majority of metallographic specimen mounting is done by encapsulating the specimen into a compression mounting compound (thermosets - phenolics, epoxies, diallyl phthalates or thermoplastics - acrylics), casting into ambient castable mounting resins (acrylic resins, epoxy resins, and polyester resins), and gluing with a thermoplastic glues.
- An added benefit of mounting is the ease with which a mounted specimen can be identified by name, alloy number, or laboratory code number for storage by scribing the surface of the mount

Mounting of Specimens

Mount Size and Shape

- As the size of the specimen increases, so does the difficulty of keeping the specimen surface area flat during grinding and polishing. A saving in the time required for the preparation of one large metallographic specimen may be realized by sectioning the specimen into two or more smaller specimens. A specimen having an area of approximately 1/4 sq in. is perhaps the most suitable; the maximum area should be limited to about 4 sq in. if possible.

Mounting of Specimens

Mount Size and Shape

- Thickness of the mount should be sufficient to enable the operator to hold the mount firmly during grinding and polishing and thereby to pervent a rocking motion and to maintain a flat surface. Circular mounts are commonly 1 to 2 in. in diameter and are the most easily handled. The length-to-width ratio of rectangular mounts should be limited to approximately 2 to 1 to facilitate handling.

Mounting of Specimens

Mounting Methods

- The method of mounting should in no way be injurious the microstructure of the specimen. Mechanical deformation and the heat are the most likely sources of injurious effects. The mounting medium and the the specimen should be compatible with respect to hardness and abrasion resistance. A great difference in hardness or abrasion resistance between mounting media and specimen promotes differential polishing characteristics, relief, and poor edge preservation. The mounting medium should be chemically resistant to the polishing and etching solutions required for the development of the microstructure of the specimen.

Mounting of Specimens

Clamp Mounting

- Clamps are used most often for mounting thin sheets of metal when preparing metallographic cross sections. Several specimens can be clamped conveniently in sandwich form. The two clamp plates are frequently made from 1/4 in. thick steel; in general, the hardness of the clamp should be approximate or exceed the hardness of the specimen. The clamp plates are cut longer and wider than specimens to be clamped.

Mounting of Specimens

Clamp Mounting

- Then two holes are drilled and tapped in the face of one clamp plate outboard of the specimen area; corresponding holes are drilled in the other clamp plate. Machine bolts are inserted through these latter holes and into the tapped holes; the clamp plates with the specimen or specimens are drawn tightly up means of these bolts.

Mounting of Specimens

Clamp Mounting

- Sometimes, a third bolt positioned near the top of the clamp midway between the ends is useful for maintaining a uniform vertical separation between the clamp plates.
- Clamp mounting affords a means of rapid mounting, and of very good edge preservation by virtue of the initimate contact between specimens. On the other hand, hairline separations between specimens occour frequently and entrap abrasive particles or liquid solutions during preparation.

Mounting of Specimens

Clamp Mounting

- Sometimes, the particle and liquids can be removed by soaking the mount in alcohol an then thoroughly drying it. If this cannot be done, the liquid eventually seeps out and stains the polished surface, and often obscures the true microstructure after etching. One solution to this difficulty is the insertion of one thickness of transparent plastic wrapping film at each interface. (The plastic must be one that is inert to alcohol and etchants).

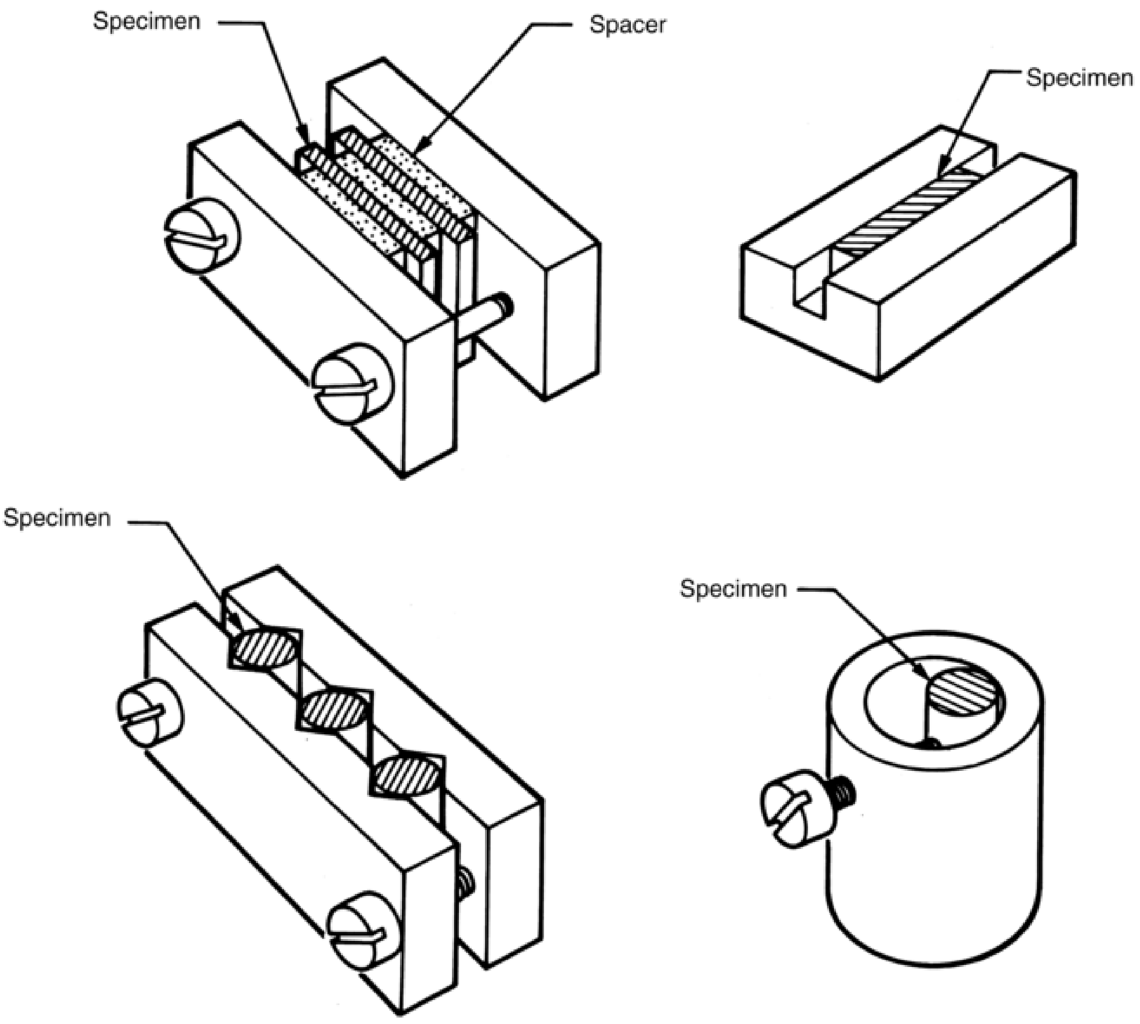
Mounting of Specimens

Clamp Mounting

- Under clamping pressure, the plastic flows readily and seals all hair-line separations. Since the film is only a fraction of a mil thick, specimen edges are preserved by adjoining specimens or clmap edges. Alternatively, soft, thin sheets of metal of the same type as that be examined can be used instead of the plastic film, or the mount can be vacuum impregnated

Mounting of Specimens

Clamp Mounting



Mounting of Specimens

Compression (Hot) Mounting

- Compression mounting, the most common mounting method, involves molding around the specimen by heat and pressure such molding materials as bakelite, diallyl phthalate resins, and acrylic resins. Bakelite and diallylic resins are thermosetting, and acrlyic resins are thermoplastic. Both thermosetting and thermoplastic materials require heat and pressure during the molding cycle, but after curing, mounts made of thermosetting materials may be ejected from the mold at maximum temperature. Thermoplastic materials remain molten at the maximum molding temperature and must cool under pressure before ejection.

Mounting of Specimens

Compression Mounting

- Mounting presses equipped with molding tools and a heater are necessary for compression mounting. Readily available molding tools for mounts having diameters of 1, 1 1/4 and 1 1/2 in. consist of a holow cylinder of hardened steel, a base plug, and a plunger. A specimen to be mounted is placed on the base plug, which is inserted in one end of the cylinder. The cylinder is nearly filled with molding material in powder form, and the plunger is inserted into open end of the cylinder.

Mounting of Specimens

Compression Mounting

- A cylindrical heater is placed around the mold assembly, which has been positioned between the platens of the mounting press. After the prescribed pressure has been exerted and maintained on the plunger to compress the molding material until it and the mold assembly have been heated to the proper temperature, the finished mount may be ejected from the mould by forcing the plunger entirely through the mold cylinder.

Mounting of Specimens

Compression Mounting

- Not all materials or specimens can be mounted in termosetting or thermoplastic mounting mediums. The heating cycle may cause changes in the microstructure, or the pressure may cause delicate specimens to collapse or deform. The size of selected specimen may be to large to be accepted by the availaible mold sizes. These difficulties are usually overcome by cold mounting.

Mounting of Specimens

Compression Mounting

- For metals, compression mounting is widely used. Phenolics are popular because they are low cost, whereas the diallyl phthalates and epoxy resins find applications where edge retention and harder mounts are required. The acrylic compression mounting compounds are used because they have excellent clarity.

Mounting of Specimens

Compression Mounting

Compression Mounting Resin Properties

Note	Phenolics	Acrylics	Epoxy (glass filled)	Diallyl Phthalates
Cost	Low	Moderate	Moderate	Moderate
Ease of use	Excellent	Moderate	Good	Good
Availability of colors	Yes	No	No	No
Cycle times	Excellent	Moderate	Good	Good
Edge retention	Fair	Good	Excellent	Excellent
Clarity	None	Excellent	None	None
Hardness	Low	Good	High	High

Mounting of Specimens

Resin	Phenolic	Acrylic	Epoxy	Diallyl Phthalate
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
Shrinkage (compression)(in/in)	0.006	-	0.001-0.003	0.001-0.003
Coefficient of Linear Thermal Expansion (in/in/°C 10^{-6})	50	-	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°-165°C (300°-330°F)	-	143°-177°C (290°-350°F)	160°-177°C (320°-350°F)
Molding pressure	21-28 Mpa (3050-4000 psi)	-	17-28 MPA (2500-4000 psi)	24-41 MPA (3500-6000 psi)
Hardness	-	Rockwell M63	Barcol 72	-
Curing time (1/2" mount @ temp.) and pressure)	90-120 seconds	2-4 minutes	90-120 seconds	90-120 seconds

Mounting of Specimens

Cold Mounting

- Cold mounting requires no pressure and little heat, and is a means of mounting large numbers of specimes more rapidly than by compression mounting.
- Materials for cold mounting are classified as polyesters, epoxides and acrylics. Polyesters are transparent and usually water clear; epoxides are almost transparent and straw color; acrylics are opaque..

Mounting of Specimens

Cold Mounting

- Cold mounting materials of all three classifications are two component systems that consist of resin and a hardener; both the resin and the hardener can be liquid, both can be solids, or one can be liquid and the other a solid. Mixing of the resin and the hardener produces exothermic polymerization, and therefore this operation is crytical in producing a satisfactory cure and limiting the temperature to a permissible level. The temperature rise may reduced at the expense of longer curing time

Mounting of Specimens

Cold Mounting

- Cold mounting is a casting method, because each of the three classifications of cold mounting materials is liquid after the resin and hardener are mixed (two-solid systems are melted before mixing). The casting molds can be of any size or shape desired. For round molds, either bakelite ring forms, or ring sections cut from plastic or metal tubes or pipes are suitable.

Mounting of Specimens

Cold Mounting

- The mold material may become part of the mount in the form of an outher shell, or mold release agents may be used to permit the mount the mount to be ejected from the mold. Rectangular molds are formed readily by wrapping heavy-duty aluminium foil around wood blocks of the desired size. The aluminium foil can be removed from the mount by peeling it away, grinding it off, or using a mold release agent. Molds any size or shape can be prepared from silicone rubber materials. The flexibility of silicone rubber molds allows cured cold mounts to be removed easily.

Mounting of Specimens

Cold Mounting

- Epoxy resins are the most widely used cold mounting materials. The are hard and adhere tenaciosly to most metalurgical, mineral and ceramic specimen. They also exhibit lower volume shinkage then either polyesters or acrylics and are very useful for impregating porous structures or cracks by vacuum method. Epoxy resin mounts may be cured in a low-temperature or placed in a low temperature oven for fast curing, depending on the mixture ratio of resin to hardener.

Mounting of Specimens

Cold Mounting

- Polyester resins have greater volume shrinkage the epoxies. They provide water-clear or slightly colored transparent mounts, which strip readily from glass casting surfaces and metal molds.
- Acrylic materials are fast curing, and the mixing and casting process for the acrylics is quick and simple. The fast curing rate results from the relatively high rate heat evolution during exothermic ploymerization, but some control of the exothermal temperature rise can be accomplished by varying the sizes of the specimen and the mount. Stripping acrylic mounts from metal od glass molds is not difficult.

Mounting of Specimens

Cold Mounting

- Castable mouting resins are commonly used for electronic and ceramic materials. Castable mounting resins are recommended for brittle and porous materials. These mounting compounds are typically two component systems (1-resin and 1-hardener). Typical curing times range from minutes to hours with the faster curing resins producing higher exothermic temperature which causes the mounting material to shrink away from the edge during curing.

Mounting of Specimens

Cold Mounting

- For example, the Acrylic Cold Mounting Resins cure in less than 10 minutes and Epoxy Castable Resins cure in approximately 4-6 hours. Note that the Epoxy Castable Resin curing cycle can be enhanced by adding an external energy source such as heat or microwave energy. It is recommended that the room temperature be less than 85° F to avoid overheating and uncontrollable curing of the mounting compound.

Mounting of Specimens

Cold Mounting

Castable Mounting Resin Properties

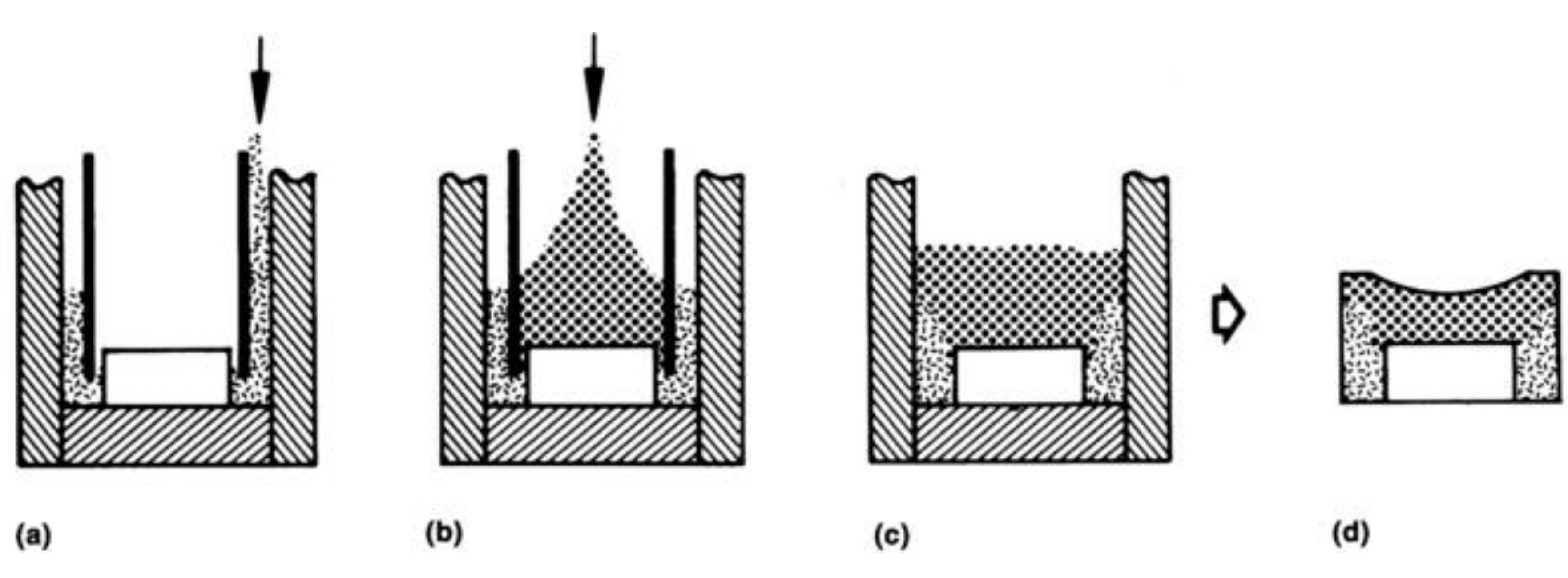
Note	EPOXY	ACRYLIC	POLYCAST Resin
Type	Epoxy resin and hardener	Acrylic resin and powder	Polyester resin and hardener
Peak Temperature	82° F	80° F	100° F
Shore D Hardness	82	80	76
Cure Time	6-8 hours	5-8 minutes	6-8 hours
Comments	Moderate hardness, low shrinkage, transparent	Very fast curt, translucent, some shrinkage	Transparent, clear

Mounting of Specimens

Conductive Mounting

- For specimens requiring metallographic preparation by electrolytic techniques, an electrically conductive mount affords a convinient means of completing the electrical circuit through the specimen; merely an electrical contact with the mount, rather than with specimen, is required. Most conductive mounting materials are mixtures of a metal, usually copper or iron powder, and thermosetting or thermoplastic molding materials.

Technique for making a mount with a conducting plastic (large dots) at the back of the specimen and a different plastic (small dots) at the section surface.



Mounting of Specimens

Conductive Mounting

- During compression mounting the metal powder particles are compacted sufficiently to provide electrical countinuity throughout the mount. An equally convenient method is to attach a copper wire to the back of the specimen and to formit an a helix to stand upright in the mounting press mold with its top in contact with the center of plunger. After ejection of the mount the free end of the helix may be dug out of the mount for electrical connection.

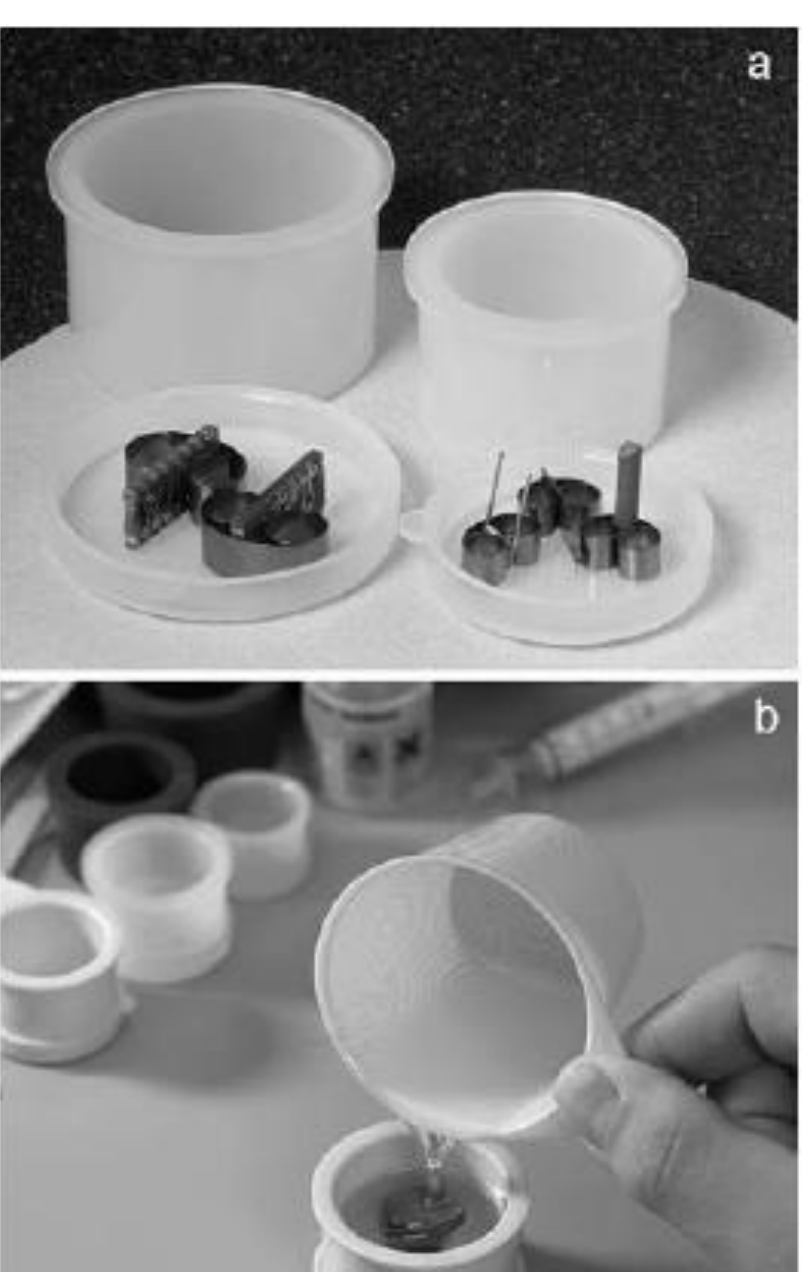
Cold Sample Mounting

- A wide range of products are available on the market. Generally faster setting products including acrylic resin types are less favorable, as these tend to develop low hardness and often suffer from 'shrinkage'. Shrinkage is the term given when the resin shrinks away from the sample surface during curing. This is undesirable as the gap which forms harbors contaminants, grit from grinding and polishing stages to cause cross contamination of polishing surfaces. It is difficult to obtain a good polished, scratch free surface when gaps in the mounting material are present.

Cold Sample Mounting

- Epoxy resin types generally have the best characteristics with respect to hardness and shrinkage. However, epoxy resins tend to be slower curing and adequate time should be allowed to ensure that the material is fully cured before proceeding. Epoxies often take a considerable period of time after initial 'setting' to develop full hardness. It is not generally possible to make cold curing resins conductive suitable for SEM examination

Cold Sample Mounting



a
b

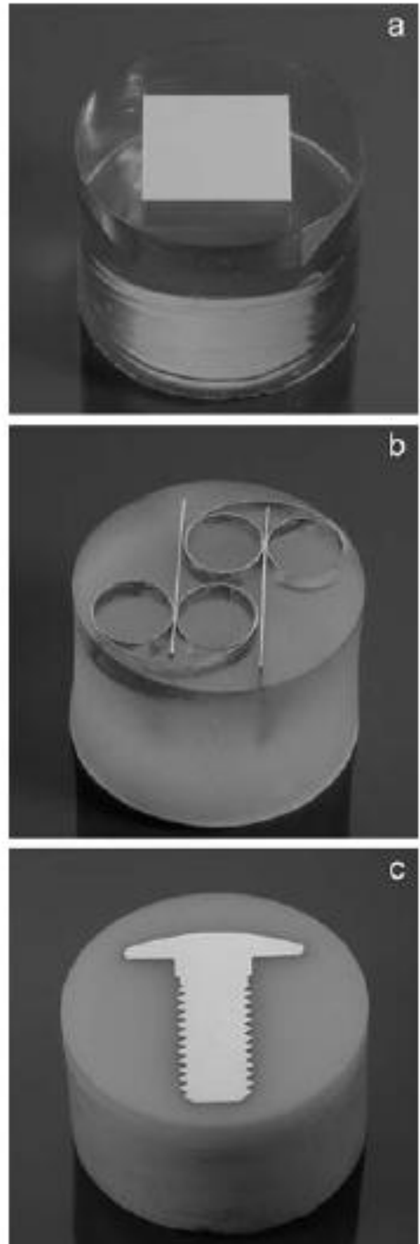
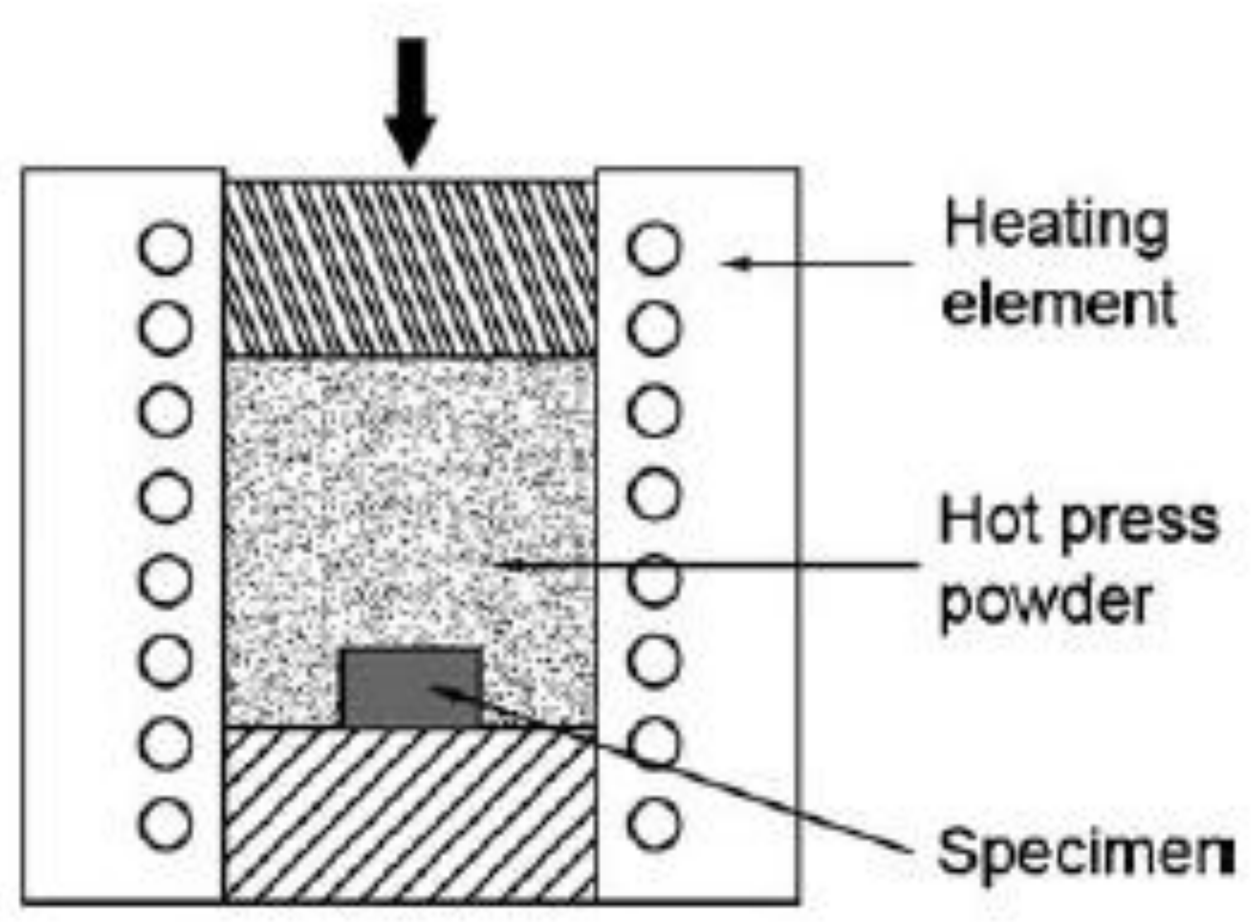


Figure 1.20 Cold mounted specimens: (a) mounted with polyester; (b) mounted with acrylic; and (c) mounted with acrylic and mineral fillers. (Reproduced with permission of Struers A/S.)

Hot Sample Mounting

- Hot mounting uses a thermosetting compound, cured in a mounting press which exerts both heat and high pressure. This mounting method produces hard mounts in a short space of time. However the heating (generally in the order of 120 °C) and considerable pressure applied may be unsuitable for delicate, soft or low melting point specimens.


Heating element
Hot press powder
Specimen

Hot Sample Mounting

Hot Sample Mounting

- Techniques may be used to protect a delicate sample from the effects of pressure, such as placing the sample under a supporting structure within the moulding cavity. Such a supporting structure can protect the sample from the initial pressure applied when the mounting material is in a granular form, and most likely to inflict damage. When the mounting material becomes fluid, infiltration should occur to encapsulate the sample which will then be subject to hydrostatic pressure.

Hot Sample Mounting

- Hydrostatic pressure can be applied to all but the most delicate of samples without problem. In the case of very soft or thermally sensitive materials, hot mounting is not appropriate. Conductive mounting resins are available, which are good for SEM examination, although the adhesion and hardness characteristics are not as good as those of epoxy hot set compounds. If the edges of the specimen are not of interest, then non conductive mounting materials can be used. In general, hot mounting is preferable to cold setting resins, when the sample is not affected by temperature and pressure (200º C & 50 kN). However, not all specimens can tolerate this.

Hot Sample Mounting

- Non-conductive mounts must be covered with adhesive conductive tape or coated with a conductive medium (the sample area can be masked if sputter coating, or using an evaporator. Aluminium foil or glass cover slips are useful for this purpose. Note: many adhesive metal tapes have non-conductive adhesive, so the use of carbon/silver conductive paint may be required at seams. Whilst very thin films of carbon can be tolerated on the sample, the ideal is that the sample surface should be bare.

Hot Sample Mounting

- Hot Mounting may be unacceptable, if the effect of temperature and pressure are expected to be inappropriate for the sample under investigation.

- Generally, the materials employed for cold setting cannot match the hardness of materials traditionally used in Hot Mounting. This may lead to compromises in the degree of edge protection and support that the mount provides for the sample. Further, the abrasion characteristics may need to be taken into account during the preparation.

Hot Sample Mounting

- The material should be stable under vacuum. Out-gassing can be a major problem leading to high contamination rates on the sample, and even microscope parts.

- Introduction
  Sectioning
  Cutting the Sample
  Abrasive Cutting
  Sawing
  Fracturing
  Mounting of Specimens
  Cold Sample Mounting
  Hot Sample Mounting
  Grinding
  Coarse Grinding
  Fine Grinding
  Preliminary Polishing
  Polishing
  Final Polishing
  Mechanical Polishing

Grinding

- Grinding Mediums
- Hand Grinding
- Belt, Disk and Surface Grinders
- Lapping
- Automatic grinding

Grinding

- Grinding is a most important operation in specimen preparation. During grinding the operator has the opportunity of minimizing mechanical surface damage that must be removed by subsequent polishing operations. Even if sectioning is done in a careless manner, resulting is severe surface damge, the damage can be elimenated by prolonged grinding. However, prolonged polishing will do little toward eliminating severe surface damage introduced by grinding.

Grinding

- Grinding is accomplished by abrading the specimen surface through a sequence of operations using progressively finer abrasive grit. Grit sizes from 40 mesh through 150 mesh are usually regarded as coarse abrasives and grit sizes from 180 mesh through 600 mesh as fine abrasives.

Grinding

- Grinding should commence with coarse grit size that will establish an initial flat surface and remove the effects of sectioning within a few minutes. An abrasive grit size 150 or 180 mesh is coarse enough to use on specimen surfaces sectioned by an abrasive cutoff wheels. Hacksawed, band sawed or other rough surfaces usually require abrasive grit sizes in the range 80 to 150 mesh. The abrasive used for each succeeding grinding operation should be one or two grit size smaller than that used in the preceeding operation. A satisfactory grinding sequence might involve grit sizes of 180, 240, 400 and 600 mesh.

Grinding

- As in abrasive-wheel sectioning, all grinding should be done wet, provided water has no adverse effects on any constituents of the microstructure. Wet grinding minimizes loading of the abrasive with metal removed from the specimen being prepared. Water flushes away most of the surface removal products before they become embedded between adjacent abrasive particles. Thus the sharp edges of the abrasive particle remain exposed to the surface of the specimen throughout the operation.

Grinding

- If the sharp edges are unexposed the result is smearing of the abraded surface rather then removal of surface metal. The operator must determine, by examining the specimen throughout the sequence of grinding steps, that the abrasive is actually cutting and not merely smearing or burnishing. Burnishing results primarily from using an abrasive beyond its effective limit. Use of worn-out abrasives and dulled cutting edges is determental to good preparation.

Grinding

- Another advantage of the wet grinding is the cooling effect of the water. Considerable frictional heat can develop at the surface of a specimen during grinding and can cuse alterations of the true microstructure - for example, tempering of martensite in steel - that cannot be removed during polishing. Wet grinding provides effective control of overheating. The abraded surface of a specimen may become embedded with loose abrasive particles during grinding. These particles may persist in the surface and appear to be nonmetallic inclusions in the polished specimen.

Grinding

- The flushing action of the water removes many of loose particles that might otherwise become embedded. Some laboratories have found that dressing the abrasive material with a solid wax lubricant recommended for grinding and other machining operations can minimize the embedding of abrasive particles.

Grinding

- The purpose of grinding is to lessen the depth of deformed metal to the point where the last vestiges of damage can be removed by series of polishing steps. The scracth depth and the depth of cold worked metal underneath the scratches decrease with decreasing particle size of abrasive. However the depth of cold worked metal is roughly inversely proportional to the hardness of the specimen and may be 10 to 50 times the depth of penetration of the abrasive particle. It is imperative that each grinding steps completely remove the deformed metal produced by the preivious step. The operator usually can assume this is accomplished if he or she grinds more than twice as long as the time required to remove the scratches incurred by the previous step.

Grinding

- To ensure the complete elimination of the previous grinding scratches found by visual inspection, the direction of grinding must be changed 45 to 90 degrees between succesive grit sizes. In addition, microscopic examination of the various ground surfaces during the grinding squence may be worthwhile in evaluating the effect of grinding. Each ground surface should have scratches that are clean-cut and uniform in size, with no evidence of previous grinding scratches.

Grinding

- Success in grinding depends in part on the pressure applied to the specimen. A very light pressure removes insufficient metal. Somewhat heavier pressure produce polishing, while still heavier pressure brings about the desired grinding action. Very heavy pressure results in nonuniform scratch size, deep gouges, and embedded abrasive particles. Generally, a medium to moderately heavy pressure applied firmly gives the best results.

Grinding

- Most grinding of metallographic specimen is performed by manually holding the specimen with its surface against a grinding material. To establish and maintain a flat surface over the entire area being ground, the operator must apply equal pressure on both sides of the specimen and avoid any rocking motion that will produce a convex surface. If grinding operation is interrupted - the operator must re-establish contact with grinding material carefully in order to resume grinding in the plane already established.

Grinding

- Specimens should be cleaned after each grinding steps to avoid any carryover of abrasive particles to the next step. Water solutions containing detergents are excellent cleaners and ultrasonic cleaning is an effective technique. Cleanness of the operator's hands is as important as cleanness of specimen. Contamination of the grinding equipment by flying abrasive particles must be avoided.

Grinding

Grinding Mediums

- The grinding abrasives commonly used in the preparation of specimens are silicon carbide (SiC), aluminium oxide (Al_2O_3), emery (Al_2O_3 - Fe_3O_4), diamond particles, etc. Usually are generally bonded to paper or cloth backing material of various weights in the form of sheets, disks and belts of various sizes. Limited use is made of grinding wheels consisting of abrasives embedded in a bonding material. The abrasive may be used also in powder form by charging the grinding surfaces with loose abrasive particles.

Grinding

Grinding Mediums

- Silicon carbide has a hardness of 9.5 on the Mohs scale, which is near the hardness of diamond. Silicon carbide abrasive particles are angular and jagged in shape and have very sharp edges and corners. Because of these characteristics, silicon carbide is very effective grinding abrasive and is preferred to other abrasives for metallographic grinding of almost all types of metal.

Grinding

Grinding Mediums

- Aluminium oxide abrasive material has a trigonal crystal structure and a hardness of 9.1 on the Mohs scale and is a synthetic corrundom.
- Emery is an impure, fine-grained variety of natural corundum containing 25 to 45 admixed iron oxide. The hardness of emery is Mohs 8.0. Emery abrasive particles have much smoother surfcaes than either silicon carbide or aluminium oxide abrasive paerticles. For this reason, emery particles do not feel as coarse as silicon carbide or aluminium oxide particles of equivalent grit size and the cutting rate of emery is inferior to that of either of the two other abrasives

Grinding

Grinding Mediums

- Another abrasive material used occasionally for grinding specimens is boron carbide, which has a hardness of nearly 10 on Mohs scale. Boron carbide is used primarily for grinding ceramic and other extremely hard materials.

Grinding

Grinding Mediums

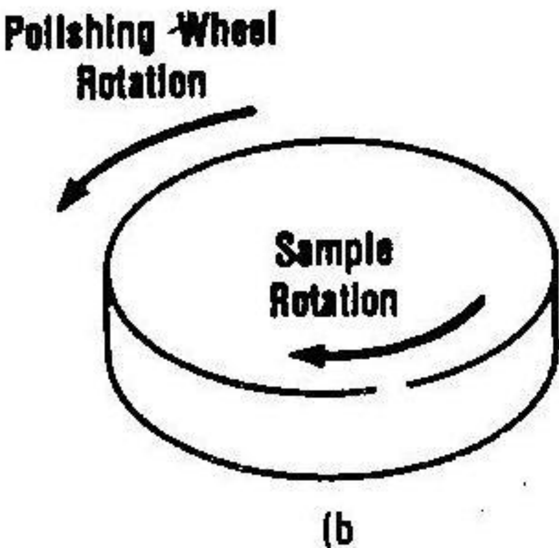
- Increasing use is being made of diamond as grinding madium as well as polishing medium. Carefully sized diamond abrasive particles are available from 280 microns (about 60 mesh) to 0.25 microns in size. The coaser grades of diamond are used in the form of resin-bonded cloth-backed disks, metal bonded lapping surfaces, and loose particles for charging of grinding surfaces.

Grinding

Grinding Mediums

- Diamond abrasives of all sizes are available as suspensions in oil-soluble and water-soluble paste vechicles known as diamond compounds. The extreme hardness (Mosh 10) and sharp cutting edges of diamond particles impart at high cutting rate to diamond abrasives. Diamond abrasives are particularly suitable for grinding the harder alloys and refractory materials.

Grinding



BUEHLER

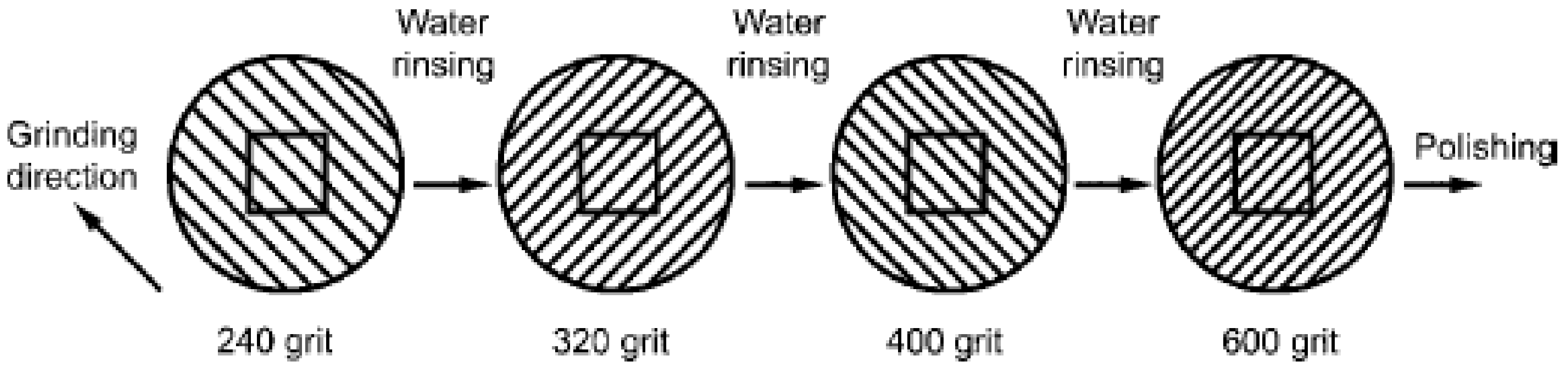


Figure 1.22 Hand grinding procedure.

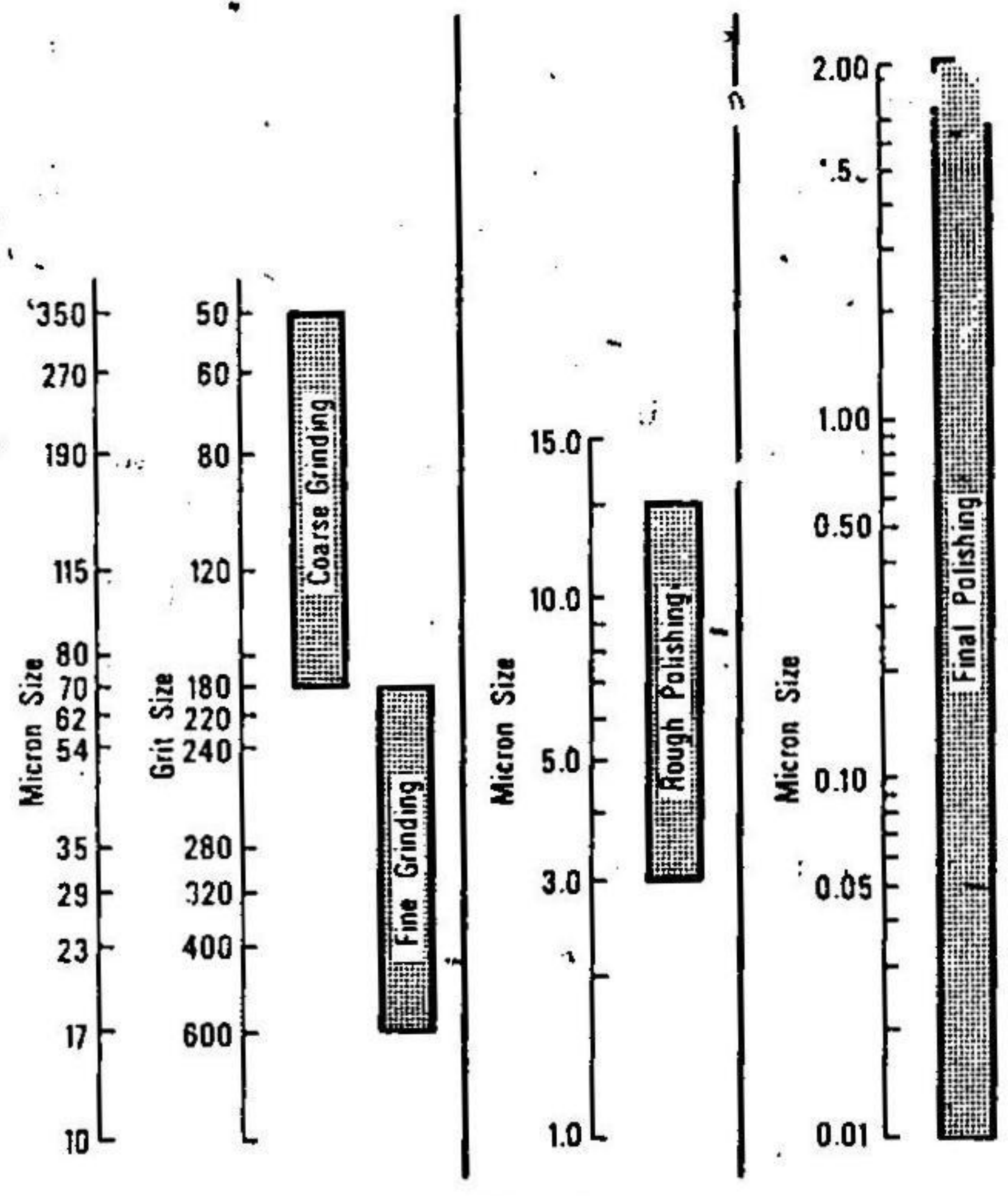


Fig. 7

Table I

SILICON CARBIDE, ALUMINUM OXIDE, GARNET

	Particle Size (AV. μm)			
Grit No.	USA	FEPA*		
	Sieve Series	Commercial	F-Series	P-Series
50	350	324		
60	270	274		
80	190	194		
120	115	115		
150	80	97		
180	70	81		
220	62	58		
240	54	49	49.3	58.5
280	35	41	41.5	52.2
320	29	34	34.4	46.2
400	23	29	23.0	35.0
600	17	20	14.3	22.8

*Federation Europeane des Fabricants des Produits Abrasifs

Table III.
DIAMOND PASTES

Micron Size* (µm)	Size Range (µm)	Mesh Equivalent (Approx.)
1/10**	0-1/0	....
1/4**	0-1/2	100,000
1/2	0-1	60,000
1	0-2	14,000
3	2-4	8,000
6	4-8	3,000
9	8-12	1,800
15	12-[illegible]	[illegible]
30	22-30	[illegible]
45	36-54	32[illegible]
60	54-80	[illegible]30 - 325
90***	....	170 - 230

* National Bureau Standards
** Ultra fine grades, not covered by NBS
*** ANSI B 74.16 - 1971, not covered by NBS

Table II.
EMERY

Grit No.	Particle Size (Av. µm)
	85
	70
1	50
1/0	33
2/0	30
[illegible]	28
4/0	25

Table IV.

A. POLISHING POWDERS

Type	Particle Size (μm)	Application
Gamma Alumina	0.05	Final Polishing
Alpha Alumina	0.3	Final Polishing
Alpha Alumina	1.0	Rough and/or Final Polishing
Magnesium Oxide	2.0	Final Polishing

B. POLISHING SUSPENSIONS

Type	Particle Size (μm)	Application
Levigated Alumina	[illegible]	[illegible] Polishing
Gamma Alumina	[illegible]	Final Polishing
Alpha Alumina	0.3	Rough and/or Final Polishing
Chrome Oxide	[illegible]	Rough Polishing
Chrome Oxide	[illegible]	Final Polishing
Chrome Oxide*	0.05	Final Polishing
Cerium Oxide**	0.05	Final Polishing

* Trade Name CRO,
Chrome Oxide / Cerium Oxide Blend

** Trade Name Finish-Pol,
Cerium Oxide / Aluminum Oxide Blend

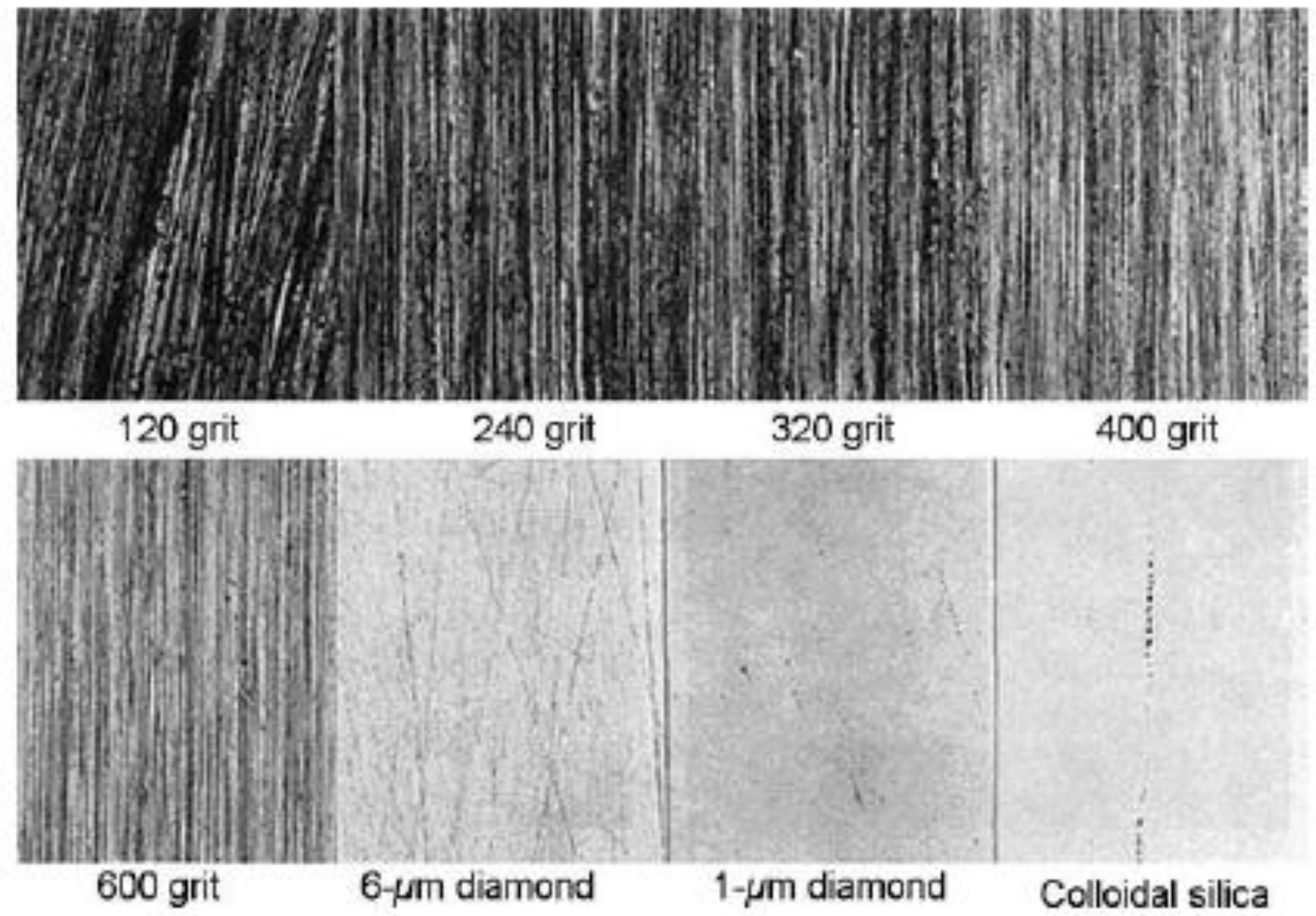


Figure 1.23 Sample of specimen surfaces after grinding and polishing with abrasives of different grits and size. (Reproduced with permission of ASM International®. All Rights Reserved. www.asminternational.org. G.F. Vander Voort, *Metallography Principles and Practice*, McGraw-Hill, New York. © 1984 ASM International®.)

Figure 1.24 Polishing on a rotating wheel with a mechanical sample holder. (Reproduced with permission of Struers A/S.)

Grinding

Hand Grinding

- Manual Preparation - In order to insure that the previous rough grinding damage is removed when grinding by hand, the specimen should be rotated 90 or 45 degrees and continually ground until all the scratches from the previous grinding direction are removed. If necessary the abrasive paper can be replace with a newer paper to increase cutting rates.

Grinding

Hand Grinding

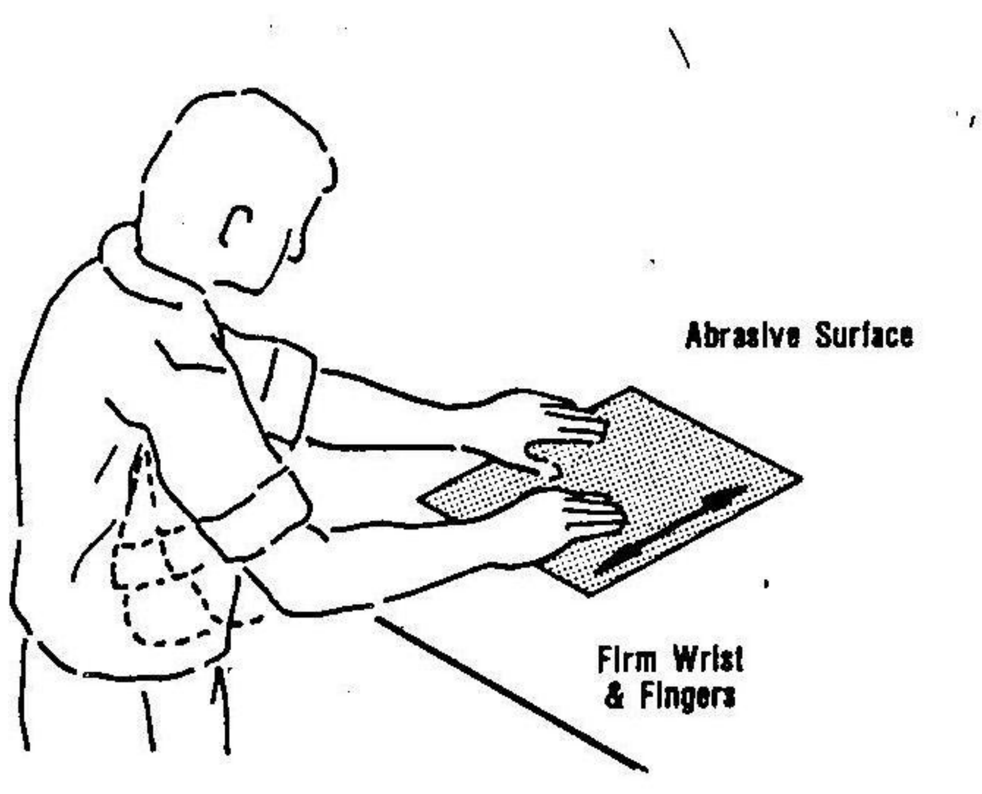


Fig. 11

Grinding

Hand Grinding

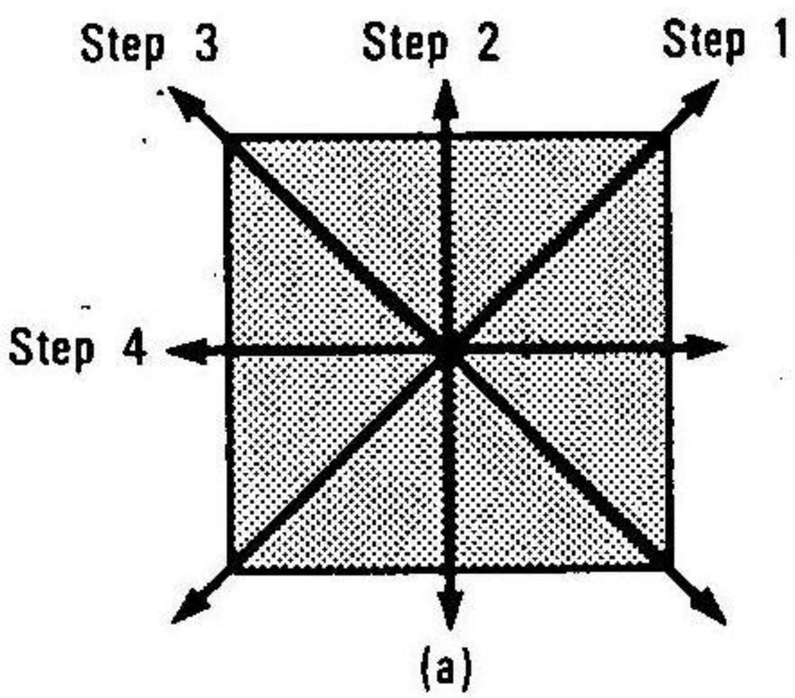


Fig. 12A

Grinding

Hand Grinding

- A simple setup for hand grinding can be provided by a piece of plate glass, or other material with hard, flat surface, on which an abrasive sheet rests. The specimen is held by hand against the abrasive sheet as the operator moves specimen in rhytmic style away from and toward him in a straight line. Heavier pressure should be applied on the forward stroke than on the return stroke. The grinding can be done wet by sloping the plate glass surface toward the operator and providing a copious flow of water over the abrasive sheet.

Grinding

Hand Grinding

- Planar Grinding - or course grinding is required to planarize the specimen and to reduce the damage created by sectioning. The planar grinding step is accomplished by decreasing the abrasive grit/ particle size sequentially to obtain surface finishes that are ready for polishing. Care must be taken to avoid being too abrasive in this step, and actually creating greater specimen damage than produced during cutting (this is especially true for very brittle materials such as silicon).

Grinding

Hand Grinding

- The machine parameters which effect the preparation of metallographic specimens includes: grinding/polishing pressure, relative velocity distribution, and the direction of grinding/polishing.

Grinding

Hand Grinding

- 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/m2 or Pa). For specimens significantly harder than the mounting compound, pressure is better defined as the force divided by the specimen surface area. Thus, for larger hard specimens higher grinding/polishing pressures increase stock removal rates, however higher pressure also increases the amount of surface and subsurface damage. Note for SiC grinding papers, as the abrasive grains dull and cut rates decrease, increasing grinding pressures can extend the life of the SiC paper.

Grinding

Hand Grinding

- 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.

Grinding

Hand Grinding

- Relative Velocity - Current grinding/polishing machines are designed with the specimens mounted in a disk holder and machined on a disk platen surface. This disk on disk rotation allows for a variable velocity distribution depending upon the head speed relative to the base speed.

Grinding

Hand Grinding

Head Speed (rpm)	Base Speed (rpm)	Relative Velocity Distribution	Characteristic	Application
150	300 to 600	High	• Aggressive stock removal • Differential grinding across the specimen surface	• Useful for gross removal on hard specimens
150	150	Minimal	• Matching head and base speed in the same direction eliminates relative velocity distributions • Uniform stock removal • Low stock removal • Produces minimal damage	• Provides superior flatness over the specimen • Useful for retaining inclusions and brittle phases

Grinding
Hand Grinding

- For high stock removal, a slower head speed relative to a higher base speed produces the most aggressive grinding/ polishing operation. The drawback to high velocity distributions is that the abrasive (especially SiC papers) may not breakdown uniformly, this can result in non-uniform removal across the specimen surface. Another disadvantage is that the high velocity distributions can create substantially more specimen damage, especially in brittle phases. In all cases, it is not recommended to have the head rotating contra direction to the base because of the non-uniform removal and abrasive break-down which occurs.

Grinding

Hand Grinding

- Minimal relative velocity distributions can be obtained by rotating the head specimen disk at the same rpm and same direction as the base platen. This condition is best for retaining inclusions and brittle phases as well as for obtaining a uniform finish across the entire specimen. The disadvantage to low relative velocity distributions is that stock removal rates can be quite low.

Grinding

Hand Grinding

- In practice, a combination of a high velocity distribution (150 rpm head speed/300 - 600 rpm base speed) for the initial planarization or stock removal step, followed by a moderate speed and low velocity distribution (120-150 rpm head speed/150 rpm base speed) step is recommended for producing relatively flat specimens. For final polishing under chemical mechanical polishing (CMP) conditions where frictional heat can enhance the chemical process, 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).

Grinding

Hand Grinding

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

Grinding

Hand Grinding

Grinding

Grit Number		
European (P-grade)	Standard grit	Median Diameter, (microns)
60	60	250
80	80	180
100	100	150
120	120	106
150	150	90
180	180	75
220	220	63
P240	240	58.5
P280		52.2
P320	280	46.2
P360	320	40.5
P400		35
P500	360	30.2
P600	400	25.75
P800		21.8
P1000	500	18.3
P1200	600	15.3
P2400	800	6.5
P4000	1200	2.5

Planar Grinding Recommendations
Metallic Specimens

- For metallic specimen grinding, sequential grinding with silicon carbide (SiC) abrasive paper is the most efficient and economical rough grinding process. Although extremely coarse grit abrasive papers can be found, it is recommended that a properly cut specimen not be rough ground with an abrasive greater than 120 grit SiC paper. A typical abrasive grinding procedure would consist of 120 or 240 grit SiC paper followed by decreasing the size of the SiC paper (320, 400, and 600 grit). Finer papers are also available for continued abrasive paper grinding (800 and 1200 grit).

Planar Grinding Recommendations
Metallic Specimens

- In addition to the correct sequence and abrasive size selection, the grinding parameters such as grinding direction, load and speed can affect the specimen flatness and the depth of damage.
- The basic idea is to remove all of the previous specimen damage before continuing to the next step while maintaining planar specimens.

Planar Grinding Recommendations
Electronic Specimens

- Grinding electronic components is very dependent upon both the substrate (silicon, alumina, barium titanate, plastic PCB's, etc) and the metallic materials used. In general, when grinding plated or coated materials, it is recommended that the coating be prepared in compression to prevent the coating from separating from the substrate.

Planar Grinding Recommendations
Electronic Specimens

- Silicon specimens should be have been sectioned with a fine grit diamond blade and cut as near as possible to the area of interest. For rough grinding, fine abrasives such as 400 or 600 grit SiC or a 15 micron diamond lapping film is recommended to avoid producing more damage than created during sectioning.

Planar Grinding Recommendations

Electronic Specimens

- Hard ceramic substrates (especially porous materials) should be rough polished with diamond lapping films to minimize edge rounding and relief between the widely varying materials hardness'.

Planar Grinding Recommendations
Plasma Spray Components

- Similar to the preparation of electronic components, plasma spray coatings should be kept in compression to minimize the possibility of delamination at the coating/ substrate interface. For ceramic plasma spray coatings, diamond lapping films are recommended to minmize edge rounding or relief and to maintain the integrity of any inclusions within the coating

Planar Grinding Recommendations

Ceramics and Ceramics Composites

- Rough grinding ceramics and ceramic matrix composites should be performed with 15 or 30 micron diamond on a metal mesh cloth in order to obtain adequate stock removal and to minimize surface and subsurface damage

Planar Grinding Recommendations
Plastics and Plastics Composites

- Plastics are generally very soft and therefore can be planar ground with sequentially decreasing SiC abrasive paper grit sizess. When plastics are used in conjunction with hard ceramics, planar grinding can be very tricky. For these composite materials cutting must minimize damage as much as possible because almost all grinding and polishing will cause relief between the soft plastic and the hard ceramic. Following proper cutting, grinding with as small as possible a diamond (6 micron diamond) on a metal steel mesh cloth or the use of lapping films is suggested

Grinding

Belt, Disk and Surface Grinders

- The most common types of motor-driven grinding equipment are the belt grinder and the disk grinder. In using either, the metallographic specimen is held by hand against a moving, fixed-abrasive grinding material supported by a platen. Belt grinders and disk grinders may be used in either a horizontal or vertical position. Abrasive belts are generally cloth-backed for strength, and the popular belt sizes are 4 by 36 in. and 4 by 54 in. Although cloth-backed disks are available, paper-backed disk are preferred for disk grinding of metallographic specimens, because paper-backed disks rest flat against the platen whereas cloth-backed disks usually curl in form the edge. Most metallographic grinding disks are 8 or 10 in. in diameter. Specimens can be belt or disk ground successfully through all grinding sequences

Grinding

Lapping

- Is a grinding technique similar to disk grinding. The grinding surface (lap) is a rotating disk whose working surface is charged with a small amount of a hard abrasive material. Laps are made of wood, lead, nylon, paraffin, paper, leather, cast iron and laminated plastics. The abrasive charge may be pressed into lap material by means of a steel block, or the lap may be charged directly with a mixture of abrasive and destilled water during lapping. A groove in the form of a spiral is a direction counter to the lap rotation is often cut in the surface of laps, particularly of lead and paraffin laps. The spiral groove aids retention of cooling water and abrasive.

Grinding

Automatic grinding

- As the name implies, is done without hand assistance. All automatic grinding devices use lap surfaces on which paper-backed disks are placed or abrasive pawder is charged. The lap is either a rotating or a vibarating disk. Use of a latter is described as vibratory grinding. The technique and equipment for automatic grinding are analogous to those described uder Automatic Polishing.

Grinding

Automatic grinding

- The key to successful automated preparation is to thoroughly clean the specimens between each abrasive grit size used. The tracking of the specimens should also uniformly break down the SiC paper, otherwise non-uniform grinding will occur (especially for hard specimens in soft mounts). In other words, the specimen should track across the entire diameter of the SiC paper.

Coarse Grinding

- Grinding can be achieved in a variety of ways, using a variety of abrasives. Fixed abrasive surfaces are available using diamond or cubic boron nitride (CBN) abrasives. The method used to bind the abrasives to the wheel affects the grinding characteristics, the harder or more rigid the bonding medium, the more aggressive the grinding action of the surface. Therefore metal bonded fixed abrasive wheels are the most aggressive grinding surfaces, whereas resin bonded wheels are less aggressive.

  Coarse grit Silicon Carbide or Alumina abrasives may be used, but the durability or characteristics of such materials may be inappropriate for certain materials. Generally, in order to maintain sharp abrasive particles, grinding papers need frequent changing. Follow the manufacturer's recommendations and advice.

Coarse Grinding

Fine Grinding

- Silicon Carbide (SiC) paper is the traditional method used for fine grinding and is adequate when used properly. SiC paper blunts quickly and therefore should be discarded after a short period of grinding in order to maintain efficient 'stock' removal. Grinding on a surface that has blunt abrasives causes a great deal of surface damage by smearing, 'burnishing' and local heating.

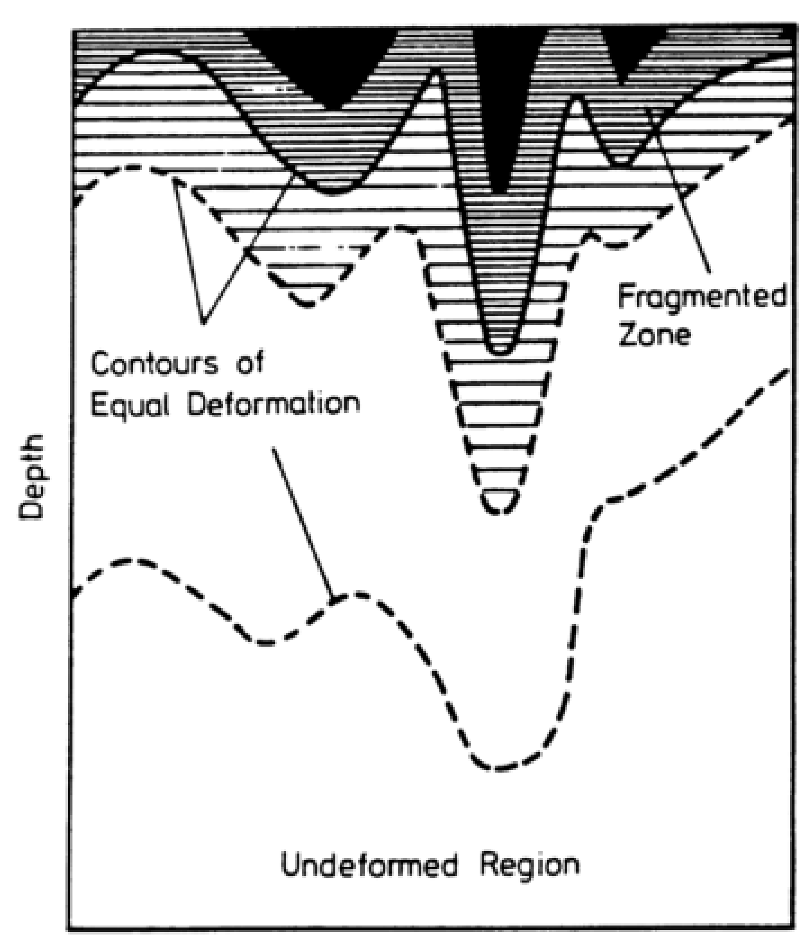
Fine Grinding

- Ensure that sharp abrasives are used and follow the manufacturers instructions with regard to grinding speeds, direction, force, times and lubricants used. Damage injected during grinding may be invisible in the polished surface. Remember that different materials have different abrasion characteristics. The selection of grinding material and conditions can therefore be specific to a given sample.

- After every grinding stage it is advisable to inspect the ground surface using a light microscope in order to ensure that all damage from the previous stage, whether that be a cutting or grinding stage, is completely removed. Advance in this manner to the finest abrasive size required, ready for polishing. Care at this stage will greatly reduce the amount of polishing required to achieve a good surface

Fine Grinding

Sketch showing depth of grinding scratches below the surface of a specimen



Preliminary Polishing

- Diamond polishing compounds or slurries are good for preliminary stages for most materials. Polishing is a similar action to grinding, accept that the supporting medium used to hold the abrasive is capable far greater 'shock absorbency' i.e. the ability of the medium to allow the abrasive to move to some degree and conform to the surface aspirates of the specimen. Thus different polishing surface materials have differing characteristics: soft cloths allow the greatest shock absorbency and therefore allow for gentle polishing with little damage associated.

Preliminary Polishing

- However soft cloths allow the abrasive to abrade different areas at different rates, giving rise to 'relief'. 'Relief' is the term used to describe the undulations that form in a polished surface. Extreme undulations or relief in the polished surface is to be avoided, although a certain amount can be tolerated (or even desirable) because the SEM generally has high depth of field. Harder polishing surfaces or cloths, conversely, produce a flatter or 'plane' surface, but may leave polishing damage in the surface of the material, and promote superficial scratching.

Preliminary Polishing

- Therefore, it is usually the case that polishing is started on a hard cloth with a coarser abrasive and finished on a softer cloth with a finer abrasive. Final polishing should not be prolonged, but just sufficient to achieve the desired surface finish without causing excessive relief

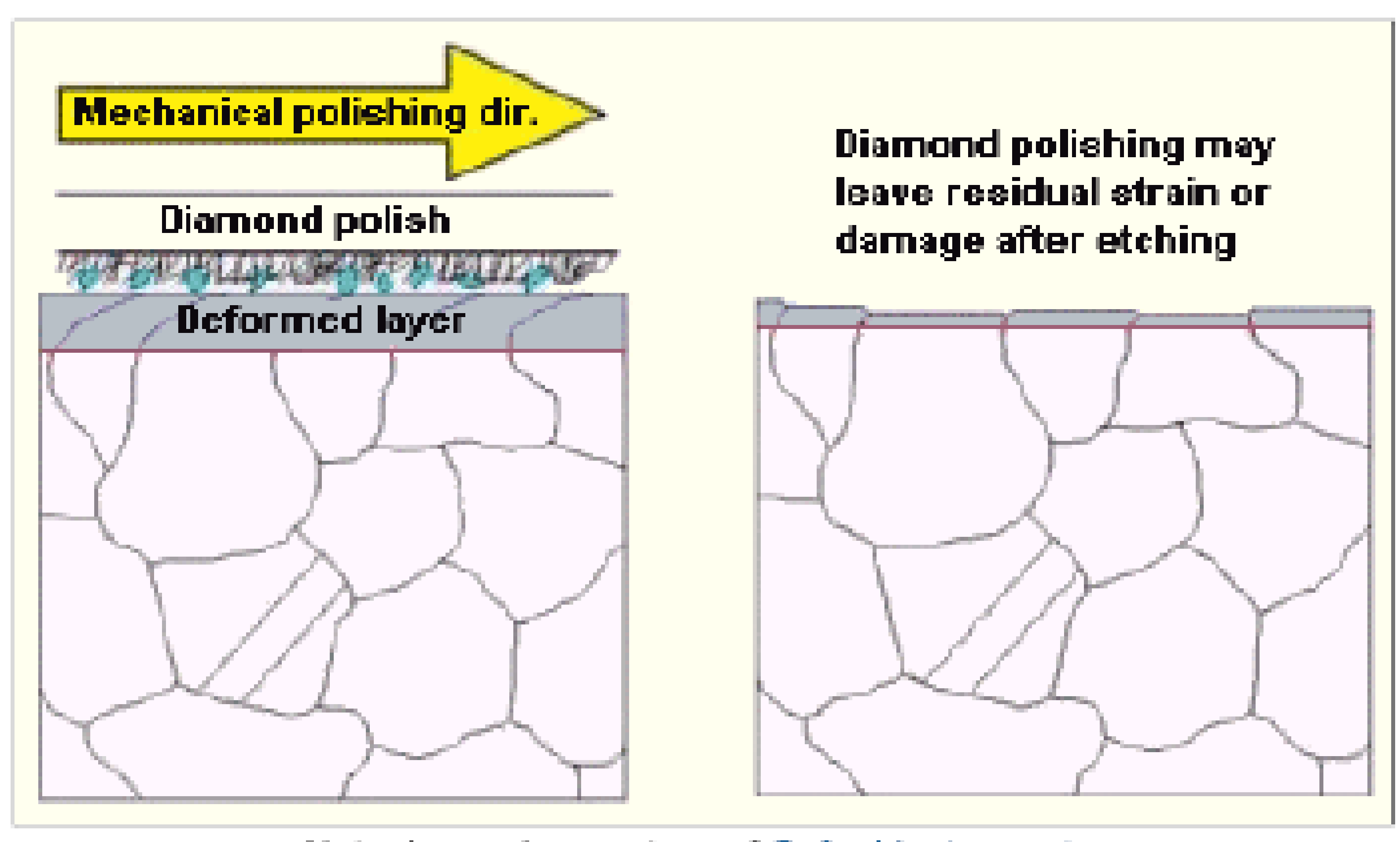
Sample Preparation
Polishing

- Polishing is the final step in production a surface that is flat, scratch free, and mirror like in appearance. Such a surface is necessary for subsequent accurate metallographic interpretation, both qualitative and quantitative. The polishing technique used should not introduce extraneous structure such as disturbed metal, pitting, dragging out of inclusions, comet tails and staining.
- Before final polishing is started, the surface condition should be at least as good that obtained by grinding with a 400-grit (25 microns) abrasive.

Final Polishing

- For sample preparation, it is generally necessary to use an additional final polishing stage using colloidal silica. Final polishing should not be prolonged, but just sufficient to achieve the desired surface finish without causing excessive relief.

Final Polishing



Final Polishing

- Colloidal Silica is a chemo-mechanical polish, i.e., it combines the effect of mechanical polishing with etching. This type of stock removal is ideal in many cases, as a damage free surface can be obtained with little effort. Typical abrasive size is 0.05 micron. Note: Colloidal Silica crystallizes readily and will ruin polishing cloths if left to dry. Further, a film can form on the polished surface of the sample which must be removed. A convenient method to achieve this is to flush the polishing wheel with water during the last few seconds of polishing to clean the sample surface.

Final Polishing

- Remove and dry the sample in the usual manner, using a solvent with low water content and not so volatile as to cause water condensation on the surface. Alcohol is ideal, whereas acetone is not. Flush the polishing wheel with water until all traces of colloidal silica is washed away, spin to drain and store in a suitable container such that contamination of the wheel cannot occur. Meticulous attention to avoiding contamination of wheels is an important aspect to achieve the best results

Sample Preparation
Mechanical Polishing

- Hand Polishing
- Specimen Movement
- Polishing Pressure
- Washing and Drying
- Cleanness
- Automatic Polishing
- Polishing Cloths
- Polishing Abrasives

Sample Preparation
Mechanical Polishing

- The term mechanical polishing is frequently used to describe the various final polishing procedures involving the use of cloth-covered laps and suitable polishing abrasives. The laps have either a rotating or a vibrating motion, and the specimen are held by hand, held mechanically, or merely confined within the polishing area.

Sample Preparation
Mechanical Polishing

- Polishing be done in a relatively dust-free area, preferably removed from the area for sectioning, mounting and rough grinding. Any contamination of a polishing lap by abrasive particles carried over from a preceding operation or by dust, dirt or other foreign matter in the air cannot be tolerated. Carryover as a result of improper cleaning between final polishing steps is another prime source of contamination. It is just as important for the operator to wash his hand meticulously as is for him to remove all traces of polishing abrasive from the specimen before proceeding to the next polishing operation.

Sample Preparation
Mechanical Polishing

- The specimen can be cleaned ultrasonically or by careful washing under running water and swabbing with cotton. In automatic equipment in which holding fixtures for the specimens are also transferred through successive polishing steps, proper cleaning of the assembly can be accomplished only by using an ultrasonic cleaner.

Sample Preparation
Mechanical Polishing

- If a polishing lap becomes contaminated, it is virtually impossible to remove all of the contaminant by washing the polishing cloth. Instead, the operator should replace the cloth and use fresh polishing solution. Cleanness cannot be overemphasized. It takes only one particle of grit on a final polishing lap to ruin all prior preparation.

Sample Preparation
Hand Polishing

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

Sample Preparation
Hand Polishing

- **Specimen Movement**
  The specimen is held with one or both hands, depending on the operator's preference, and is rotated in a direction counter to the rotation of the polishing wheel. In addition, the specimen is continually moved back and forth between the center and the edge of the wheel, thereby ensuring even distribution of the abrasive and uniform wear of the polishing cloth. The main reason for rotating the specimen is to prevent the formation of "comet tails“.

Sample Preparation
Hand Polishing

- **Polishing Pressure**
  The correct amount of applied pressure must be determined by experience; in general, firm hand pressure is applied to the specimen in the initial polishing step and is proportionally decreased with successively finer polishing steps. For very soft materials, pressure other than that from the weight of the specimen itself may be eliminated entirely in the last polishing operation.

Sample Preparation
Hand Polishing

- **Washing and Drying**
  The specimen is preferably washed and swabbed in worm running water, rinsed with methanol or any other alcohol that does not leave a residue, and dried in a stream of warm air. Alcohol can usually be employed for washing when the abrasive carrier is not soluble in water.

Sample Preparation
Hand Polishing

- **Cleanness**
  The precautions for cleanness, as previously mentioned, must be strickly followed.

Sample Preparation

Automatic Polishing

- High quality preparation of most metallographic specimens often can be expedited by the use of automatic polishers. Automatic polishing equipment usually allows the preparation of several specimens simultaneously. Some methods of specimen preparation can be done only with automatic polishers, such as remote polishing of radioactive materials, chemical-mechanical polishing, and polishing in special atmospheres. There is no ideal automatic polisher; each has its merits and shortcomings and each metallographer must determine which is best for his particular requirements.

Sample Preparation

Polishing Cloths

- The requirements of any good polishing cloth include the ability to hold an abrasive, long life, absence of any foreign material that may cause scratches, and absence of any processing chemicals that may react with the specimen

Sample Preparation

Polishing Cloths

- A cloth without nap or with a very low nap is preferred for the preliminary or rough polishing operation. The absence of nap ensures maximum contact with the polishing abrasive, and results in fast cutting with minimum of relief. The cloths most frequently used are canvas, low-nap, cotton, nylon, silk and Pelon. These cloths are stretched tight on the laps and fastened securely, usually by a band-type clamp. Some cloths are available with a contact adhesive on the back, which greatly simplifies installation on the wheel.

Sample Preparation
Polishing Cloths

- After installation, the cloths are charged with the appropriate abrasive (usually in sizes from 15 microns down to 1 microns) and carrier. Rough polishing is usually done with the laps rotating at 500 to 600 rpm. Cloths with a medium or high nap are ordinarily used on slow rotating laps (less than 300 rpm) for intermediate and final polishing. Felt or billiard cloths (100% virgin wool), used with 0.3 micron aluminium oxide or other comparable abrasive, are excelent for intermediate polishing of soft metals (most nonferrous alloys and low carbon steels) and final polishing of hard materials (such as hardened alloy steels).

Sample Preparation

Polishing Cloths

- One of the most popular cloths for final polishing of most metals is composed of densely packed, vertically aligned, synthetic fibres bonded to a suitable backing. For some metals or for particular types of polishing, other cloths, such as velvets, satins, cashmeres or cottons, may be required. The ability to select the proper combination of cloth, abrasive, carrier, polishing speed (rotational speed of the polishing wheel), and pressure applied can be acquired only by experience.

Sample Preparation

Polishing Abrasives

- Polishing usually involves the use one or more of five types of abrasive: aluminium oxide (Al_2O_3), magnesium oxide (MgO), chromic oxide (Cr_2O_3), iron oxide (Fe_2O_3), and diamond compound. With the exception of diamond compound these abrasives are normally used in a distilled water suspension, but if the metal to be polished is not compatible with water, other suspensions, such as ethylene glycol, alcohol, kerosine or glycerin, may be required. The diamond compounds should be extended only with the carrier recommended by the manufacturer.

Sample Preparation
Polishing Abrasives

- Aluminium oxide (aluminia) is the polishing abrasive most widely used for general metallographic polishing. The alpha grade aluminium oxide is used in a range of particle sizes from 15 microns to 0.3 micron. For some hard materials the 0.3 micron size is sufficient for a final polish. The gamma grade of aluminium oxide is available in a 0.05 micron particle size for final polishing.

Sample Preparation

Polishing Abrasives

- Magnesium oxide (magnesia) is recommended for final polishing, especially for the preparation of magnesium and aluminium, and their alloys. Only the metallographic grades, which contain no water soluble alkalis, should be used; otherwise, any free alkalis present could stain and chemically attack the specimen. Magnesium oxide also reacts slowly with water to form magnesium hydroxide. This in turn reacts with carbon dioxide present in the atmosphere and in tap water to form magnesium carbonate, which can contaminate the polishing lap.

Sample Preparation

Polishing Abrasives

- If carbonate is present, the polishing cloth must be replaced or treated in acidified destilled water (2% HCl solution) to convert the carbonate to water-soluble magnesium chloride. If the polishing cloth is to be retained for future polishing, it should be removed from the wheel immediately after use, washed in running water and stored in the 2% hydrochloric acid solution.

Sample Preparation
Polishing Abrasives

- Chromic oxide, available in a range of particle size down to 0.05 micron, is used for rough and final polishing of steel and cast iron.

- Iron oxide, known as jeweler's rouge is available as a powder of aproximately 3 micron particle size. It may be used for rough and final polishing of steel and cast iron, depending on the finish required.

Sample Preparation

Polishing Abrasives

- Diamond polishing compounds are becoming increasingly popular for preparing metallographic specimen. Diamond is the only substance hard enough and with good enough cutting qualities to be used for mechanical polishing of materials such as boron carbide and sintered tungsten. Specimens that have both hard and soft constituents, such as graphite in cast iron and silicon in aluminium, can be polished without causing relief, with diamond compounds on an appropriate lap. These polishing compounds are available either in water soluble and oil soluble carriers or in the form of dry diamond powder in particle size down to 0.25 microns.

Sample Preparation

Polishing Abrasives

- Rough Polishing - The purpose of the rough polishing step is to remove the damage produced during cutting and planar grinding. Proper rough polishing will maintain specimen flatness and retain all inclusions or secondary phases. By eliminating the previous damage and maintaining the microstructural integrity of the specimen at this step, a minimal amount of time should be required to remove the cosmetic damage at the final polishing step. Rough polishing is accomplished primarily with diamond abrasives ranging from 9 micron down to 1 micron diamond. Polycrystalline diamond because of its multiple and small cutting edges, produces high cut rates with minimal surface damage, therefore it is the recommended diamond abrasive for metallographic rough polishing on low napped polishing cloths.

Basic Rough Polishing Guidelines

- For Metals (ferrous, non-ferrous, tool steels, superalloys, etc

- Rough polishing typically requires two polishing steps, such as a 6 micron diamond followed by a 1 micron diamond on low napped polishing cloths

Basic Rough Polishing Guidelines

- For Ceramics and ceramic matrix composites (CMC)

- Low nap polishing pads using polycrystalline diamond alternating with colloidal silica. This provides a chemical mechanical polishing (CMP) effect which results in a damage free surface finish.

Basic Rough Polishing Guidelines

- For Polymer matrix composites (PMC)

- Diamond lapping films

Basic Rough Polishing Guidelines

- For Biomaterials

- Low napped polishing pads with polycrystalline diamond alternating with colloidal silica.

- Alternatively, diamond lapping films may work well.

Basic Rough Polishing Guidelines

- For Microelectronic specimens

- Diamond lapping films.

Basic Rough Polishing Guidelines

- For Plastics and polymers
- 800 and 1200 grit SiC abrasive paper.

Basic Rough Polishing Guidelines

- For Plasma spray materials

- Diamond lapping films or low napped polishing pads with alternating diamond and colloidal silica abrasives

Final Polishing

- Final Polishing –
- The purpose of final polishing is to remove only surface damage. It should not be used to remove any damage remaining from cutting and planar grinding. If the damage from these steps is not complete, the rough polishing step should be repeated or continued.

Recommended Final Polishing Guidelines

- For Metals (ferrous, non-ferrous, tool steels, superalloys, etc.)

- High napped polishing pads with a colloidal alumina polishing abrasive. The polishing times should nominally be less than 30 seconds.

Recommended Final Polishing Guidelines

- For Ceramics and ceramic matrix composites (CMC)

- Low napped polishing pads using 1 um polycrystalline diamond alternating with colloidal silica or colloidal silica alone.

Recommended Final Polishing Guidelines

- For Polymer matrix composites (PMC)

- Fine abrasive diamond lapping films followed by a very light polish on a high napped polishing pad.

Recommended Final Polishing Guidelines

- For Biomaterials

- Low napped polishing pads with polycrystalline diamond, alternating with colloidal silica.

- Alternatively, diamond lapping films may work well.

Recommended Final Polishing Guidelines

- For Microelectronic specimens

- Diamond lapping films followed by a very light polish on a high napped polishing pad.

Recommended Final Polishing Guidelines

- For Plastics and polymers

- Light polish with alumina on a high napped polishing pad.

Recommended Final Polishing Guidelines

- For Plasma spray materials

- Diamond lapping films followed by a very light and short alumina or colloidal silica polish on a high napped polishing pad.

Sample Preparation

Electrolytic Polishing

- Even with the most careful mechanical polishing, some disturbed metal however small the amount, will remain after preparation of a metallographic specimen. This is no problem if the specimen is to be etched for structural investigation, because etching is usually sufficient to remove the slight layer of disturbed metal. If the specimen is to be examined in the as-polished condition, however, or if no surface disturbance can be tolerated, either electrolytic polishing or chemical polishing is preferred.

Sample Preparation
Electrolytic Polishing

- In electrolytic polishing (elektropolishing), the specimen is the anode in an electrolytic cell. Direct current from an external cell is applied to the electrolytic cell under specific conditions, and anodic dissolution results in a leveling and brightening of the specimen surface. The most widely accepted theory postulated to explain the leveling action concerns the layer of viscous material (usually visible) that is present on the specimen surface during electropolishing. This layer (termed the anolyte layer) is composed of products that result from the reaction between the metal and the electrolyte, and is, according to the theory, necessary for proper electropolishing.

Sample Preparation
Electrolytic Polishing

- At areas of elevation on the specimen surface, this anolyte layer is thinner and offers less resistance to the flow of current than areas of depression. The resulting higher current density at elevated areas causes the metal in those areas to dissolve more rapidly than metal in depressed areas, and thus levels the surface of the specimen. The brightening of the anodic surface is atributed to the formation of a thin film that follows contours of the surface and to uniform attack of this film by the electrolyte.

Sample Preparation
Electrolytic Polishing

- Electropolishing does not disturb any metal on the specimen surface and therefore is ideally suited for the metallographic preparation of soft metals, most single-phase alloys, and alloys that work harden readily. The disadvantages of electropolishing include preferential attack in multiphase alloys caused by differences in electrical potential between phases, and chemical attack of nonmetalic inclusions by the electrolyte. Proper choice of electrolyte and operating conditions will minimize these disadvantages.

Sample Preparation
Etching

- Chemical Etching
- Electrochemical Etching
- Physical Etching
- Cathodic Vacuum Etching

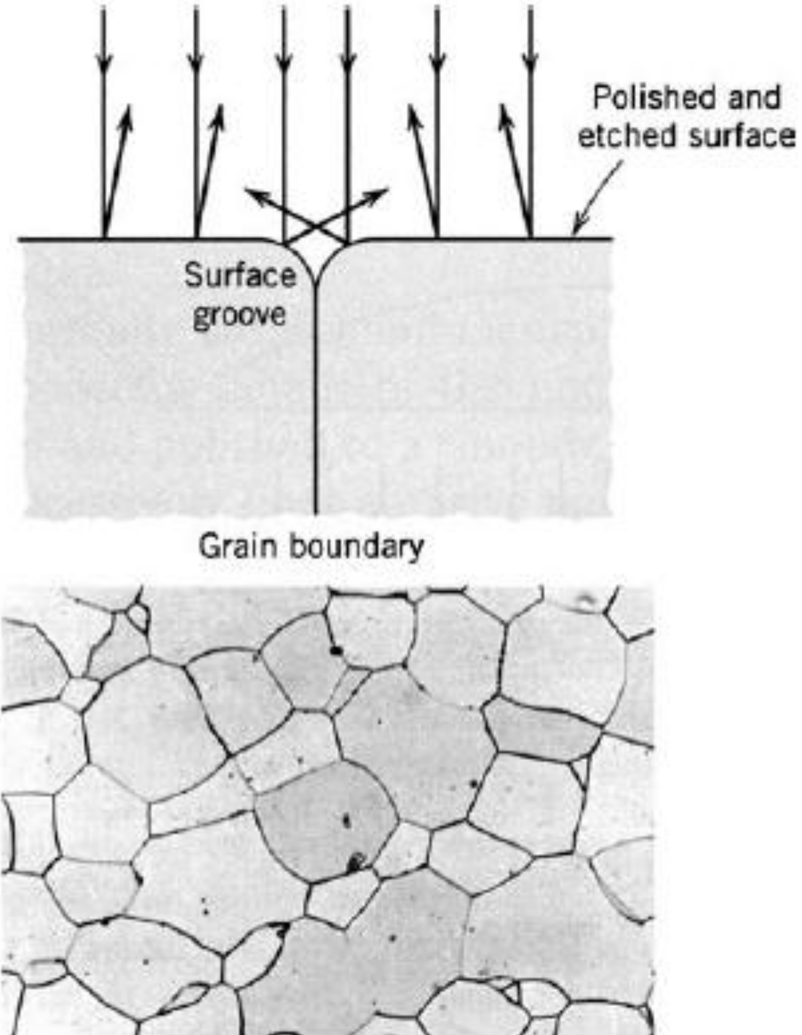
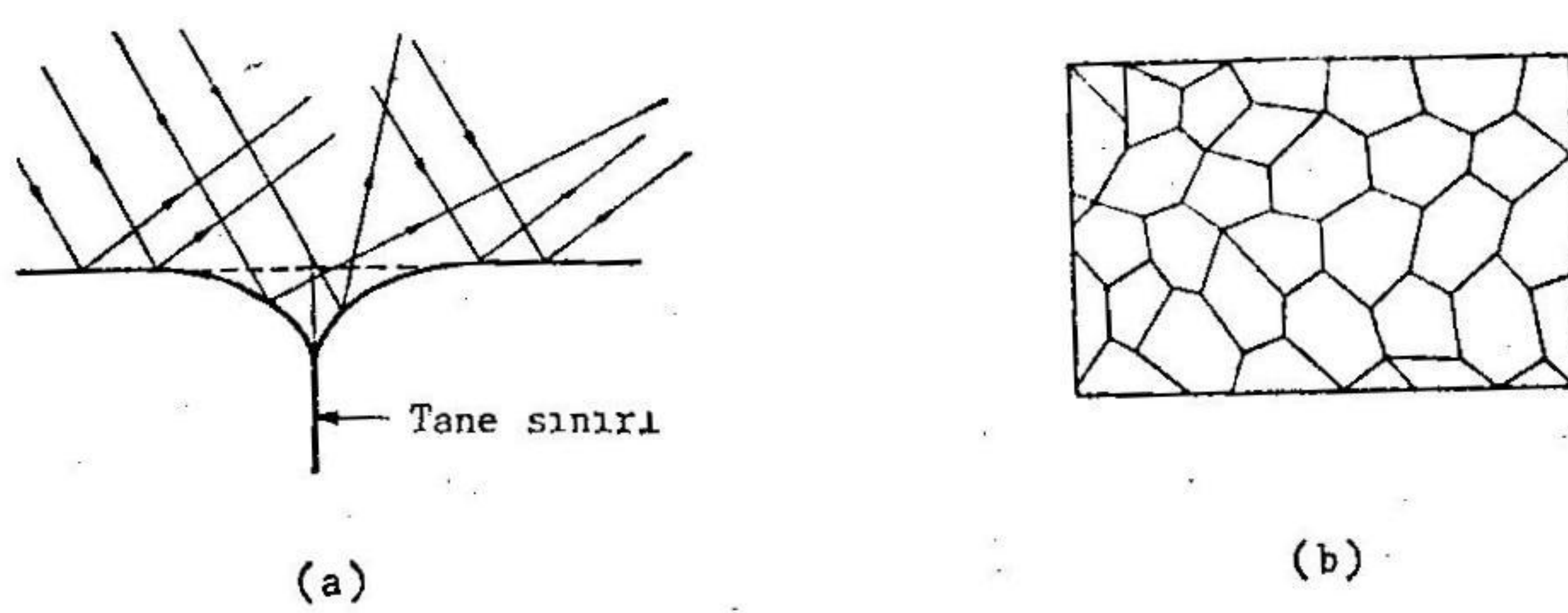
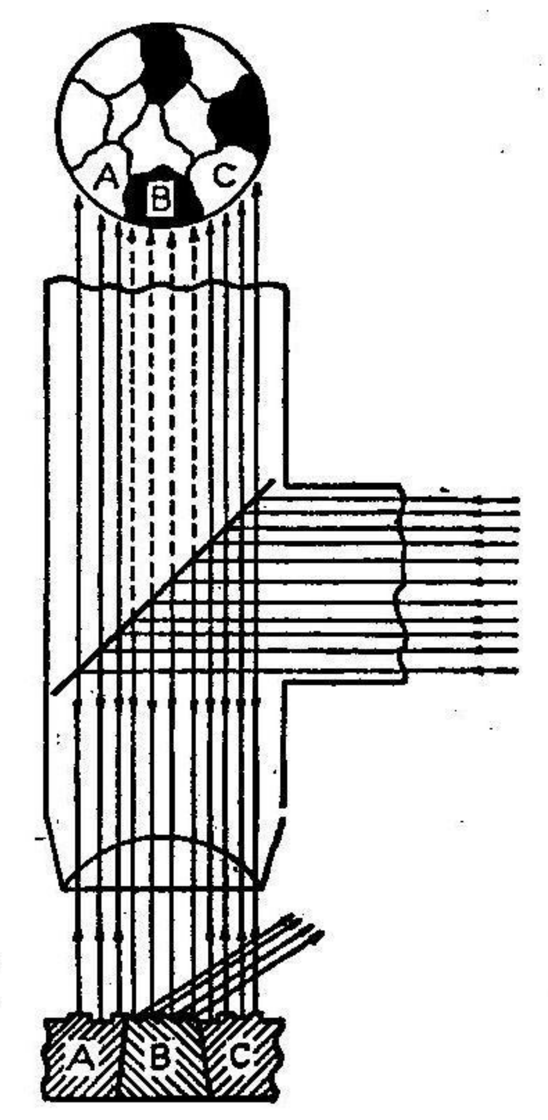


Figure 1.26 Contrast generated by etching grain boundaries in light microscope: (a) reflection from different parts of a surface (Reproduced with permission from W.J. Callister Jr., *Materials Science and Engineering: An Introduction*, 7th ed., John Wiley & Sons Inc., New York. © 2006 John Wiley & Sons Inc.); and (b) micrograph of grain boundaries which appear as dark lines. (Contribution of the National Institute of Standards and Technology.)



Şekil 2-22 — Tane sınırlarında, a) dağlama sonucu vadi teşekkülü ve b) mikroskopta görünümü



Şekil 2-21 — Çok fazlı yapılarda dağlama mekanizması

Sample Preparation

Etching

- Although certain information may be obtained from as-polished specimens, the microstructure is usually visible only after etching. Only features which exhibit a significant difference in reflectivity (10% or greater) can be viewed without etching. This is true of microstructural features with strong color differences or with large differences in hardness causing relief formation. Cracks, pores, pits, and nonmetallic inclusions may be observed in the as-polished condition. In most cases, a polished specimen will not exhibit its microstructure because incident light is uniformly reflected.

Sample Preparation

Etching

- Since small differences in reflectivity cannot be recognized by the human eye, some means of producing image contrast must be employed. Although this has become known as "etching" in metallography, it does not alway refer to selective chemical dissolution of various structural features. There are numerous ways of achieving contrast. These methods may clasified as optical, electrochemical (chemical), or physical, depending on whether the process alters the surface or leaves if intact.

Sample Preparation

Etching

- The purpose of etching is to optically enhance microstructural features such as grain size and phase 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 and plasma etching have also found specialized applications.

Sample Preparation

Chemical Etching

- Chemical etching is based on the aplication of certain illumination methods, all of which use the Kohler illumination principle. This principle also underlies common bright-filed illumination. These illumination modes are dark field, polarized light, phase contrast and interference contrast. They are available in many commercially produced microscopes, and in most cases, the mode may be put into operation with few simple manipulations.

Sample Preparation

Chemical Etching

- There is distinct advantage in employing optical etching rather than those technique which aleter the specimen surafce. Chemical and physical etching require considerable time and effort and there is always a danger of producing artifacts which lead to misinterpretations.

Sample Preparation

Chemical Etching

- Chemical etching - slectively 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), often known as Eh-pH diagrams or Pourbaix diagrams. Over the years numerous chemical etchants have been formulated.

Common Chemical Etchants

Etchant	Composition	Conc.	Conditions	Comments
Kellers Etch	Distilled water Nitric acid Hydrochloric acid Hydrofluoric acid	190 ml 5 ml 3 ml 2 ml	10-30 second immersion. Use only fresh etchant	
Kroll's Reagent	Distilled water Nitric acid Hydrofluoric acid	92 ml 6 ml 2 ml	15 seconds	
Nital	Ethanol Nitric acid	100 ml 1-10 ml	Seconds to minutes	
Kallings Reagent	Distilled water Copper chloride ($CuCl_2$) Hydrochloric acid Ethanol (85%) or Methanol (95%)	40 ml 2 grams 40 ml 40-80 ml	Immerse or swab for few seconds to a few minutes	
Lepito's Reagent	Acetic acid Nitric acid	50 ml 50 ml	Swab	
Marble's Reagent	Distilled Water Hydrochloric acid Copper sulfate	50 ml 50 ml 10 grams	Immersion or swab etching for a few seconds	
Murakami Reagent	Distilled Water $K_3Fe(CN)_6$ NaOH or KOH	100 ml 10 grams 10 grams	Immerse or swab for seconds to minutes	Use fresh
Picral	Ethanol Picric acid	100 ml 2-4 grams	Seconds to minutes	Do not let etchant crystallize or dry **-explosive**
Vilella's Reagent	Glycerol Nitric acid Hydrochloric acid	45 ml 15 ml 30 ml	Seconds to minutes	

Guide to Acid Concentrations

Acid/ Base	Specific gravity	Acid concentration
Nitric (HNO_3)	1.4	68-70%
Hydrofluoric (HF)	-	40%
Hydrochloric (HCl)	-	37-38%
Ammonium Hydroxide (NH_4OH)	-	35%

Sample Preparation

Electrochemical Etching

- During the process of electrochemical etching of metallic specimen, reduction and oxidation process (redox process) take place. All metals in contact with the solution have a prononounced tendency to become ionized by releasing (losing) electrons. The degree to which this reaction takes place may be recorded by measuring the electrochemical potential. This is done by comparing the potential of metal versus the standard potential of a reference electrode. The tabulation of various metals results in the electromotive series of elements:
  Li+ , Na+ , K+, Ca++ , Ba++ , Be++ , Mg++ , Al+++ , Mn++ , Zn++ , Cr++ , Cd++ , Ti+ , Co++ , Ni++ , Pb++ , Fe+++ , H+ , Sn++++ , Sb+++ , Bi+++ , As+++ , Cu++ , Ag+ , Hg++, Au+++ , Pt+++.

Sample Preparation

Electrochemical Etching

- The elements are listed in order of decreasing electroaffinity. All elements preceding hydrogen are attacked by acids with the evolution of hydrogen (H_2). All elements following hydrogen cannot be attacked without the addition of an oxidizing agent. Thus, microstructural elements of different electrochemical potential are attacked at different rates. This produces differential etching, resulting in microstructural contrast. Electrochemical etching may be considered as "forced corrosion". The differences in potential of the microstructural elements cause a subdivison into a network of very small anodic or cathodic regions. These miniature cells cannot originate from differences in phase composition only, but also have to come from irregularities in the crystal structure as they are present - for example, at grain boundaries and from other inhomogeneities such as:

Sample Preparation

Electrochemical Etching

- Inhomogeneities resulting from deformation, which are less reistant to attack than undeformed material.
- Unevenness in the formation of oxidation layers
- Concentration fluctuation in the electrolyte
- Differences in electrolyte velocity
- Differences in the oxygen content of the electrolyte
- Differences in the illumination intensity, which can initiate diferences inpotential

Sample Preparation
Electrochemical Etching

- Because of differences in potential between microstructural features, dissolution of the surface proceeds at different rates, producing contrast. Contrast can also originate from layers formed simultaneously with material dissolution. This is true in precipitation etching and heat tinting where surface reactions are involved. In precipitation etching the material is first dissolved at the surface; it then reacts with certain components of the etchant to form insoluble compounds. These compounds precepitate selectively on the surface, causing interference colors or heavy layers of a specific color. During heat tinting, coloration of the surface takes place at different rates according to the reaction charcteristics of different microstructural elements under the given conditions of atmosphere and temperature.

Sample Preparation

Electrochemical Etching

- A wide variety of etchants is available, including acid, bases, neutral solutions, mixtures of solutions, molten salts and gases. The stability of many etching solutions is limited; redox potentials change with time. Changes may also occur while the etchant isin use, such that it must be discarded after a limited time.

Sample Preparation
Electrochemical Etching

- Etching times range from several seconds to some hours. When no instractions are given, progress is judged by the appearance of the surface during etching. Usually, the surface will become less reflective as etching proceeds. Etching time and temperature are closely related; by increasing the temperature, the time can usually be decreased. Most etching is performed at room temperature.

Sample Preparation
Electrochemical Etching

- Conventional chemical etching is the oldest and most commonly applied technique for production microstructural contrast. In this technique, the etchant reacts with the specimen surface without the use of an external current supply. Etching proceeds by selective dissolution according to the electrochemical characteristic of the componenet areas.

Sample Preparation
Electrochemical Etching

- In electrolytic or anodic etching, an electrical potential is applied to the specimen by means of an external circuit. Typical setup consist, the specimen (anode) and its counterelectrode (cathode) immersed in an elctrolyte.
- Potentiostatic etching is an advanced form of electrolytic etching, which produces the ultimate etching contrast through highly controlled conditions.

Sample Preparation
Electrochemical Etching

- On completion of any chemical or electrochemical etching process, the specimen should be rinsed in clean water to remove the chemicals and stop any reactions from proceeding futher. After specimens are water rinsed, they should be rinsed in alcohol and dried in a stream of warm air. The use of alcohol speeds up the drying action and prevents the formation of water spots.

Common Electrochemical Etchants

Common Electrochemical Etchants

Application	Etchant	Conditions	Comments
Wrought stainless steel	Concentrated NH4OH	Stainless steel cathode 6 volts DC Room temperature 30-60 seconds	General structure
Austenitic stainless steels	100 ml Distilled water 10 gram CrO_3	3-6 volts DC 5-60 seconds	Attacks carbides and sigma phase
Copper and copper alloys (Cu)	5-14% H_3PO_4 Remainder water	1-4 volts DC 10 seconds	Copper and brass
Titanium (pure)	25 ml Distilled water 390 ml Methanol (95%) Ethylene glycol 35 ml perchloric acid (70%)	Stainless steel cathode 30-50 volts DC 5-10 °C (40-50 °F) 10-40 seconds	
Titanium carbide (TiC)	10 ml Distilled water 2 grams potassium hydroxide	Pt cathode 2 volts DC 30-60 mA/cm^2 2-30 seconds	
Wrought Fe-Ni-Cr Heat resisting alloys (Fe)	100 ml Distilled water 5 gram CrO_3	Stainless steel cathode 5-10 volts DC 15-20 seconds Room temperature	General structure
Steels	10 grams Chromic acid 100 ml Distilled water	Pt or stainless steel cathode 3-6 volts DC 5-60 seconds	Carbide and cementite etching
Nimonic alloys (Ni)	45 parts Hydrochloric acid 15 parts Nitric acid 40 parts glycerol	Stainless steel cathode 2-4 volts DC, 0.5 A/dm^2 5-15 seconds	Nimonic PK31
Stainless Steels	100 ml Distilled water 10 grams oxalic acid	Stainless steel cathode 6 volts DC Room temperature	General structure

Sample Preparation

Physical Etching

- Basic physical phenomena are also often used to develop strucural contrast, mainly when conventional chemical or electrolytic techniques fail. They have the advantage of leaving surfaces free from chemical residues and also offer adavantages where electrochemical etching is difficult - for example, when there is an extremely large difference in electrochemical potential between microstructural elements, or when chemical etchants produce ruinous stains or residues. Some probable applications of these methods are plated layers, welds joining highly dissimular materials, porous materials, and ceramics.

Sample Preparation

Cathodic Vacuum Etching

- Cathodic vacuum etching, also referred to as ion etching, produces structural contrast by selective removal of atoms from the sample surface. This is accomplished by using high-energy ions (such as argon) accelerated by voltages of 1 to 10 kV. Individual atoms are removed at various rates, depending on the microstructural details such as crystal orientation of the individual grains, grain boundaries, etc.

Sample Preparation

Cathodic Vacuum Etching

- Plasma etching is a lesser known technique that has been used to enhance the phase structure of high strength ceramics such as silicon nitride. For silicon nitride, the plasma is a high temperature flouride gas, which reacts with the silicon nitride surface producing a silicon flouride gas. This etching technique reveals the intragrain microstructure of silicon nitride.

Molten Salt-etching

- Molten Saltetching 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 materials grain boundaries. At the elevated temperature of molten salts, the higher energy at the grain boundaries is relieved, producing a rounded grain boundary edge which can be observed by optical or electron microscope techniques. Some common molten salts are listed in the following table.

Common Molten Salt Etchants

Common Molten Salt Etchants

Application	Etchant	Conditions	Comments
Cr_2O_3, CeO_2, Al_2O_3	Potassium hydrogen sulfate melt	Pt crucible 3-10 minutes	Toxic
Al_2O_3, Al_2SiO_5	Potassium hydrogen fluoride melt	Pt crucible 5-10 minutes	Toxic
Si_3N_4	Sodium or potassium bicarbonate melt	10 minutes	Toxic

Thermal Etching

- Thermal etchingis a useful etching technique for ceramic materials. Thermal etching is technique that relieves the higher energy associated at the grain boundaries of a material. By heating the ceramic material to temperatures below the sintering temperature of the material and holding for a period of minutes to hours the grain boudaries will seek a level of lower energy. The result is that the grain boundary edge will become rounded so that it can be evaluated by optical or electron microscope techniques.

Thermal Etching

- 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.

Common Thermal Etching Conditions

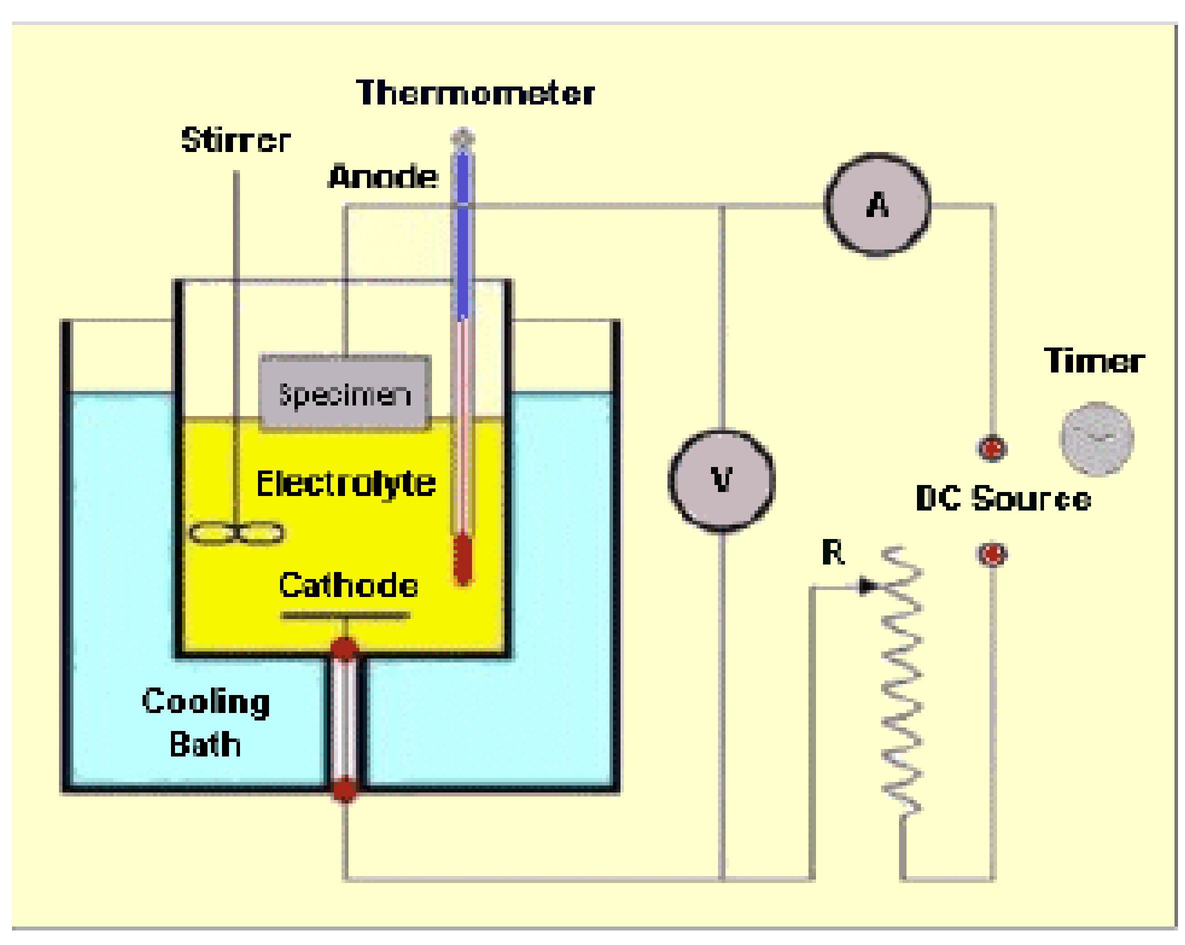
Common Thermal Etching Conditions

Application	Conditions	Comments
SiC	Vacuum (< 10^{-3} Torr) 1200 °C (2200 °F)	-
UO_2	10 Torr 1600 °C (2900 °F) 1 hour	-
Si_3N_4	Vacuum (< 10^{-5} Torr) 1250 °C (2282 °F) 15 minutes	-

Electrolytic Polishing

- Even with the most careful mechanical polishing, some disturbed metal however small the amount, will remain after preparation of a metallographic specimen. This is no problem if the specimen is to be etched for structural investigation, because etching is usually sufficient to remove the slight layer of disturbed metal. If the specimen is to be examined in the as- polished condition, however, or if no surface disturbance can be tolerated, either electrolytic polishing or chemical polishing is preferred.

Electrolytic Polishing



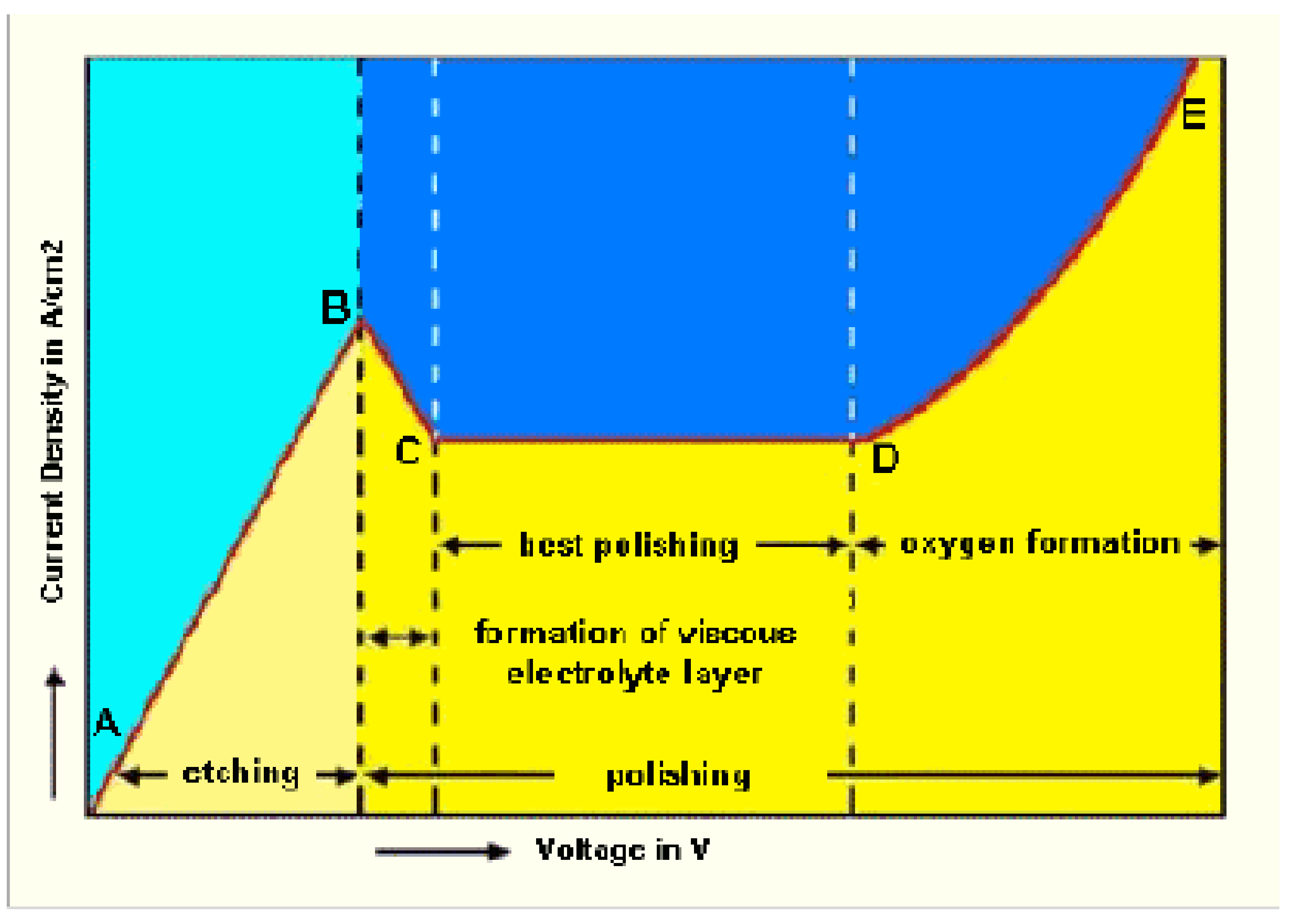
Electrolytic Polishing

- Electrolytic preparation uses an electrolytic reaction cell containing a liquid electrolyte with two electrodes: an anode and cathode. The sample to be polished/etched forms the anode. The electrodes are connected to an external power supply and voltage applied to cause reaction within the cell. In electrolytic polishing (elektropolishing), the specimen is the anode in an electrolytic cell. Direct current from an external cell is applied to the electrolytic cell under specific conditions, and anodic dissolution results in a leveling and brightening of the specimen surface. The most widely accepted theory postulated to explain the leveling action concerns the layer of viscous material (usually visible) that is present on the specimen surface during electropolishing.

Electrolytic Polishing

- This layer (termed the anolyte layer) is composed of products that result from the reaction between the metal and the electrolyte, and is, according to the theory, necessary for proper electropolishing. At areas of elevation on the specimen surface, this anolyte layer is thinner and offers less resistance to the flow of current than areas of depression. The resulting higher current density at elevated areas causes the metal in those areas to dissolve more rapidly than metal in depressed areas, and thus levels the surface of the specimen. The brightening of the anodic surface is atributed to the formation of a thin film that follows contours of the surface and to uniform attack of this film by the electrolyte

Electrolytic Polishing



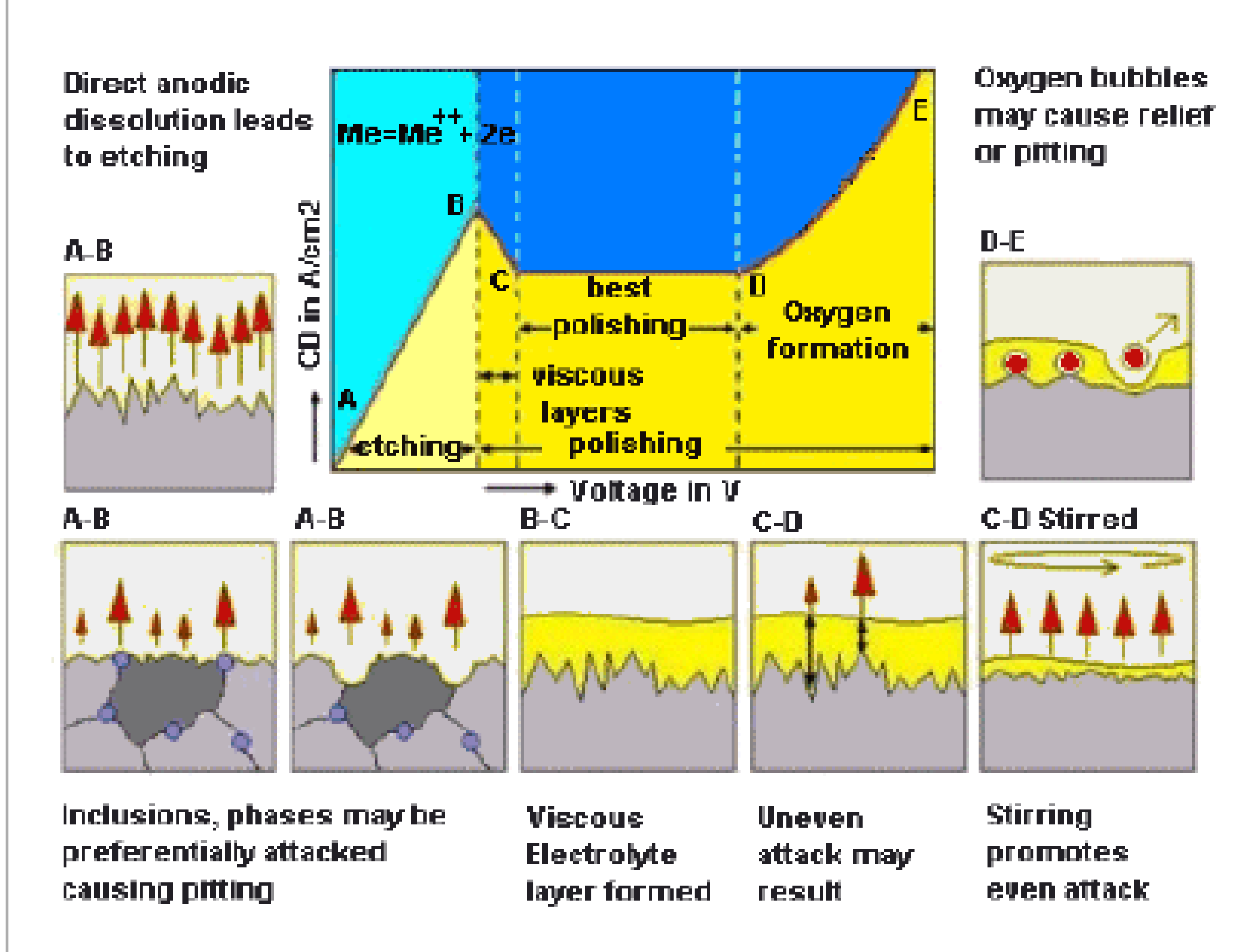
Electrolytic Polishing

- Shown above is the characteristic curve for an electrolytic cell. This curve is dependant on the electrolyte used and will vary for different electrolytes. Control of the voltage and current density at the anode, plus electrolyte composition, temperature and agitation are all critical in achieving the desired etching/polishing characteristics. Establishing adequate control of these parameters can be difficult and further, many of the electrolytes are hazardous or even explosive. In the case of the latter, temperature control is critical. Do not attempt electro polishing or etching without the necessary experience and safety measures in place.

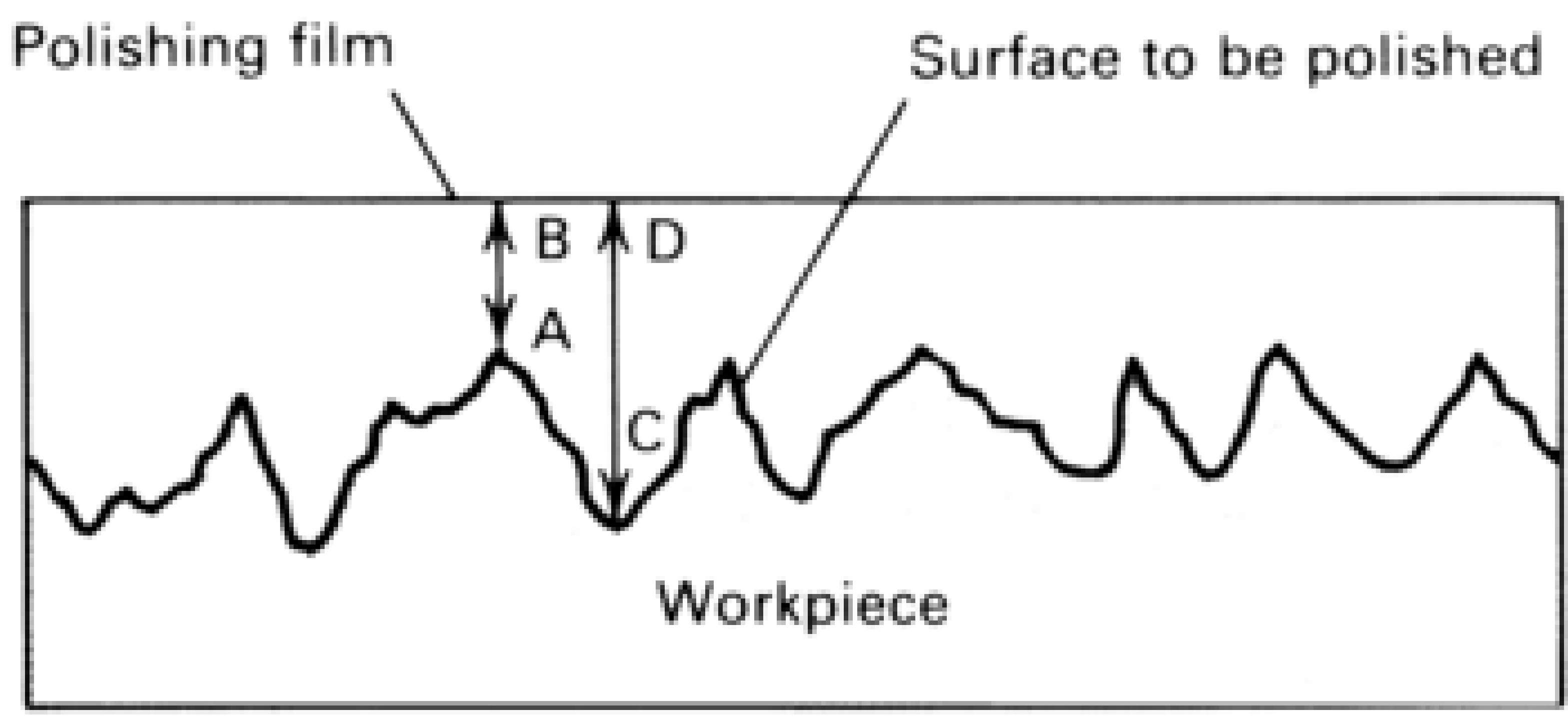
Electrolytic Polishing

- Electropolishing does not disturb any metal on the specimen surface and therefore is ideally suited for the metallographic preparation of soft metals, most single-phase alloys, and alloys that work harden readily. The disadvantages of electropolishing include preferential attack in multiphase alloys caused by differences in electrical potential between phases, and chemical attack of nonmetalic inclusions by the electrolyte. Proper choice of electrolyte and operating conditions will minimize these disadvantages.

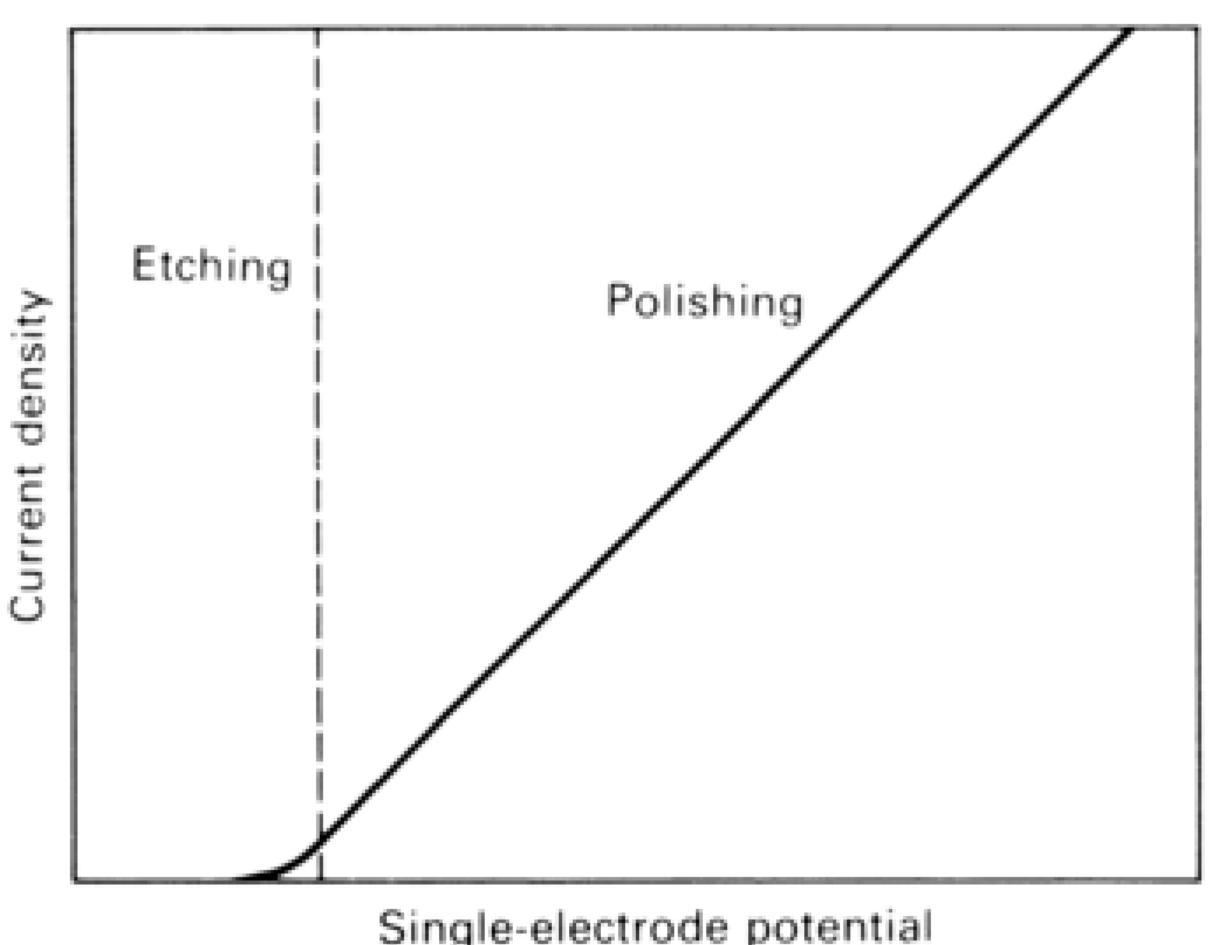
Electrolytic Polishing



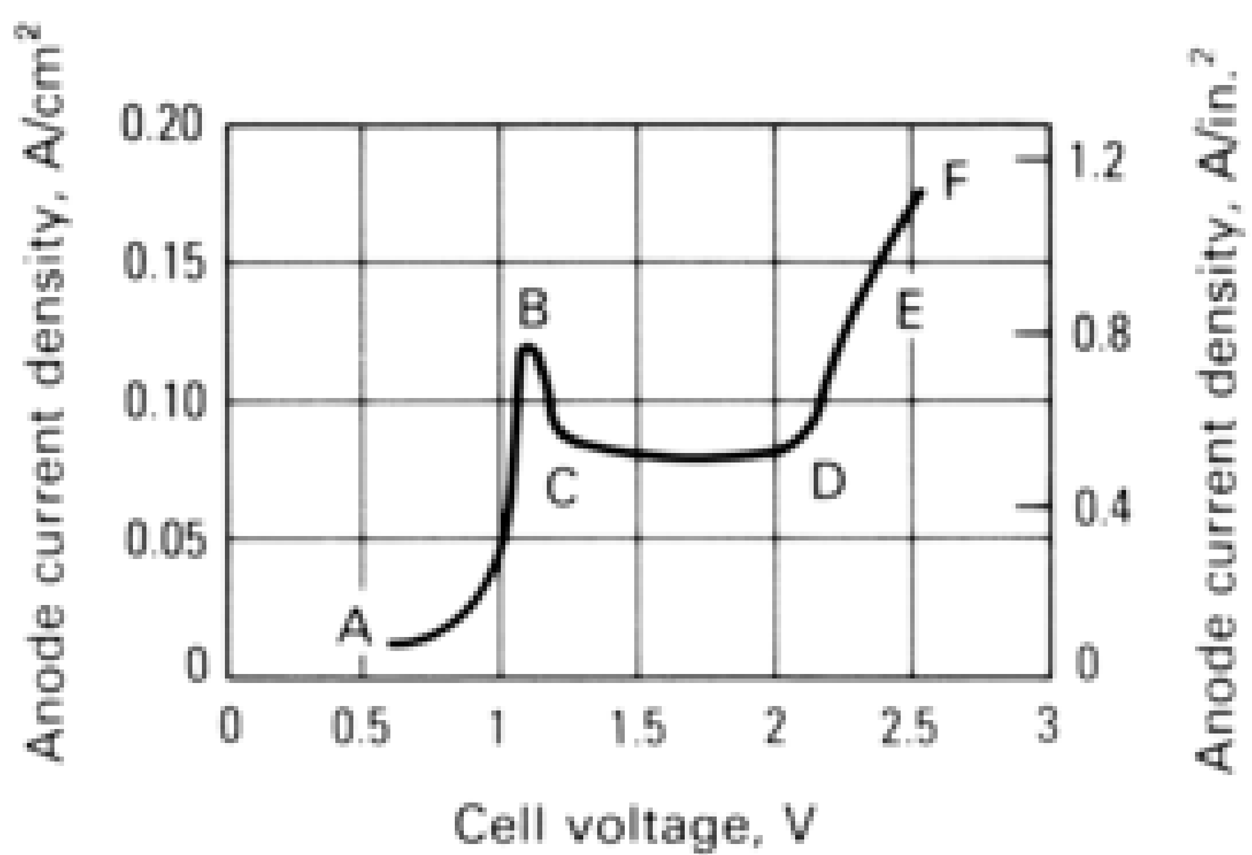
Mechanism of electrolytic polishing



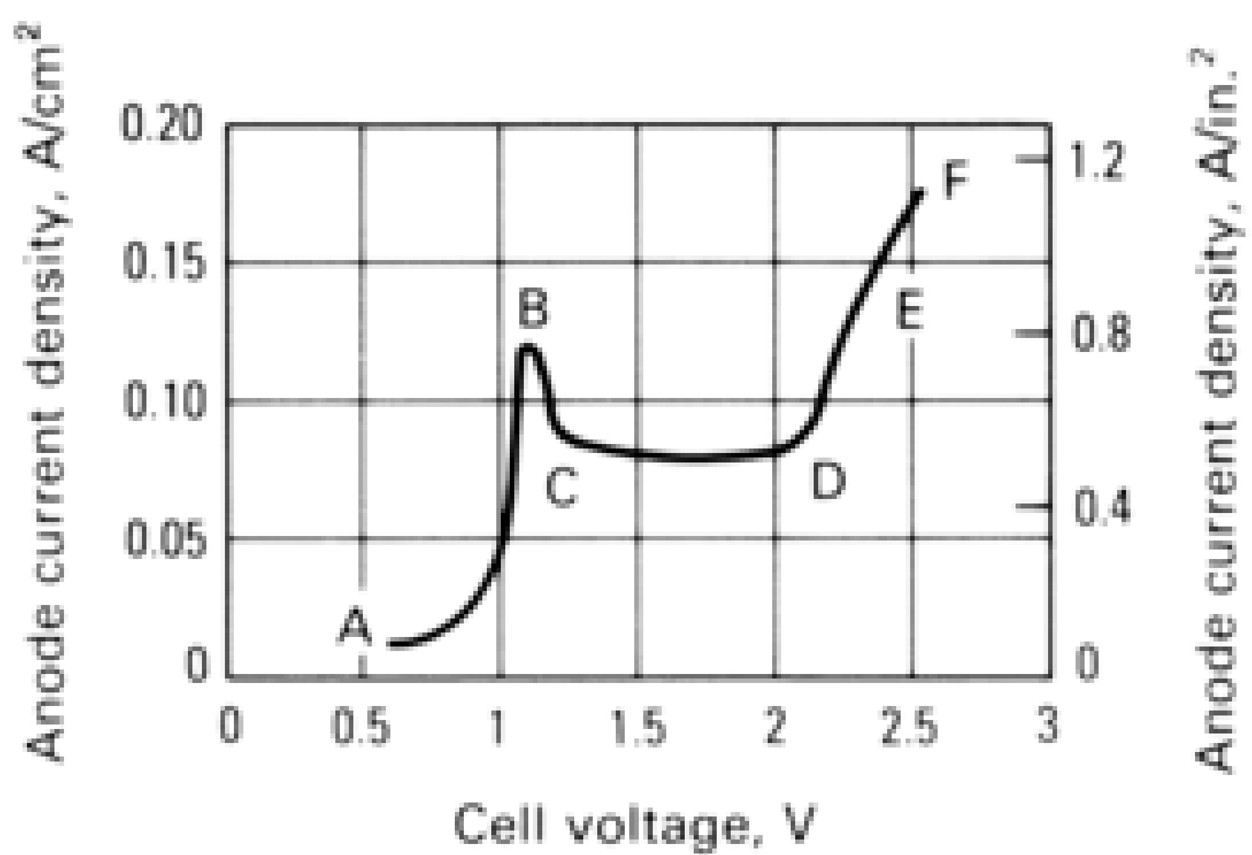
Relationship between current density and single-electrode potential for electrolytes possessing polishing action over a wide range of voltages and currents



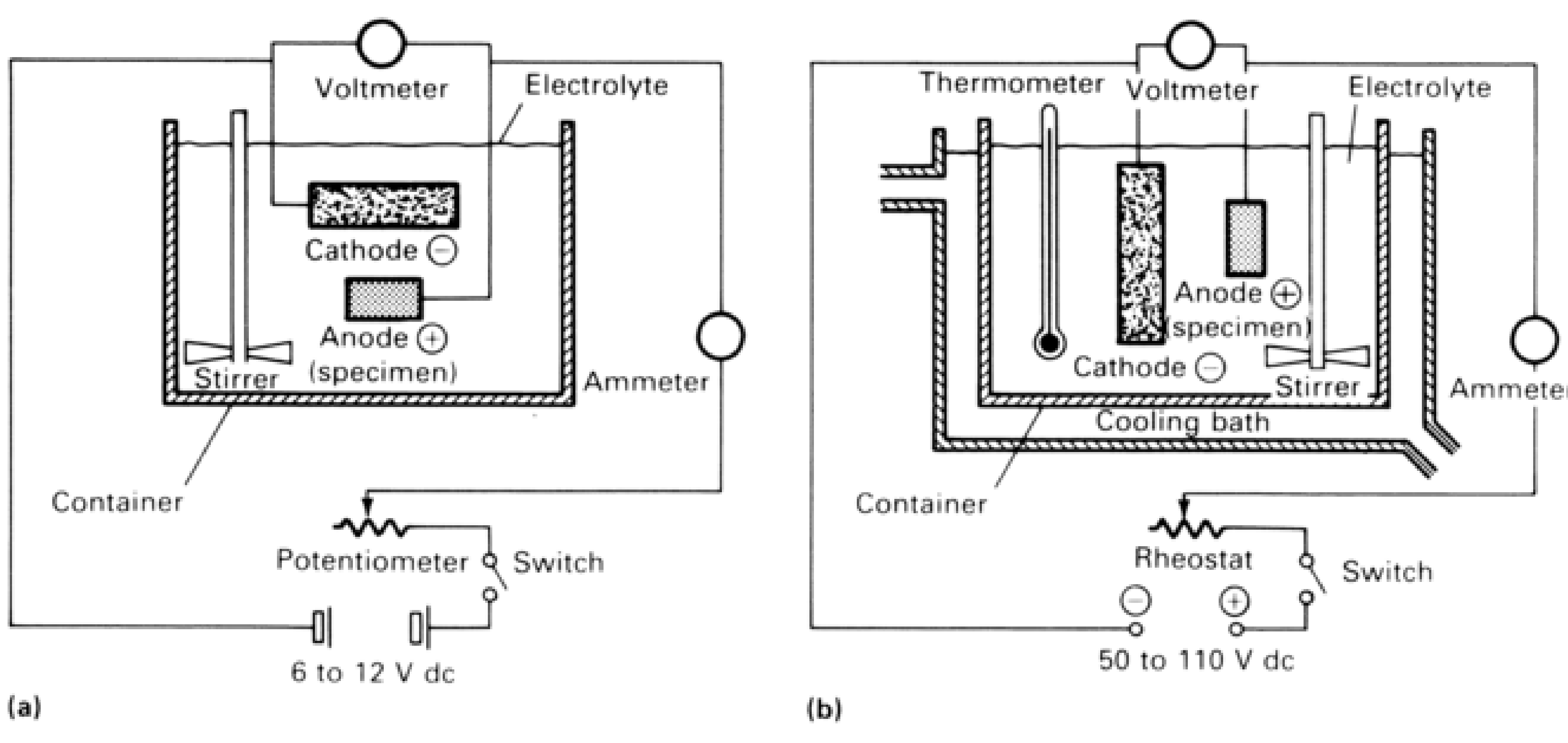
Cell voltage as a function of anode current density for electropolishing copper in ortho-H3PO4 (900 g per 1000 mL H2O), using a potentiometric circuit



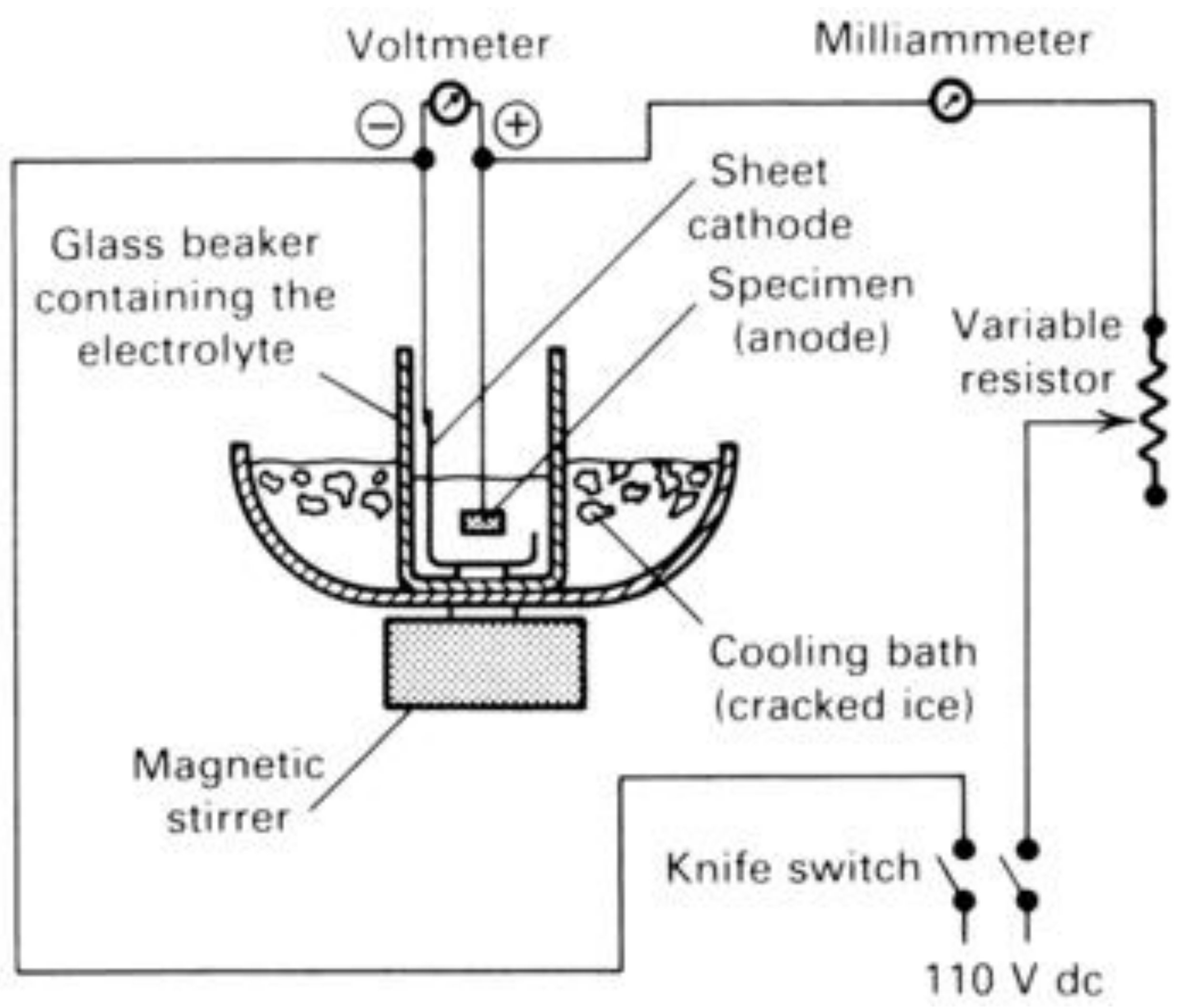
Cell voltage as a function of anode current density for electropolishing copper in ortho-H3PO4 (900 g per 1000 mL H2O), using a potentiometric circuit



Typical electrical circuits and equipment setups used for electropolishing. (a) Potentiometric circuit (for low current densities). (b) Series circuit (for high current densities)



Basic laboratory setup for electropolishing and electrolytic etching



Factors controlling etching/polishing characteristics include:

- Electrolyte composition
- Electrolyte temperature
- Electrolyte stirring
- Area to be polished/etched (current density)
- Voltage

Advantages - Disadvantages of Electrolytic Etching or Polishing:

- Fast
- Can be reproducible
- No mechanical deformation
- Can be automated
- Can produce excellent surfaces for examination
- Conductive specimens only
- Not all alloys can be polished
- Preferential attack or pitting can occur
- No edge retention
- Limited polishing area
- Limited scratch/material removal
- Hazardous Electrolytes
- Temperature control vital
- Establishing correct conditions can be difficult

Sample Preparation
Cleaning

- Cleanliness is an important requiremet for succefful sample preparation. Specimens must be cleaned after each step, all grains from one grinding and polishing step must be completely removed from the specimen to avoid contamination, which would reduce the efficiency of the next preparation step. Through cleaning is particularly critical after fine grinding and before rough polishing and all subsequent steps.

Sample Preparation
Cleaning

- Clean, grease free surfaces are essential for subsequent chemical or electrolytic treatment. Residues, fingerprints, and inconspicuous films may interfere with etching, causing various areas to be attacked at different rates. Every single microsection preparation procedure must be followed by through cleaning, which can be performed in different ways.

Sample Preparation

Cleaning

- Rinsing is most frequently used and consists of holding specimen under a stream of running water and wiping the surafce with a soft brush or cotton swab.
- Ultrasonuc cleaning is the most effective and thorough method of cleaning. Not only area surface contaminants removed, but particulate matter held in crevicesm, crack, or pores is removed by the action of cavitation. Usually this ultrasonic cleaning needs only 10 to 30 s.

Sample Preparation
Cleaning

- After cleaning, specimens may be dried rapidly by rinsing in alcohol, benzene, or other low-boiling-point liquids, then placed and a hot-air drier for sufficient time to vaporize liquids remaining in cracks and pores.

Sample Preparation
Specimen Storage

- When polished and etched specimens are to be stored for long periods of time, they must be protected from atmospheric corrosion. Desiccators and vacuum desiccators are the most common means of specimen storage, althrough plastic coatings and cellophane tape are sometimes used.

Definitions of etching methods

Method	Definition
Anodic etching	Reveals the microstructure by selective anodic dissolution of the polished surface using a DC current. Variation with layer formation: anodizing
Attack polishing	Simultaneous etching while mechanical polishing
Cathodic etching	See *ion etching*.
Cold etching	Reveals the microstructure at room temperature and below
Controlled etching	Electrolytic etching with selection of suitable etchant and voltage, resulting in a balance between current and dissolved metal ions
Crystal figure etching	Discontinuity in etching depending on crystal orientation. Distinctive sectional figures form at polished surface. Closely related to *dislocation etching*
Deep etching	Macroetching, especially for steels, to determine the overall character of the material (presence of imperfections such as seam defects, rolling defects, forging bursts, remnant shrinkage voids, cracks, and coring)
Dislocation etching	Reveals exit points of dislocations on the sample surface. Etching of dislocations is caused by their strain field ranging over a distance of several atoms. Crystal figures (etch pits) are formed at the exiting points. For example, etch pits for cubic materials are cube faced.
Dissolution etching	Reveals the microstructure by surface removal
Double etching	Two etchants are used sequentially, the second one will accentuate a particular microstructural feature.
Drop etching	Placing a drop of an etchant on a selected area of the sample surface to develop the alloying microconstituents (drip reaction)

Definitions of etching methods

Dry etching	Develops the microstructure by gaseous exposure
Electrochemical (chemical etching)	General term for revealing the microstructure by redox reactions
Electrolytic etching	See *anodic etching*.
Etch rinsing	Pouring the etchant over a tilted sample surface until the structure is revealed. Used for etching with severe gas evolution
Eutectic cell etching	Reveals eutectic grains (cells)
Grain-boundary etching	Reveals the intersections of individual grains. Grain boundaries have a higher dissolution potential than the individual grains because of their high density of structural defects. Accumulation of impurities in grain boundaries increases this effect.
Grain-contrast etching	Etching the surface of the grains according to their crystal orientation. They become distinct by the different reflectivity caused by reaction layers or surface roughness.
Heat tinting	Formation of interference colors in air or other gases, usually at elevated temperature
Hot etching	Development and stabilization of the microstructure at elevated temperature in etching solutions or gases
Identification (selective) etching	Etching for the identification of particular microconstituents without attacking any others
Immersion etching	The sample is immersed in the etchant with the polished surface up and is agitated. This is the most common etching method.

Definitions of etching methods

Immersion etching (cyclic)	Alternate immersion into two etchants: 1, the actual etchant; 2, solution to dissolve the layer formed during the etching process of 1
Ion etching	Surface removal by bombardment with accelerated ions in a vacuum (1 to 10 kV)
Long-term etching	Etching times of a few minutes to several hours
Macroetching	Reveals the macrostructure for the examination with the unaided eye or at a magnification of 50× or less
Microetching	Reveals the microstructure for microscopic observation at a magnification of 50× or higher
Multiple etching	A sample is etched sequentially with specific etchants to reveal certain constituents.
Network etching	Formation of networks (subgrain boundaries), especially in mild steels after etching in nitric acid
Optical etching	Develops the microstructure by using special illumination techniques (dark-field, phase contrast, interference contrast, polarized light)
Physical etching	Develops the microstructure through removal of surface atoms or lowering the grain surface potential
Plasma etching	High-frequency electromagnetic vibrations produce radicals in a gas mixture that react with the sample surface and cause its removal.
Potentiostatic etching	Anodic development of the microstructure at a constant potential enables a defined etching of singular phases.
Precipitation etching	Develops the microstructure by the formation of reaction products at the sample surface
Primary etching	Develops the cast microstructures including coring
Print etching (printing)	A carrier material is soaked with an etching solution and is pressed onto the sample surface. The etchant reacts with one of the microstructural constituents forming substances that affect the carrier material. The result is a direct imprint as a life-size image. It is used for the identification of specific elements, for example, sulfur.

Definitions of etching methods

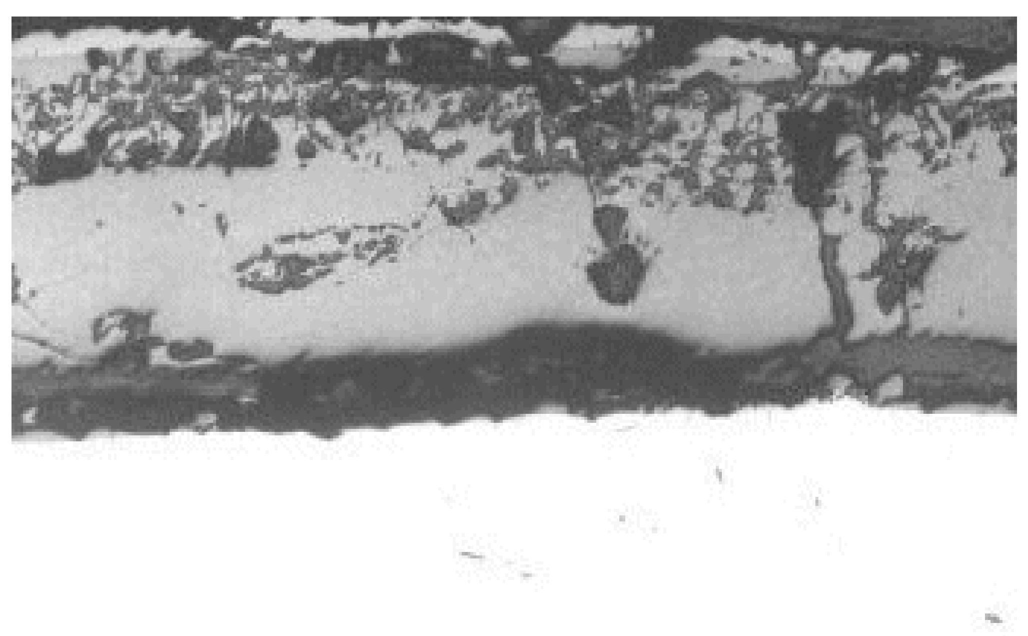
	image. It is used for the identification of specific elements, for example, sulfur.
Secondary etching	Develops the microstructures that differ from primary structures through transformation and heat treatment in the solid state
Segregation (coring) etching	Develops segregation (coring) mainly in macrostructures and microstructures of castings
Short-term etching	Etching time of seconds to a few minutes
Shrink etching	Produces a precipitate coating on the grain surfaces and shrinks upon drying, generating cracks. Crack orientation depends on the underlying crystal structure.
Staining	Precipitation etching that causes contrast by distinctive staining of microconstituents; different interference colors originate from surface layers of varying thickness. Identifies inhomogeneities
Strain etching	Reveals regions of high deformation within undeformed areas. Strained areas show increased segregations of precipitates.
Swabbing	Wiping the sample surface with cotton saturated with the etchant; this will simultaneously remove undesired reaction products.
Thermal etching	Annealing the specimen in a vacuum or inert atmosphere. This is a preferred technique for high-temperature microscopy and for ceramics.
Wet etching	The sample surface has been wetted before immersion into the etching solution. This is important when using color etchants.
Wipe etching	See *swabbing*.

Preparation Artifacts

- Cracks
- Comet Tails
- Contamination
- Deformation
- Edge Rounding
- Embedded Abrasive
- Gaps
- Lapping Tracks
- Porosity
- Pull-Outs
- Relief
- Scratches
- Smearing
- Staining

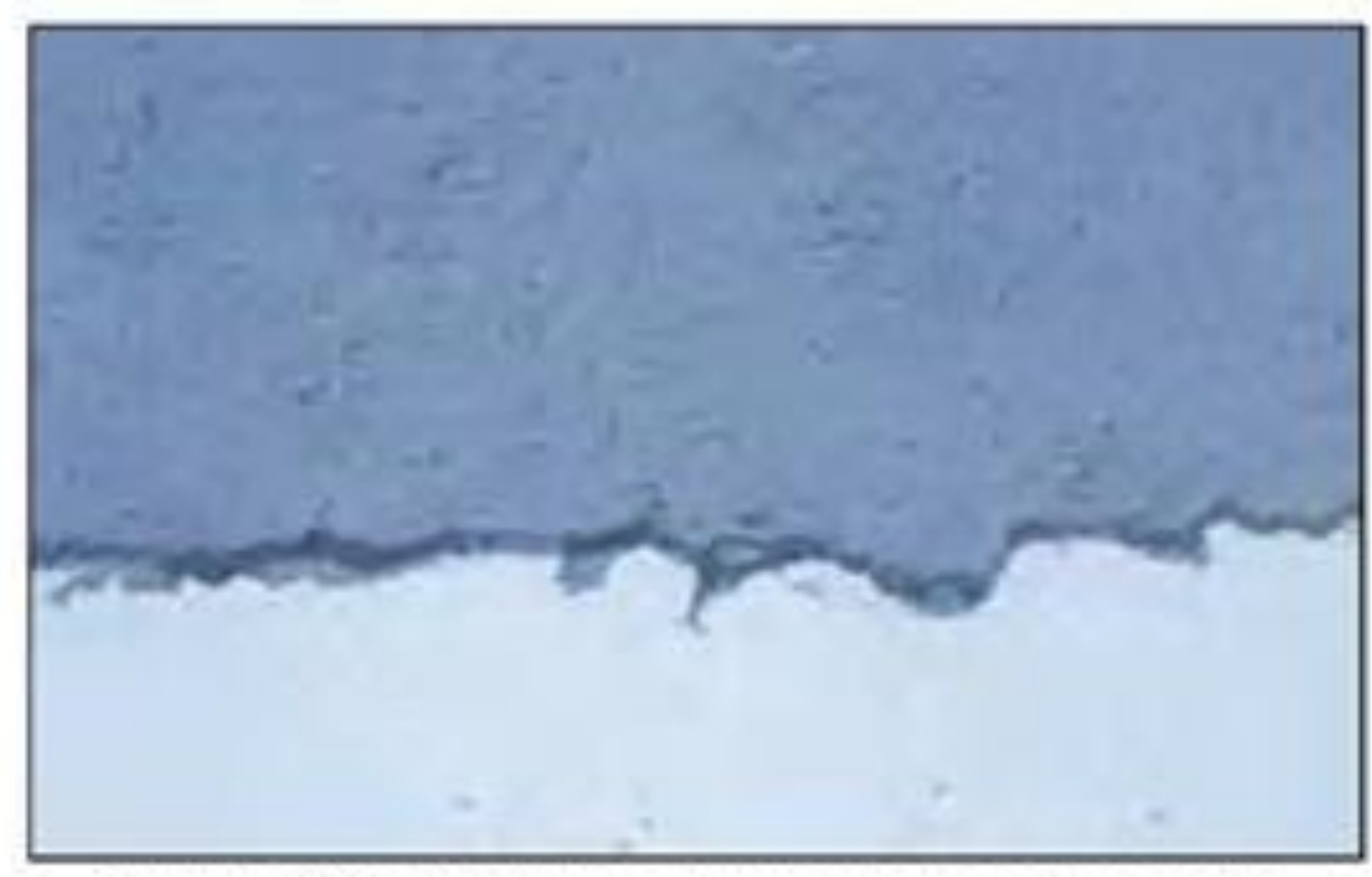
Preparation Artifacts

Cracks



Example: Crack between oxide layers and the steel substrate, Mag. 200 X.

Cracks



Example: Crack between plasma spray coating and the substrate. Mag. 500 X.

Preparation Artifacts
Cracks

- Cracks are fractures in brittle materials and materials with different phases. The energy used to machine the sample is greater than can be absorbed. The surplus energy results in the cracks. Cracks do occur in brittle materials and samples with layers. Care has to be taken through- out the complete preparation process. When cutting coated samples, the wheel should pass through the layer(s) first, so that the base material can act as support. Clamping of the sample should be carried out in a way that no damage can occur.

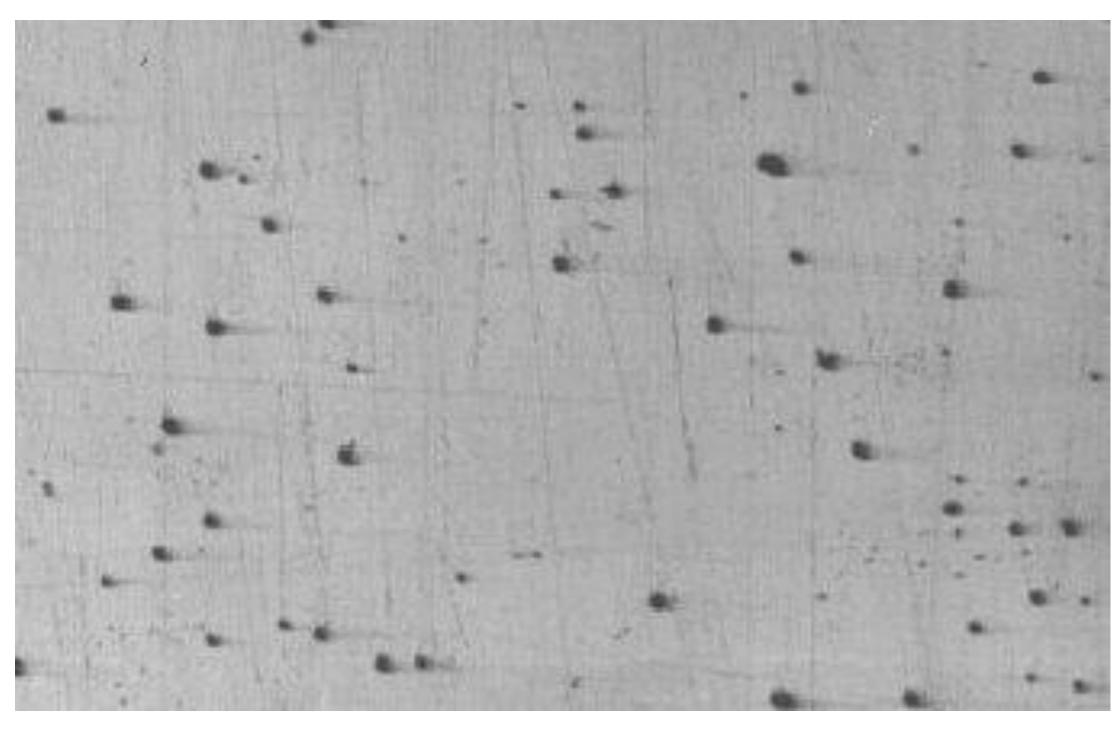
Preparation Artifacts

Cracks

- If necessary soft packing between sample and clamp has to be used. Mounting: for fragile materials, hot compression mounting should be avoid and cold mountig, preferably vacuum impregnation, should be used instead. Note: vacuum impregnation will only fill cracks and cavities connected with the surface.

Preparation Artifacts

Comet Tails



Example: Comet tails, Mag. 200 X.

Comet Tails

Example: Comet tails, Mag. 200 X, DIC.

Preparation Artifacts

Comet Tails

- Comet tails accur adjacent to inclusion or pores, when the motion between sample and polishing disc is undirectional. Their charcteristic shape earns the name "comet tails". A key factor in avoiding comet tails is the polishing dynamics. There are many variables in the metalographic polishing process. The most commonly recognized are items such as cloth and abrasives. While these gave a great effect on the polishing process, other critical parameters are often neglected.

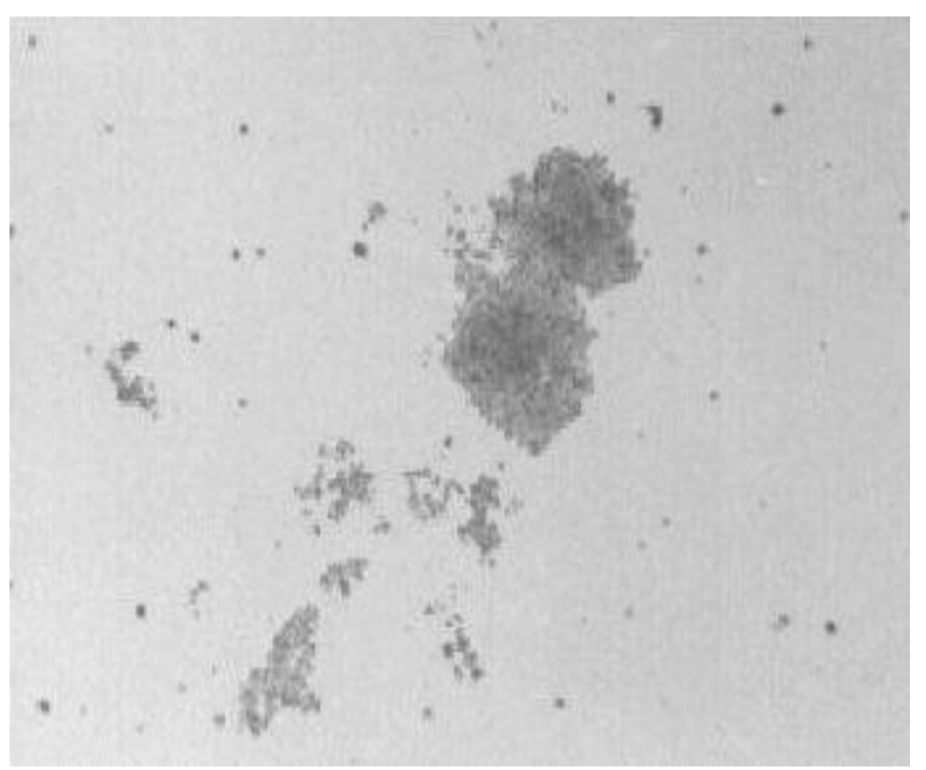
Preparation Artifacts

Comet Tails

- These parameters are the polishing dynamicss. The dynamics or speed of the sspecimen in relation to the cloth plays an important role in the final otcome of the polishing process. Artifacts such as commt tails, pull-outs, and edge rounding can be attributed to an imbalance in the dynamics.

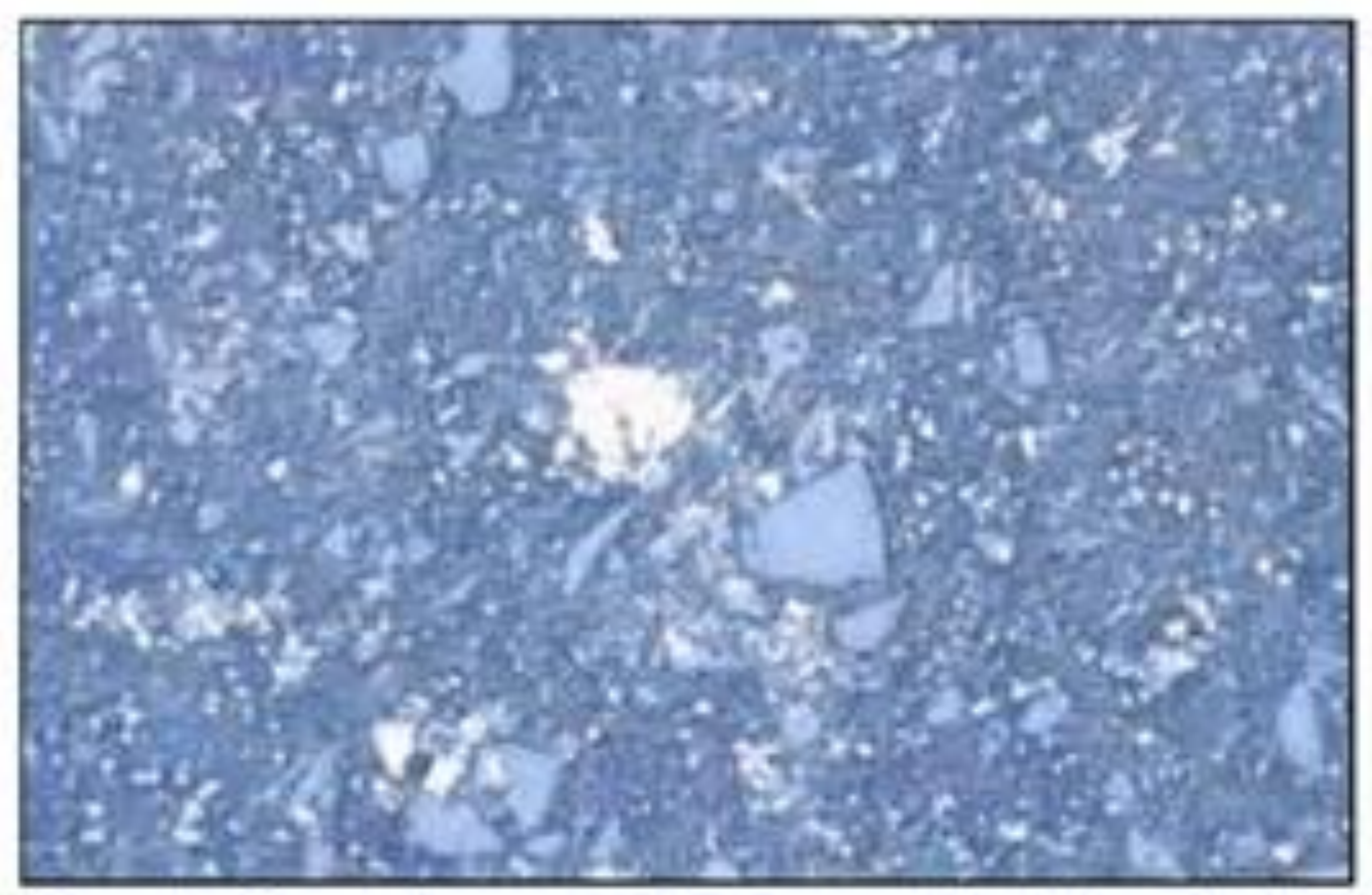
Preparation Artifacts

Contamination

- 

Example: Al_2O_3 particles from previous preparation is deposited on surface of the sample, Mag. 200 X.

Contamination



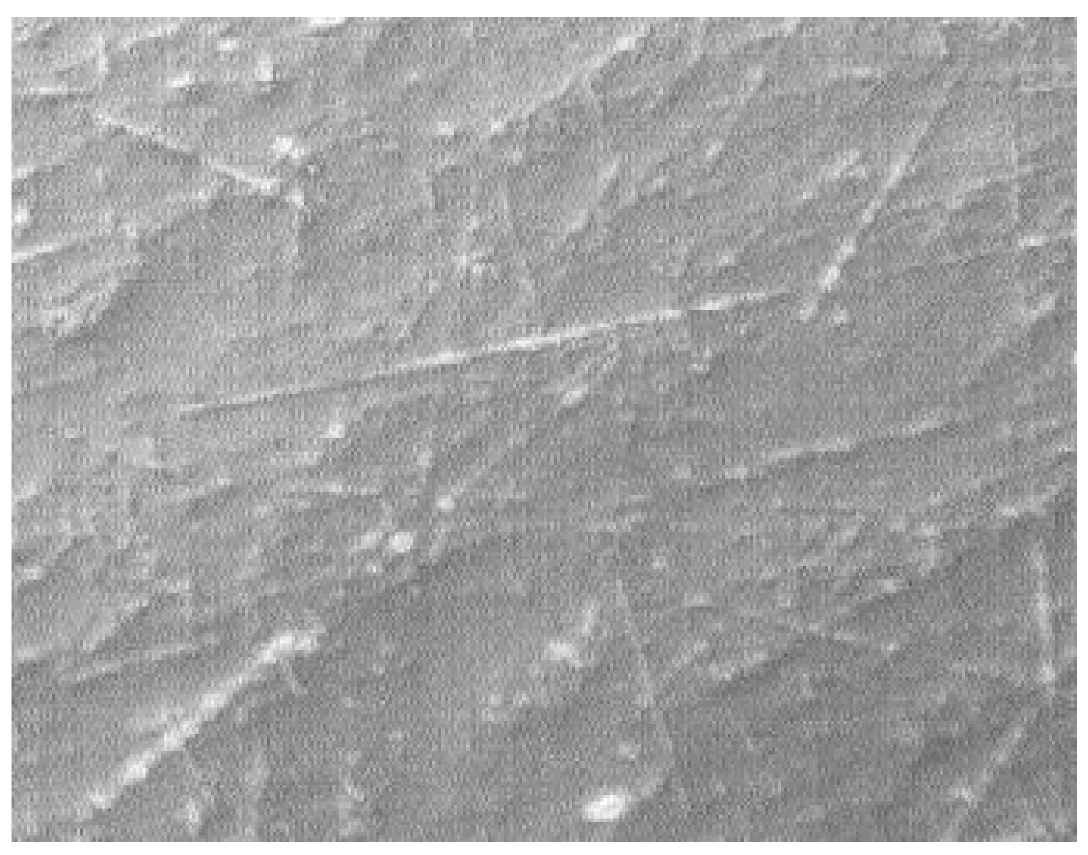
Example: Copper from a previous preparation is deposited on the surface of the sample, Mag. 200 X.

Preparation Artifacts
Contamination

- Material from a source other than the sample itself which is deposited on the sample surface during mechanical grinding or polishing is called contamination. Contamination can occur on all type of material. During polishing, possible dirt particles or material removed during a previous step can be deposited on the specimen. Microscopic examination can show "inclusions" or parts of a structure which should not actually be there.

Preparation Artifacts

Deformation



Example: Sharp deformation lines on sample, Mag. 200 X.

Deformation

Example: Sharp deformation lines on sample, Mag. 200 X, DIC.

Preparation Artifacts
Deformation

- There are two types of deformation, elastic and plastic. Elastic deformation dissappears when the applied load is removed. Plastic deformation may also be refered to as cold work. It can result in subsurface defects after grinding, lapping or polishing. Remaining plastic deformation can first be seen after polishing. Only deformation introduced during the preparation is covered here.

Preparation Artifacts

Deformation

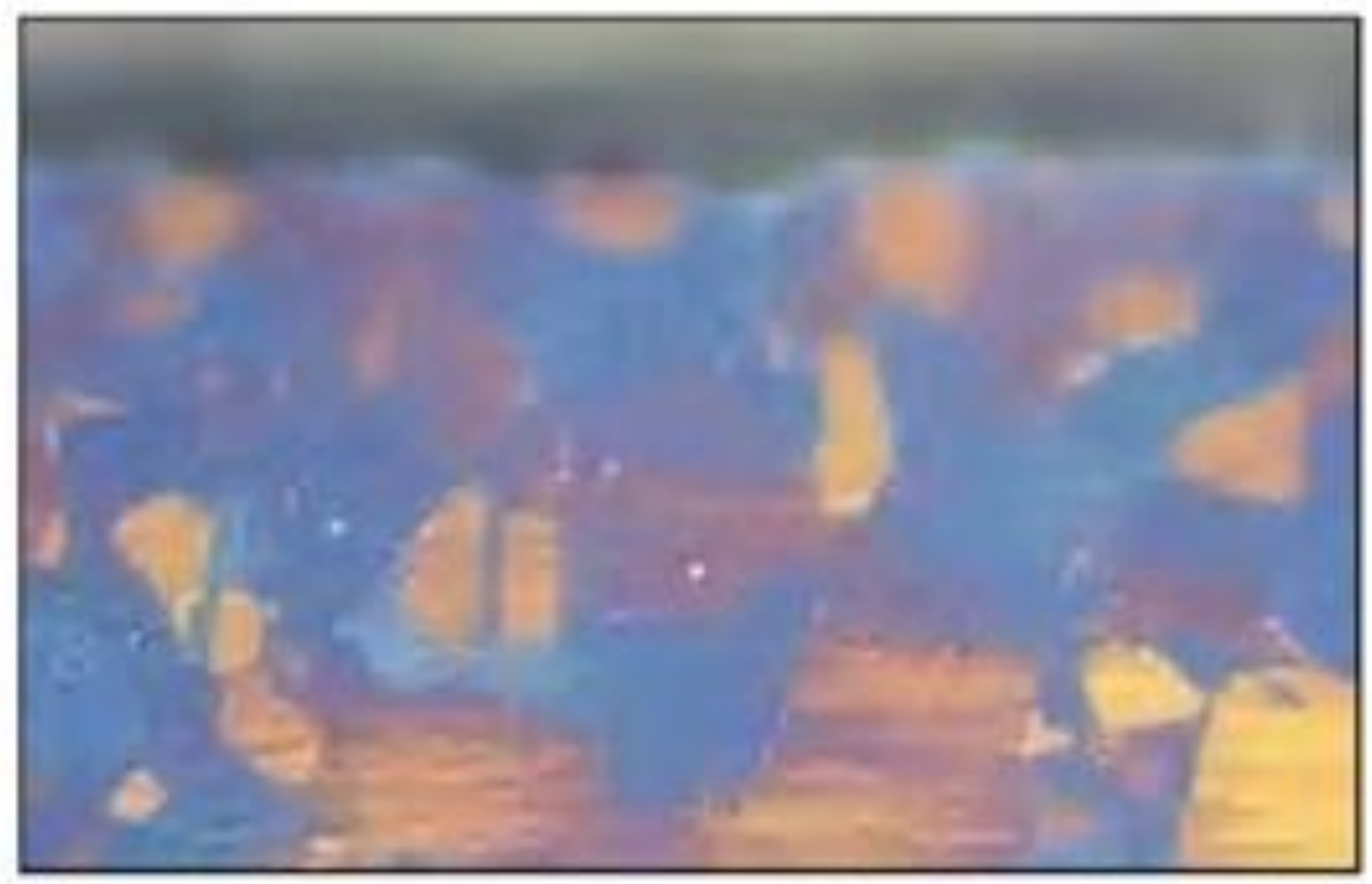
- All others types from previous operations like bending, drawing and stretching is not considered, bacause it can not be changed or improved by changing the preparation method. Deformations are artifacts which first show up after etching (chemical, physical or also optical etching).

Preparation Artifacts

Edge Rounding

Example: Due to a gap between resin and sample the edge is rounded, Mag. 500 X.

Edge Rounding



Example: Due to a gap between resin and sample the edge is rounded, Mag. 500 X.

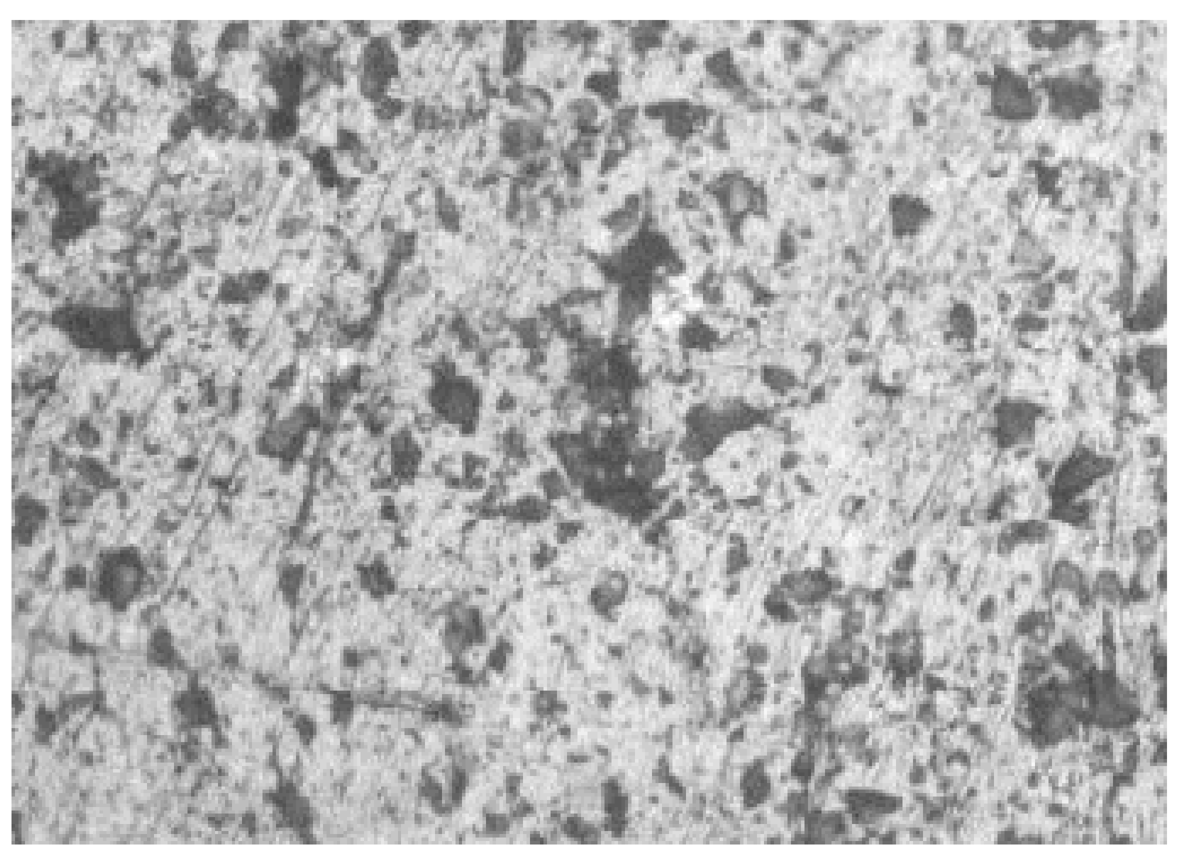
Preparation Artifacts

Edge Rounding

- Using a polishing surface with high resilience will result in material removal from both the sample surface and around the sides. The effect of this is edge rounding. With mounted specimens this effects can be seen, if the wear rate of the resin is higher than that of the sample material.

Preparation Artifacts

Embedded Abrasive



Example: Diamond particles embeded in the sample, Mag. 200 X.

Embedded Abrasive

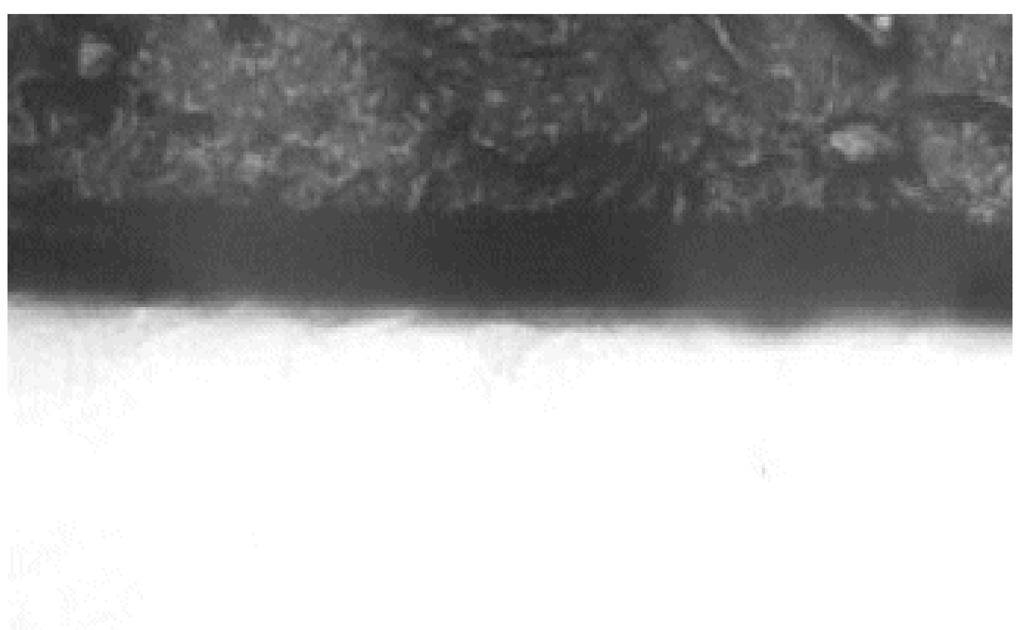
Example: Diamond particles embeded in the sample, Mag. 500 X.

Preparation Artifacts
Embedded Abrasive

- Loose abrasive particles pressed into the surface of the sample. With soft materials, abrasive particles can become embedded. That happen because of these reasons: Too small abrasive particle size, disc/cloth used for grinding or polishing with too low resilience or a lubricant with too low a viscosity. Often a combination of these reasons take place.

Preparation Artifacts

Gaps



Example: Gap between resin and sample, Mag. 200 X.

Gaps

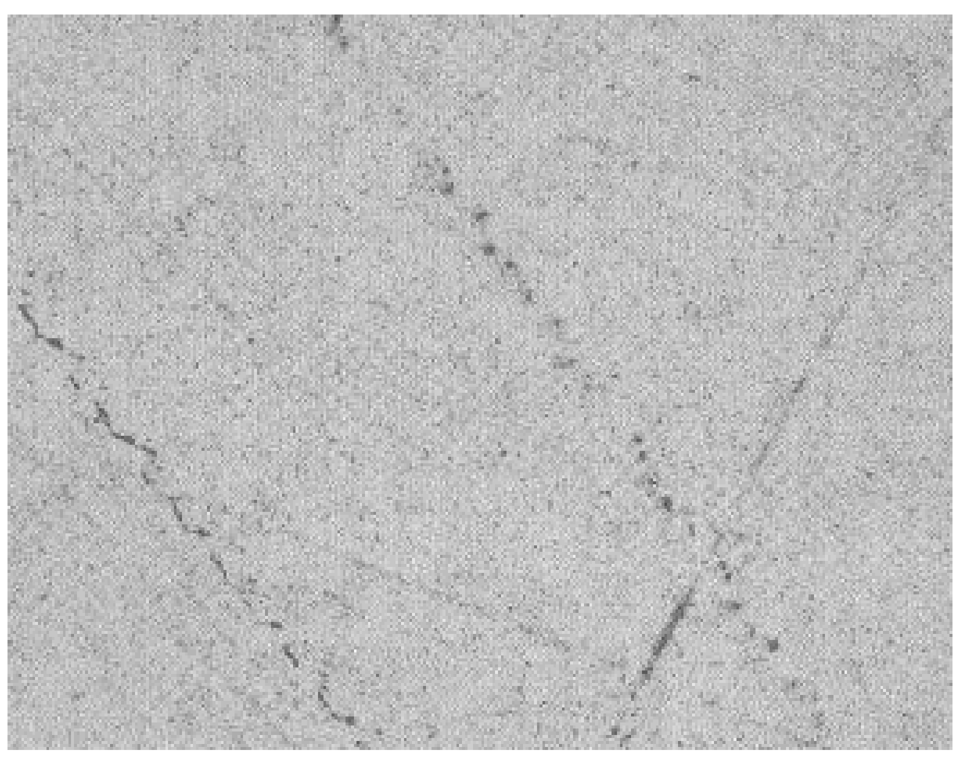
Example: Gap between resin and sample, Mag. 200 X.

Preparation Artifacts
Gaps

- Gaps are voids between mounting resin and sample material.

Preparation Artifacts

Lapping Tracks



Example: Lapping tracks, Mag. 200 X.

Lapping Tracks

Example: Lapping tracks on Zicalloy, Mag. 200 X.

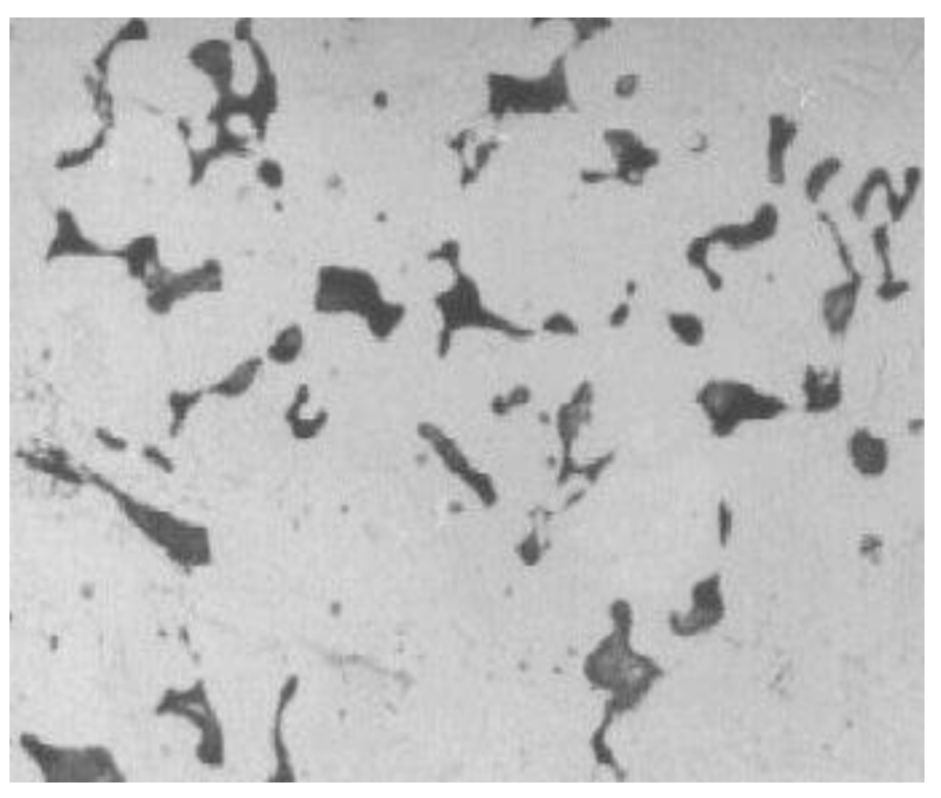
Preparation Artifacts

Lapping Tracks

- These are indentations on the sample surface made by abrasive particles moving freely on a hard surface. There are no scratches, like from a cutting action. Instead, there are the distinct tracks of particles tumbling over the surface without removing material. If abrasive particle is not held in a fixed position while the sample is passing over it, it will start rolling. Instead of removing, cuttingm chips of matrial, the grain is forced into the sample material, creating deep deformation, and only pounding small particles out of the sample surface.

Preparation Artifacts

Porosity



Example: Porosity in the Fe sintered alloy, Mag. 200 X.

False Porosity

Example: Superalloy after 5 min polishing, Mag. 500 X.

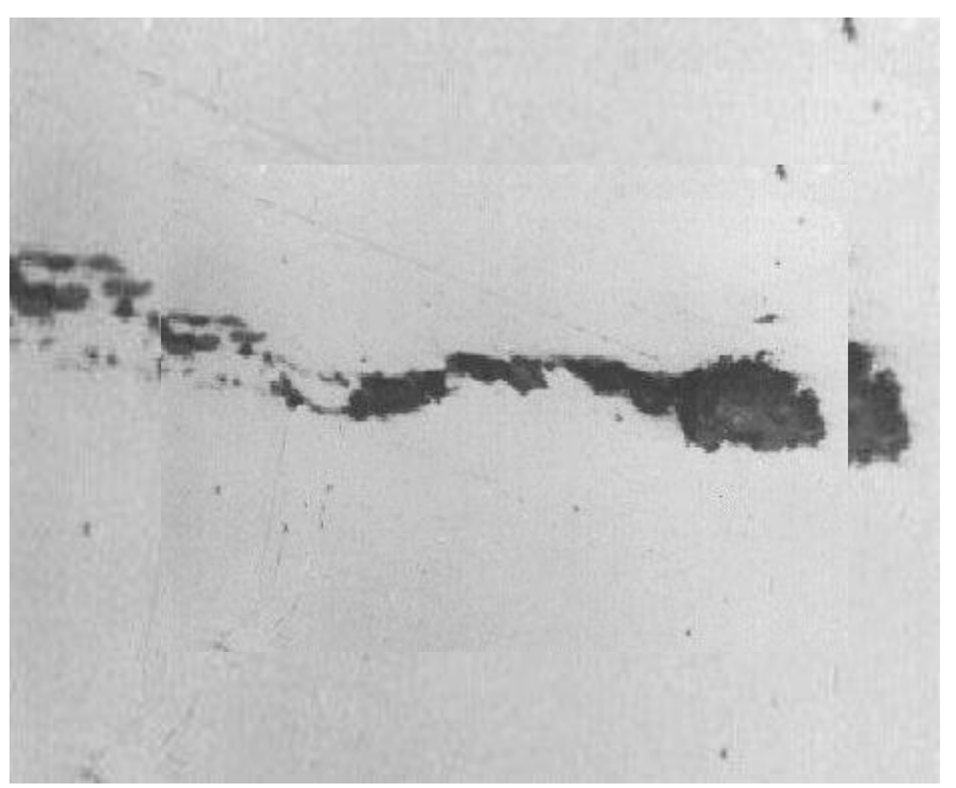
Preparation Artifacts

Porosity

- Some materials have natural porosity, e.g.cast metals, spray coatings or ceramic. It is important to get the correct values, and not wrong readings because of preparation faults. Depending on the properties of a material, two contrary effects regarding porosity can be seen. Soft and ductile materials can be deformed easily. Therefore pores can be covered by smeared material. Hard, brittle materials often get fractured at the surface during the first mechanical preparation steps. Contrary to the ductile material, where the initial porosity seems to be low and the pores have to be opened, brittle materials seem to have a high porosity. The apparent fracturing of the surface has to be removed.

Preparation Artifacts

Pull-Outs



Example: Inclusions pulled out, Mag. 100 X.

Pull-Outs

Example: Inclusions pulled out. Scratches orginating from the pulled out inclusions can be seen, Mag. 100 X, DIC

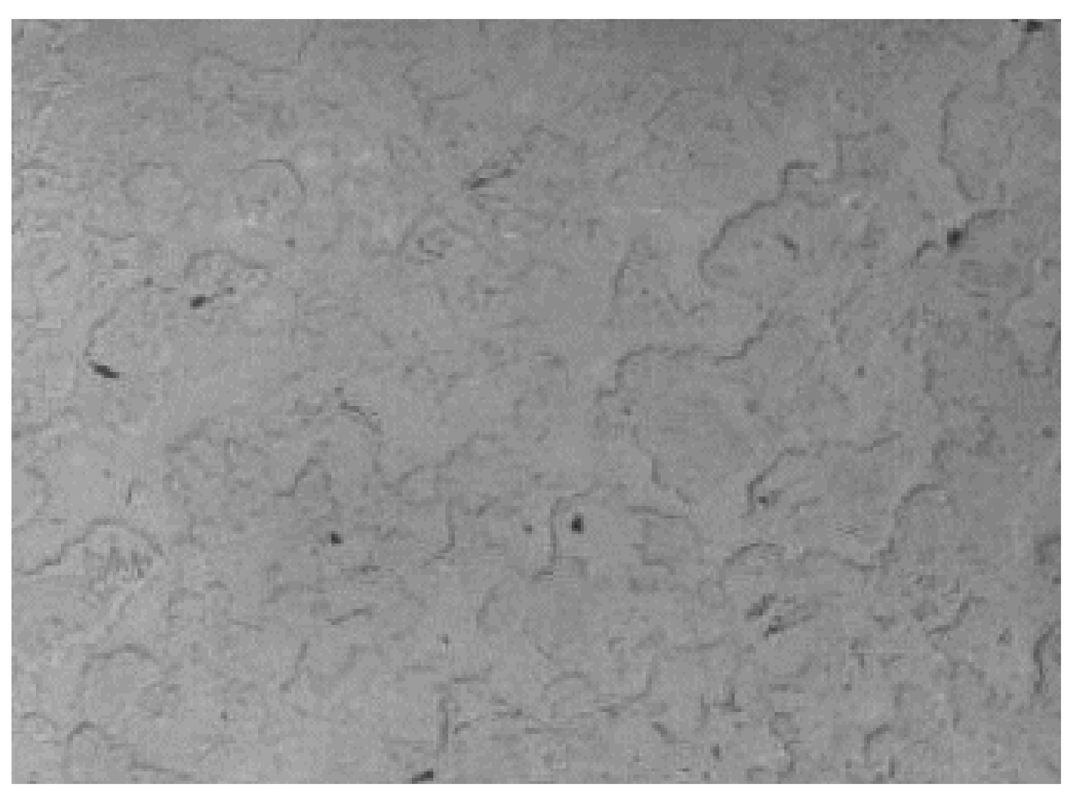
Preparation Artifacts

Pull-Outs

- Pull-outs are the cavities left after grains or particles which are torn out of the sample surface during abrasion. They are found in hard and brittle materials, and in materials with inclusions. Hard or brittle materials can not be deformed plastically, so small parts of the surface material shatter and may fall out or be pulled out by the polishing cloth. Inclusions may also be brittle ir have other thermal expansion values than the matrix. In this case, the relatively loose or broken inclusions can be pulled out by long naped polishing cloth.

Preparation Artifacts

Relief



Example: Polishing relief, Mag. 200 X.

Relief

Example: B4C fibres in AlSi, relief between fibres and base material, Mag. 200 X.

Preparation Artifacts

Relief

- Material from different phases is removed at different rates, due to varying hardness or wear rate of the individual phases. Relif does normally first occur during polishing. The most important parameters to avoid relief are preparation time and polishing cloth. The preparation time should be kept as short as possible. When developing a new method the samples have to be checked in short intervals, 1-2 min. The polishing cloths have a strong influence on the planeness of the sample. Polishing cloth with low resilience produces samples with les relief than cloth with high resilience.

Preparation Artifacts

Scratches

Example: Scratches on steel sample, Mag. 100 X.

Scratches

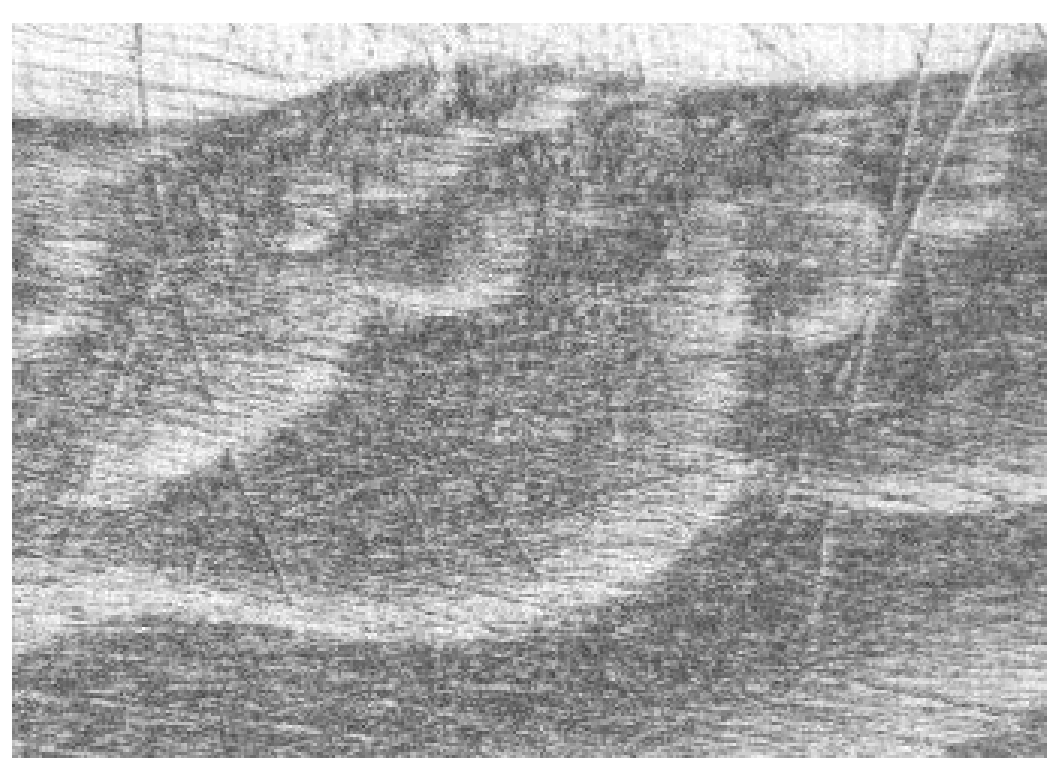
Example: After diamond polishing, scratches from grinding still remain, Mag. 200 X.

Preparation Artifacts
Scratches

- Stratches are grooves in the surface of a sample, produced by points of abrasive particles.

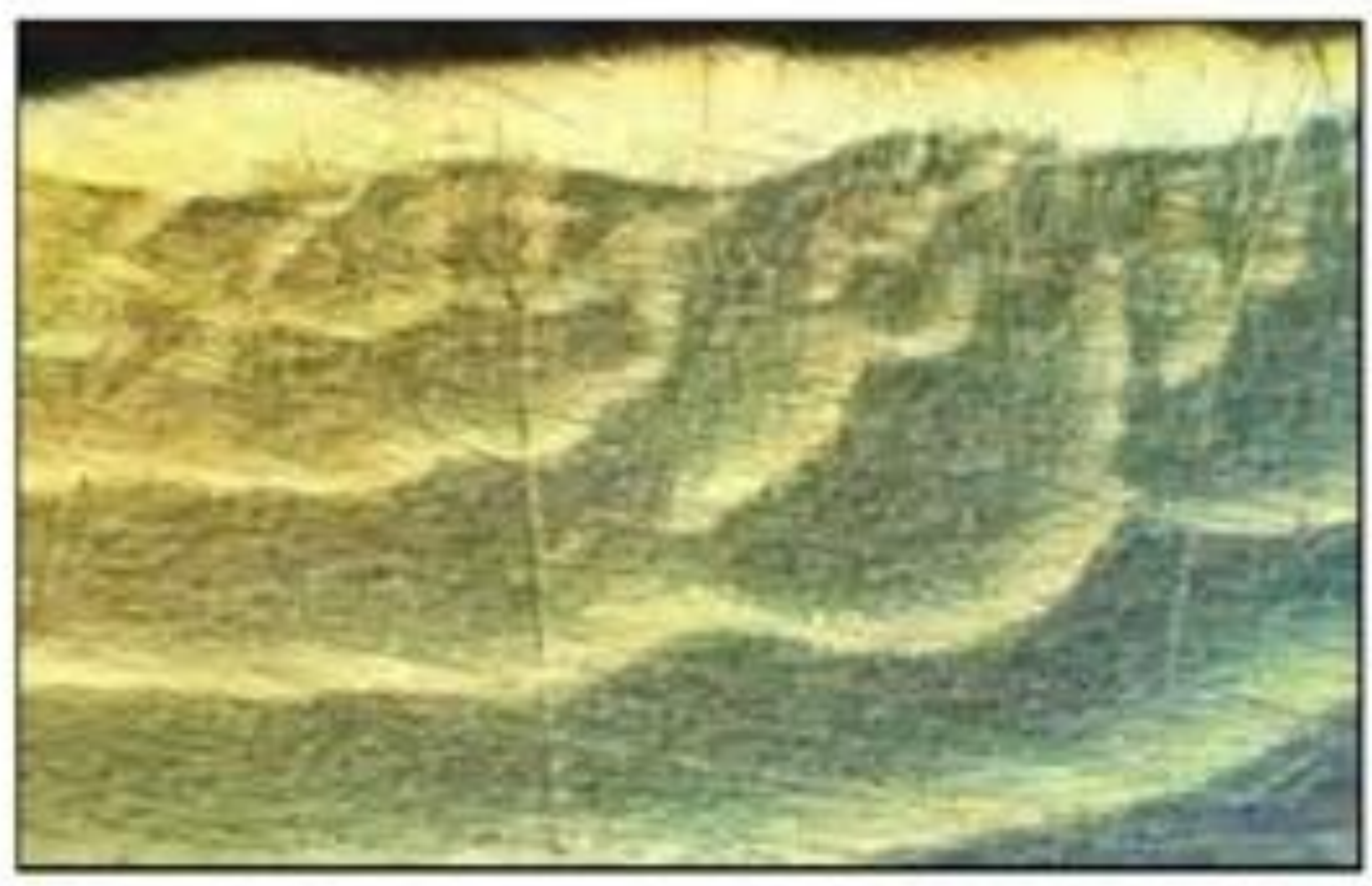
Preparation Artifacts

Smearing



Example: Smearing on soft ductile steel, Mag. 50 X.

Smearing



Example: Smearing on soft ductile steel, Mag. 15 X, DIC

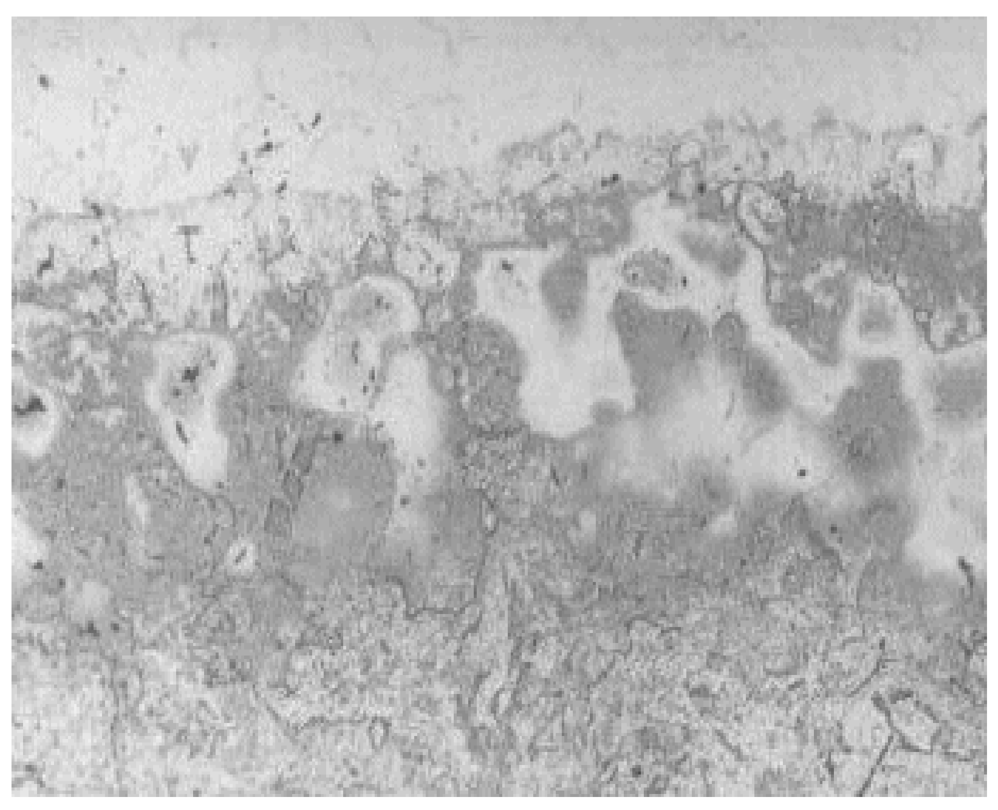
Preparation Artifacts

Smearing

- The plastic deformation of lager sample areas is called smearing. Instead of being cut, the material is pushed, moved across the surface. The reason is an incorrect application of abraive, lubricant or polishing cloth, or a combination of these, which makes the abrasive act as if it was blunt.There are three possibilities to avoid smearing:1.Lubricant: check the amount of lubricant, smearing often occurs at too low a lubricant level. If increase amount of lubricant. 2. Polishing cloth: Due the high resilience of th cloth the abrasive can be pressed too deep into the cloth and can not cut. Change cloth with lower resilience. 3. Abrasive: the diamond grain size might be to small, the prticles can penetrate into the material and because of that can not cut.

Preparation Artifacts

Staining



Example: Stainning of sample, Mag. 100 X.

Staining

Example: Staining of sample due to gaps between resin and sample, Mag. 20 X.

Preparation Artifacts

Staining

- Staining is a discoloration of the sample surface, tipically because of contact with a foreign body. Staining is often seen after claning or etching samples. There is a gap between sample and resin, water, alcohol or etchant can bleed out of the gap during drying or even on the microscope. Areas on the sample surface can be discolored, and make the examination difficult or even impossible.

Preparation of Ceramics

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

Preparation of Ceramics

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

Preparation of Electronic Materials

- The term, ‘microelectronic materials’ encompasses an extremely wide range of materials. This is due to the fact that most microelectronic devices are composites, containing any number of individual components. For example, present day microprocessor failure analysis might require the metallographer to precisely cross section through a silicon chip plated with multiple thin-film layers of oxides, polymers, ductile metals such as copper or aluminum, and refractory metals such as tungsten and/or titanium-tungsten. In addition, the packaging of such a device might contain materials of such varying mechanical properties as toughened aluminum oxide and solder.

Preparation of Electronic Materials

- The solder materials may have compositions ranging up to 97 % lead. With such a vast number of materials incorporated into a single device, and with these materials having such highly disparate mechanical properties, it is virtually impossible to develop a general method for achieving perfect metallographic results. Instead, we must focus on a few individual materials, and develop a philosophy of preparation in which we give our attention specifically to the materials of interest.

Preparation of Electronic Materials

- First and foremost in the class of ‘microelectronic materials’ is silicon. Silicon is a relatively hard, brittle material, which does not respond well to grinding with large silicon carbide abrasives. Silicon carbide papers contain strongly bonded abrasive particles which, when they collide with the leading edge of a piece of silicon, create significant impact damage. In addition, they create tensile stresses on the trailing edge of silicon, which results in deep and destructive cracking. Cutting close to the target area is preferable to grinding, but to accurately approach the target area within a silicon device, fine grinding is still necessary.

Preparation of Polymers

- Plastics and polymers are normally quite soft. Many different sectioning methods have been used. A sharp razor blade or scalpel, or even a pair of scissors, can be used. The precision saw produces excellent surfaces, while an abrasive cut-off saw yields a bit more damage and a rougher surface. Blades and wheels for sectioning polymers are available from the different manufacturers. Damage from sectioning is quite easy to remove.

Preparation of Polymers

- Surface quality can be degraded by abrasion from the debris produced during grinding and polishing. Mounted specimens are much easier to prepare than nonmounted specimens. Castable resins are preferred as the heat from a mounting press may damage or alter the structure of the specimen. However, there may be a visibility problem if a transparent, clear polymeric specimen is mounted in a clear, transparent epoxy. In this case, color resin, with its deep red color, will produce excellent color contrast between specimen and mount in darkfield or polarized light. Always use practices that minimize the exotherm during polymerization.

Preparation of Polymers

- Preparation of plastics and polymers for microstructural examination follows the same basic principles as for other materials. Rough grinding abrasives are unnecessary, even for the planar grinding step. Pressures should be lighter than used for most metals. Water is generally used as the coolant, although some plastics and polymers may react with water. In such cases, use a fluid that will not react with the particular plastic or polymer. Embedding can be a problem with plastics and polymers. ASTM E 2015 (Standard Guide for Preparation of Plastics and Polymeric Specimens for Microstructural Examination) describes additional procedures for preparing several types of plastics and polymers.

Preparation of Printed Circuit Boards

- The vast majority of printed circuit boards (PCB's) are of a rigid variety, the bulk of which are composed of layers of woven glass fiber cloth in a polymeric matrix. Flex circuits, which are becoming quite common, do not typically contain glass fiber, but instead, their bulk is often composed of layers of polyimide. The circuitry in both types of boards is composed of plated and/or foil metal. The metal used is generally copper, while in a few cases, gold and/or nickel plating may be present. Furthermore, depending upon whether the boards have undergone assembly or shock testing, solders of various compositions might also be present. Luckily for the metallographer, the variety of materials present in PCB's generally do not complicate the preparation methods due to the fact that extremely hard and brittle materials are not commonly found in the boards. This changes, however, when 'packed' boards with ceramic or semiconductor components must be sectioned.

Preparation of Sintered Carbides

- Sintered carbides are very hard materials made by the powder metallurgy process and may by reinforced with several types of MC-type carbides besides the usual tungsten carbide (WC). The binder phase is normally cobalt although minor use is made of nickel. Modern cutting tools are frequently coated with a variety of very hard phases, such as alumina, titanium carbide, titanium nitride and titanium carbonitride. Sectioning is normally performed with a precision saw, so surfaces are very good and rough abrasives are not usually required.

Preparation of Thermally Spray Coated Specimens

- Thermally sprayed coatings (TSC) and thermal barrier coatings (TBC) are widely used on many metal substrates. Invariably, these coatings are not 100 % dense but contain several types of voids, such as porosity and linear detachments. Hot compression mounting is not recommended as the molding pressure can collapse the voids. Use a low-viscosity castable epoxy and use vacuum infiltration to fill the connected voids with epoxy. Fluorescent dyes, may be added to the epoxy. When viewed with fluorescent illumination, the epoxy-filled voids appear bright yellow green. This makes it easy to discriminate between dark holes and dark oxides, as would be seen with bright field illumination. Filling the pores with epoxy also makes it easier to keep the pore walls flat to the edge during preparation. Aside from this mounting requirement, TSC and TBC specimens are prepared using all of the factors needed for good edge retention.

Electrolytic Polishing
Problem Solver

- **Defect: The centre of the specirnen is deeply attacked.**
  Probable cause: polishing film did not form in the centre if the specimen.
  Suggest corrections:
  1. Increase the voltage
  2. Reduce stirring
  3. Use a more viscous electrolyte

Electrolytic Polishing
Problem Solver

- **Defect: Pitting and attack at the edge of the specimen.**
  Probable cause: Film too viscous or too thin.
  Suggest corrections:
  1. Decrease the voltage
  2. Increase stirring
  3. Use a less viscous electrolyte

Electrolytic Polishing
Problem Solver

- **Defect: Deposits on the surface.**
  Probable cause: Insoluble products of the anode.
  Suggest corrections:
  1. Choose a different electrolyte
  2. Raise the temperature
  3. Raise the voltage

Electrolytic Polishing
Problem Solver

- **Defect: Rough or matt surface.**
  Probable cause: Polishing film is inadequate.
  Suggest corrections:
  1. Raise the voltage
  2. Use a more viscous electrolyte

Electrolytic Polishing
Problem Solver

- **Defect: Undulations or scrateches on the polished surface.**
  Probable cause:
  1. Insufficient time
  2. Inadequate stirring
  3. Unsuitable preparation
  4. Time too long
  Suggest corrections:
  1. Increase or decrease stirring
  2. Improve the preparation
  3. Raise the voltage while decreasing the time

Electrolytic Polishing
Problem Solver

- **Defect: Stains on the polished surface.**
  Etch after switching off the current.
  Suggest corrections:
  1. Remove the specimen immediately when the current is switched off
  2. Choose a less active eleclrolyte

Electrolytic Polishing
Problem Solver

- **Defect: Places that have not been polished.**
  Probable cause: Gas bubles.
  Suggest corrections:
  1. Increase stirring
  2. Decrease the voltage

Electrolytic Polishing
Problem Solver

- **Defect: Phases in relief.**
  Probable cause: Polishing film is inadequate.
  Suggest corrections:
  1. Raise the coltage
  2. Improve the preparation
  3. Reduce the time

Electrolytic Polishing
Problem Solver

- **Defect: Pitting.**
  Probable cause:
  1. Time too long
  2. Voltage to high

  Suggest corrections:
  1. Improve the preparation
  2. Lower the voltage
  3. Decrease the time
  4. Try various electrolytes

Suggestions for Handling Hazardous Materials

- All chemicall, including many metals and oxides, pose some degree of danger to the human organism. This may come about by ingestion through the respiratory or digestive tracts or by external contact with the skin or eyes. Basically, the same precautions apply to the metallographic laboratory as to all chemical laboratories, except that certain specific areas are particularly critical. Some significant precautions are:

Suggestions for Handling Hazardous Materials

- Clearly label all storage containers.
- Dilute concentrated chemicals before disposal and observe all local waste-disposal regulations.
- Critical substances (flammable, explosive, toxic, or corrosive) should be stored in approved containers in cool, fireproof, isolated areas.

Suggestions for Handling Hazardous Materials

- Caustic materials, such as acids, bases, peroxides, and some salts should be handled only when wearing protective devices such as safety glasses, rubber gloves, and laboratory coats or aprons. Vapors of such materials are often harmful, too. Actual work should be carried out in an effective fume hood with an additional gas mask if evolution of toxic gases and vapors is suspected

Suggestions for Handling Hazardous Materials

- When preparing etchants containing aggresive chemicals such as sulfuric acid, the chemical should always be added to the solvent (water, alcohol, etc.) slowly with gentle stirring. External cooling may also be required if haet evolution is particularly strong.

- Volatile, flammable and explosive materials, such as benzone, acetone, ether, perchlorate, nitrate, etc. should not be heated or kept near open flames.

Suggestions for Handling Hazardous Materials

- When preparing microsections of toxic materials such as beryllium, and radioactive substances or alloys conrtaining uranium, thorium and plutonium, a glove box or hot cell must be used.

Suggestions for Handling Hazardous Materials

- Perchloric acid in concentration exceeding 60% is highly flammable and explosive. This danger is greatly increased by the presence of organic materials such bismuth, which oxides readily. Avoid the high concentration and heating of these solutions, particularly in electrolytic polishing and etching; never store high-concentration perhloric acid in plastic containers.

Suggestions for Handling Hazardous Materials

- When mixing perchloric acid and alcohol, highly explosive alkyl perchlorates may form. Perchloric acid should be added slowly under constant stirring. Keep the temperature of the solution below 35 degrees of Celsius and, if necessary, use a coolant bath. Wearing safety glasses is helpful, but working behind a safety shield is preferable.

Suggestions for Handling Hazardous Materials

- Mixtures of alcohol and hydrochloric acid can react in various ways to produce aldehydes, fatty acids, explosive nitrogen compounds, etc. The tendency toward 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.

Suggestions for Handling Hazardous Materials

- Mixtures of alcohol and phosphoric acid can result in the formation of esters, some of which are potent nerve poisons. If absorbed through the skin or inhaled, serve personal damage may result.

Suggestions for Handling Hazardous Materials

- Mixtures of methanol and sulfuric acid may form dimethylene sulfate, an odorless, tasteless compound that may be fatal if absorbed in sufficient quantities into skin or respiratory tract. Even gas masks do not offer adequate protection. Sulfates of their higher alcohols, however, are not potentially dangerous poisons.

Suggestions for Handling Hazardous Materials

- Mixtures of chromium (VI) oxide and organic materials are explosive. Mix with care and do not store.

- 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.

Suggestions for Handling Hazardous Materials

- All cyanide compounds (CN) are highly dangerous becaouse hydrocyanic acid (HCN) may easily form. They are fast-acting poisons taht can cause death, even in relatively low concentrations.

Suggestions for Handling Hazardous Materials

- 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 skin do not heal readily. Hydrofluoric acid also attack glass, and fumes from specimen etched in HF solution could easily damage front element of microscope lenses. Specimen should be rinsed throughly and some cases placed in a vacuum desiccator for one or two hours before examination.
- Picric acid anhydride is an explosive.

ASTM Sample Preparation Standards

E7-96 Metallography

E3-95 Preparation of Metallographic Specimens

E340-95 Macroetching Metals and Alloys

E381-94 Macroetch Testing Steel Bars, Billets, Blooms, and Forgings

E807-96 Metallographic Laboratory Evaluation

A561-71(1985) Macrotech Testing of Tool Steel Bars

A604-93 Macroetch Testing of Consumable Electrode Remelted Steel Bars and Billets

F1049-95 Shallow Etch Pit Detection on Silicon Wafers

ASTM Sample Preparation Standards

F1404-92 Crystallographic Perfection of Gallium Arsenide by Molten Potassium Hydroxide (KOH) Etch Technique

F416-94 Detection of Oxidation Induced Defects in Polished Silicon Wafers

F47-94 Crystallographic Perfection of Silicon by Preferential Etch Techniques

F80-94 Crystallographic Perfection of Epitaxial Deposits of Silicon by Etching Techniques

F950-88(1993)e1 Measuring the Depth of Crystal Damage of a Mechanically Worked Silicon Slice Surface by Angle Polishing and Defect Etching

D5671-95 Polishing and Etching Coal Samples for Microscopical Analysis by Reflected Light.

Etching and Other Terms

Abrasive Clean

Dry or wet abrasive powders used as a method of cleaning material surfaces. Applied as dry powder uder gas pressure. SiC is a major compound for general processing.

Abrasive

The abrasive used for grinding and polishing.

Acid Etch

Controlled preferential attack by acid on a metal surface for the propose of revealing structural details.

Acid Slurry Etch

A form of mechanical polish lapping where an acid is mixed an abrasive.

Adhesion Etch

Etching of thin films to relieve stress and evaluate the film failure, as lifting, peeling crazing, etc.

Aged Etch

Has two meanings: 1.) any solution mixed and allowed to stand for a specified period of time, 2.) any solution used more than on time may be called an aged or used solution.

Agitation Etch

Any solution used with some form of physical movement, for example: rod in solution, hand or electrically operated (rotational stirring).

Alcohol Etch

Alcohols are normally considered cleaning or rinsing solutions rather than etchants, but they will attack certain alcohol soluble materials.

Alkali Etch

Alkaline solutions are greater than pH 7, are base or basic chemical solutions according to Sorenson scale. They mey be in liquid form or in solid form. Alkalies also are used as molten fluxes for preferential etching, such as on silicon, or as growth for high temperature type single crystals, such as garnets and ferrites.

Alloy Etch

Any solution used to clean an alloyed joint or structure, such as water for flux removal. The use of an alloy material acting as the etching agent, such as an AlSi (5%) for froming a pit in a single crystal wafer.

Air Etch

Alternative therm for atmospheric atch. Actual ecthing is due oxygen, salt (chlorine), water moisture, or other contaminants in the air such sulfur, acids, smog.

Angle Etch

There are two meanings: 1.) specimen: the part is lapped at a secified angle an then etched. Used to measure diffusion depths, or 2.) containers: the etch conatiner is placed on a rotating spindle. Specimen is mounted on discs of Teflon, placed face-up on container bottom, wuth ething done at rotation speeds of between 130 to 300 rpm.

Anisotropic Etch

Another term for preferential etch. Any etch that attacks crystallographic planes at different rates.

Anodic Etch

Electrolytic etching with the specimen as the anode for cleaning, removal, polishing, structuring and may include switching from anode-to-cathode. Development of microstructure by selective dissolution of the polished surface under application of a direct current.

Argon Etch

Primary used to: 1.) Argon ion, Ar+, cleaning or etching of surfaces, secondary for etching of single crystal argon.

Artifact

A false microstructural feature that is not an actual characteristic of the specimen; it may be present as a result of improper or inadequate preparation, handling methods, or optical condition for viewing.

Atmosphere Etch

Air, nominally 24% oxygen and 75% nitrogen with water vapor, contaminating gases will attack all known inorganic materials with time.

Attack-Polishing

Simultaneous etching and mechanical polishing.

Atomizer Etch

Use of an atomizer to apply a fine mist/spray to etch or clean a surface.

Autoclave Etch

A closed metal container capable of handling high pressure (low pressure, medium pressure, high pressure, cryogenic).

Barrel Etch

A barrel-like container or bottle for cleaning and etching, used closed and rotated horizontally on roller bars, open-topped and mounted on a rotationg spindle similar to an angle ecth beaker, or close-topped on a shaker table.

Base Etch

Therm used in three ways: 1.) Any chemical solution with pH greater than pH 7 is chemical base. 2.) The first etch solution used in a clean/etch sequence or the primary solution in such sequence. 3.) A specific etch mixture with established characteristics against which other solutions are evaluated.

Basic Etch

Any solution with pH > 7 as established hydroxyl-ion concentration. The chemical compound is a base as against an acid.

Basket Etch

The etching of wafers, dice or other parts in some form of a basket holder that is submerged in an etch solution; passed through an etch solution; held in hot vapor, or held for spraying.

Batch Etch

The etching or cleaning of any two or more wafers, specimens or parts in a solution at one time.

Beaker Etch

Any open-topped conatiner used to hold an etch or cleaning solution. Quartz, Pyrec, polyethylene, or Telflon beaker from 200 to 1000 ml are common.

Blank Etch

Any etch solution used on a relatively small flat part, such as 1 x 1 x 0.010 inch ceramic or metal substrate. Also called a flat or coupon.

Boiling Bead Etch

Ceramics, glass, or metal beads of various sizes placed in the bottom of an etch beaker to introduce and control a boble/agitation action of a hot to boiling solution.

Bomb Etch

A metal conatiner capable of holding higher pressurem, and used to hold an etching or cleaning solution/gas.

Bottle Etch

Use as a closed bottle for etching.

Bright Field Illumination

Bright Field (BF) illumination is the most common illumination technique for metallographic analysis. The light path for BF illumination is from the source, through the objective, reflected off the surface and 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 an angle.

Brine Etch

Natural or artificial salt water used as an ecth solution. Not common; some test evaluation on metals and compunds in corrosion study.

Brush Etch

To use of a brush to apply an etch solution to a material surface.

Bulk Etch

After mechanical lap and polish of a material surface, the surface is etched to remove the residual subsurface damage down to the undamaged bulk metal.

Cap Etch

Special terms spplied to Solid State processing and etching single crystals with thin films.

Cascade Etch

Though often called etching, more commonly a method of dinal water quenching and washing following ann etch period, with reference to the container structure used: a rectangular trough divided into three or four progressively lower sections, usually fabricated from polyethylene sheet or simular high purity plastic.

Cathodic Etch

Electrolytic etching is normaly anodic, but switching from anode to cathode as a deplating and removal system, not an actual method of etching. In metallography is cathodic etching: Surface removal by bombarding with accelerated ions (1-10 kV) in vacuum.

Caustic Etch

An alkali or hydroxide solution with pH > 7.

Centrifuge Etch

Rapid spinning motion of a part during etching or cleanning. A photo resist spinner has been used for acetone cleaning during photo resist application and etching of semiconductor wafers with the holder platen rotating at about 3500 rpm.

Chemical Etch

General expression for all developments of microstructure through reduction and oxidation process (redox reactions).

Chem/Mech Etch

Chemical/Mechanical etching is being more and more widely used in a single crystal wafer processing to remove residual subsurface damage introduced by previous cutting, lapping or polishing steps.

Channel Etch

The etching of a groove into surface. Used in selective structuring of a single crystal devices.

Cleave Etch

The etch polishing or cleaved single crystal wafer surface to remove residual cleavage steps.

Cleaning Etch

Any solution or gas used to clean a surface with minimum etch removal of the material.

Cold Etch

Any solution used between about 2-10 ^{o}C. Development of microstructure at room temperature and below.

Conditioning Etch

Any solution used to prepare a surface for subsequent processing. Very common in the plating of metals, such as zincating aluminium before metal deposition.

Contamination Removal Etch

Any solution used to clean a surface of unwanted solid material, such as dirt, oil, or grease, etc.

Control Etch

Any etching for specified time period or at a particular temperature.

Controlled Etch

Electrolytic etching with selection of suitable etchant and voltage resulting in a balance between current and dissolved metal ions.

Cool Etch

Any solution used between about 10-20 ^{o}C.

Corrosion Etch

The attack or alteration of any material surface from the action of a solid, liquid or gas.

Coupon Etch

Etching of a specimen cut as a flat form from sheet material.

Cover Etch

The part to be etched is slowly lowered into the BRM (bromine-methanol) solution, etched, then slowely withdrawn through the pure methanol "cover" to quench etching action without exposure to air.

Crucible Etch

The use of graphite, ceramic, or high temperature metals, such as platinum, as a cup to hold an etching solution.

Cryogenic Etch

The use of cryogenic liquids as an etching medium, such as liquid chlorine, at - 102 °C. Also refers to the use of cryogenic liquid as a chilling agent for other acid solutions, such as liquid nitrogen.

Crystal-Figure Etch

Discontinuity in etching depending on crystal orientation. Distinctive sectional figures form at polished surface. Closely related to dislocation etching.

Crystallographic Etch

Any solution that will develop single crystal plane structure by preferential attack. Specific solutions have been developed on most metals and metallic compounds during evaluation development, and include etching spheres (convex), and pits (concave) surfaces.

Cup Etch

Any open container when used to hold an etching solution may be referred to as a cup. Usually a small ceramic, graphite, or high temperature metal (Pt, Mo, Ti, Ta) used as a crucible for either an etch solution or solid molten metal, and may include a cap or cover.

Cutting Etch

The use of an etch solution, alone, or a wire soaked in the etch to cut material.

Cutting Force

The specific pressure between sample surface and abrasive grain surface.

Cyclic Etch

Etching with sequential periods of time. May include water or alcohol quenching between etch cycles, or the removal to air and return to etch without quenching.

Damage Removal Etch

Any etch solution used to remove either the surface or subsurface damage present or induced by previous abrasive lapping, etc. It is usually a slow polish type etch, such as bromine: methanol that both remove the damaged zone and polish simultaneously.

Decoration Etch

The use of a metal thin film diffused or alloyed into a single crystal surface to enhance defect structure for observation.

Defect Etch

A general term denoting the etch development of any bulk or surface anomaly in a material whether or not it is single crystal, colloidal, amorphous or crystalline in structure.

Definition Etch

Term used in two ways: (1) an etch used to develop a particular structure, such as fine-line definition, and (2) a solution developed for a particular purpose - a definitive etch.

Degreasing Etch

Usually a solvent for removal of oils or greases, rather than etch solution, although an etch may be used. Laboratory glass is etch cleaned in a solution of H_2SO_4:$K_2Cr_2O_7$, and soda-lime glass plates used for chrome photo resist masks are scrub cleaned with soap.

Deep Etch

Macroetching; etching preliminary to macro examination, intended to develop gross features such as segregation, grain flow, cracks and porosity.

Descaling Etch

An etch used to remove heavy contamination from metal surfaces. Term widely used in metal processing, but not in Solid State development where most materials are supplied as nominally clean parts.

Dicing Etch

Any etch solution used to cut and separate discrete devices or units from a semiconductor or other type material in wafer or thin sheet form.

Differential Interferance Contract

Differential Interferance Contract (DIC) - is a very useful illumination technique for providing enhanced specimen features. DIC uses a Normarski prism along with a polarizer in the 90 degrees crossed positions. The two light beams are made to conincide at the focal plane of the objective, thus rendering height differences visible as variations in color.

Dip Etch

The etching or cleaning of a specimen for a very short period of time - the "time" is difficult to define but can be arbitrarily said to be between 1-3 sec.

Dislocation Etch

The preferential etch development of structure in a single crystal material that can be related to crystallographically oriented defects associated with bulk structure or surface defects. Dislocations can be introduced during ingot growth; by heat treatment, alone, or in conjunction with alloying, diffusion, epitaxy, cutting, and lapping; by controlled bending or striking of a surface in defect studies; or inadvertent damaging from process handling, etc. Etching of exit points of dislocations on a surface. Depends on the strain field ranging over a distance of several atoms. Crystal figures (etch pits) are formed at exit points. For example, etch pits for cubic materials are cube faces.

Dissolutionment Etch

In the broadest context the term dissolution or dissolutionment etch refers to any solution that will dissolve a material. In the study of single crystals, it is used with specific reference to the etching of spheres to finite crystal form with development of crystal facets (planes) on convex surfaces.

Double Etching

Use of two etching solutions in sequence. The second etchant stresses a particular microstructural feature.

Drip Etch

To apply a single, or series of droplets on a surface. Usually applied to a limited area.

Drop Etch

Term has been used in three ways: (1) the free etching or cleaning of a part by physically dropping free into the solution; (2) placing of a drop of solution on a surface to etch and/ or plate a semiconductor p-n junction; develop pinholes in oxide/nitride thin films, etc., and (3) dropping of a part down through a column of solution with distance, time, and temperature used to control fabrication, e.g., the shot tower technique used in sphere forming.

Drop Etch

Placing of a drop of etchant on the polished surface. Suitable for precious etchants.

Dry Chemical Etch

The use of an ionized gas for cleaning or etching surfaces. Ar+ ion cleaning of single crystal wafer surfaces has become a standard technique in processing.

Dry Etch

Not considered a true form of etching, yet drying can affect the surface or bulk of a specimen. Water removal from a surface can cause crazing, cracking or leave stains. Bulk removal can alter both chemical formula and crystal structure. Heat treatment or annealing and hydrogen firing are two methods. The latter a method of surface cleaning in a reducing atmosphere.

Dry Ice Etch

Solid CO_2 used as a direct etching medium. Also used as a mixture with alcohols or acetone for chilling another etch solution, or for removal of water vapor from process gases.

Electrolytic Etch

The use of electric current applied to any etching or cleaning solution. he specimen being etched in the anode and a metal (such as Cu, Pb, Fe) as the cathode. Development of microstructure by selective dissolution of the polished surface under application of a direct current. Variation with layer formation: anodizing.

Eutectic-Cell Etching

Development of eutectic cells (grains).

Evaporation

The vaporization of a material by heating it, usually in a vacuum. In electron microscopy this process is used for shadowing or the produce thin support films by condensation of the vapors of metals or salts.

Extraction

A general term concerning chemical methods of isolating phases from the metal matrix: 1.) acid extraction-removal of phases by dissolution of the matrix metal in an acid. 2.) chlorine extraction-removal by formation of a volatile chloride, 3.) electrolytic extraction-removal by using an electrolytic cell containing an elctrolyte which preferentially dissolves the metal matrix.

Etch/Clean Sequences

See clean/etch sequences under composition.

Etch-Stop

An etch solution that will attack one material but not another in a multilayer thin film structure. Hot H_3PO_4 will etch remove Si_3N_4 much more rapidly than SiO_2, such that the oxide can work as an etch-stop mechanism. The method is used in structuring devices, and in the removal and thinning of layers for TEM study.

Failure Etch

Any solution use in the study of device failure, and used to etch develop, surface stain or otherwise expose the causes of failure.

Fatigue Etch

Specific solutions used on metals and their alloys in the study of material failure due to fatigue, such as cracking from bending or crazing from atmospheric corrosion. This is a major test evaluation method of study applied to metals and alloys with specimens prepared by metallographic techniques for observation after the test period.

Figure Etch

Any form of defect or structure developed in a surface by etching, regardless of the type etchant, e.g., gas, liquid, solid. Figures also can be formed by temperature, pressure, or direct flame. As a pressure formed figure, called a "percussion figure" on mica, (0001) surfaces developing as a six-rayed star. Star line pattern representative of bulk prism plane directions and used for orienting the micas.

Finish Etch

The final etch used on a material surface. Term primarily used in the metals industries, and can apply generally or to a specific etch or technique developed to produce a particular surface finish.

Finite Form Etch

Preferential etching of single crystal spheres of any metal or metallic compound to produce a solid with crystallographically oriented exterior planes that are the fast etching planes of a convex surface. Planes developed vary with solution mixture, as cube, (100); octahedron, (111); dodecahedron, (110); or tetrahexahedron, (hkO) as most common forms, e.g., also called finite crystal form etching, and primarily applies to materials that form in the isometric (cubic) system.

Flame Etch

Use of a propane torch or similar gas torch to produce etching action on a surface. Has been used to develop surface etch figures on high melting point temperature metals and alloys. Also used for surface cleaning of metals and their alloys during brazing operations.

Flash Etch

Any very rapid etch applied for a short period of time. It can be a liquid solution or an electrical spark. The latter used in spectrographic analysis.

Force

The total force with, which the specimen holder is pressed against the grinding/polishing disc in unit Newton (N).

Flat Etch

Either to etch a surface to be planar and flat, or etching of a material sheet in the form of a "flat".

Float-Off Etch

The removal of thin film layers from a surface for microscope study by TEM, SEM, etc. Etch solution attacks the substrate, but not the film.

Flood Etch

To rapidly cover a specimen surface with an etching or cleaning solution, usually with reference to washing or quenching acid reaction, rather than etching. An etching container is flooded with water to stop etch action.

Flush Etch

To cover a surface with a moving liquid etch solution. Used as a light surface cleaner, or to produce an etch-washed pattern for a decorative effect.

Flux Etch

Use of a molten metal or solid chemical compound for etching. May also refer to the use of a flux, such as borax, in metal alloying and brazing, and solutions used to clean and remove residual borax after joint fabrication.

Form Etch

Either etching a material to a specific shape, or etching of a particular shape. Spheres me etched to finite crystal form; rectangular bars are control-thinned as electronic reeds; silicon is via hole pattern etched as an inking mask; many metal shim stocks are electro formed or pattern etched as evaporation masks; tantalum is etch formed as an antenna, etc.

Forming Etch

Preferential, selective, or electrolytic etching of a specimen to a bulk shape, or structuring a surface by etching pits, via holes, channels. Diamond is etched with a saw-tooth structure as a filter element.

Free Etch

The etching of specimens by dropping them loose into a solution without being held in any manner.

Freeze Etch

The use of a solution below O°C to effect etching action, or, the quenching of a material from an elevated temperature into a liquid bath solution.

Fume Etch

Use of hot acid or solvent vapors for etching or cleaning action, hot HCl/H_2 vapors are used in epitaxy systems for general cleaning of quartz tubes, graphite susceptors and specimens.

Gas Etch

The use of a gas to produce etching action in its molecular form, such as argon, hydrogen, nitrogen, etc. It usually includes heat and/or pressure.

Gate Etch

Specialized term denoting channel etching for the active area of a Schottky Barrier device, such as a field effect transistor (FET). The channels are either wet chemically etched or electron photolithographically etched on the submicron width scale.

General Etch

Any etch solution can be so called when used without other specific definition, such as a general removal etch; and there is an etch solution with the name General Etch.

Graphic Etch

A repetitive pattern of figures etched onto a surface. Such patterns can be controllably produced for decorative effects or developed in nature - meteorites have a distinctive etched structure called Widmanstatten.

Gravity Etch

A highly specialized method of etching where the specimen is subjected to either a positive (+) or negative (-) gravitational force by centrifugal action during preferential etching to form controlled structures. Brass and gallium arsenide have been via hole and pit patterned in this manner. A specimen may be dropped down through an etch solution column for a controlled distance for forming or cleaning.

Grinding

The removal of material from the surface of a specimen by abrasion through the use of randomly oriented hard-abrasive particles bonded to a suitable substrate, such as paper or cloth, where the abrasive particle size is generally in the range of 60 to 600 grit (approximately 150 to 15 microns) but may be finer.

Grit/Grain Size

The grit or grain size (microns), of the abrasive used.

Grain-Boundary Etch

Development of intersections of grain faces with the polished surface. Because of severe, localized crystal deformation, grain boundaries have higher dissolution potential than grains themselves.

Accumulation of impurities in grain boundaries increases this effect.

Grain Contrast Etch

Development of grain surfaces lying in the polished surface of the microsection. These become visible through differences in reflectivity caused by reaction products on the surface or by grain differences in roughness.

Groove Etch

In single crystal wafer structuring of devices, the etching of a channel in surfaces. It may be as a "V" groove, have a bottom curvature of known radius; as a saw-tooth series of ridges; have vertical side walls with a flat bottom, and side walls may be specific crystal planes.

Halogen Etch

Refers to a chemical class of elements as etching agents: fluorine, chlorine, bromine, and iodine.

Heat Etch

The use of heat only, to effect an etching or cleaning action. Used under vacuum conditions to flash clean specimen surfaces. Used in air to oxidize a specimen surface.

Heat Tinting

Formation of colors (interference colors) in air or other gases, mostly at elevated temperature. Heating of a specimen on a hot plate in air to produce oxide colors. Used in the study of metallographic specimens. A crystalline material varies in color by rate of oxidation of individual crystallite grains according to their crystallographic plane orientation. A single crystal material or alloy can be differentiated by phase structure or internal crystallographic plane directions by similar color variation. In an alloy mixture or similar specimen, different metal or compounds can be determined by their characteristic oxide color when he'at tinted for a specific time. Single crystal spheres of metals and compounds have been widely studied by controlled furnace oxidation in the study of oxidation kinetics and growth rates related to internal crystal plane location and orientation.

Heavy Etch

Any etch solution used to remove a large volume of material. Measuremont is done by mils of surface removal, or by total specimen gram-weight loss.

Hot Etch

Any solution used for etching or cleaning above room temperature as either a liquid or gas, and may be shown in °F, °C, or K. Also shown as warm, hot, or boiling. Development and stabilization of the microstructure at elevated temperature in etchants or gases.

Ice Etch

Solid ice has been used as an etching medium in the sense of the freeze-out of a hot, liquid metal poured on the ice surface. Mixed with water or alcohol it is used to cool other etch solutions and, along with snow, has been used as a constituent in formulating a series of "cold etches" for specific temperature levels. Also can refer to the etching of single crystal ice specimens grown under cryogenic conditions in cold cryostats.

Identification Etching

Etching to expose particular microconstituents; all others remain unaffected.

Immersion Etch

Complete submersion of a specimen in a liquid etch solution, or in a molten flux solid rhemical solute. It is the most common form of etching as wet chemical etching (WCE) or electrolytic etching (EE), which are two of the Etchant Formats used in the next section. (Dry chemical etching (DCE) is the third format.) Method in which a microsection is dipped into etching solution face up and is moved around during etching. This is the most common etching method.

Immersion Etching, Cyclic

Alternate immersion into two etchants: (1) actual etchant; (2) solution used to dissolve layer formed during process 1.

Inclusion

Foreign material held mechanically, usually referring to non-metallic particles, such as oxides, sulfides, silicates, etc.

Interference

The effect of a combination of wave trains of varous phases and amplitudes.

Induced Damage Etch

Preferential etching to develop deferts or structure in a surface that has been subjected to some form of damage. Such damage may be a controlled scratch or a point of damage introduce by a diamond-type stylus to develop specific defects, or to initiate the etch forming of a pit, via hole, channel, etc. Also has been used with reference to residual subsurface damage remaining after cutting or mechanical lap and polish. Such damage being removed by chem/mech polishing or straigbt chemical etching in Solid State device fabrication.

Initial Etch

Any solution that is the first in a series, or the first etch applied irl a specific material process.

Ion Etch

A gas in its ionized rather than molecular state used to clean or etch a surface. RF plasma N2 or 02 cleaning systems are widely used in Solid State material processing. Argon, as ionic Ar, also is widely used in Solid State processing to final clean surfaces under vacuum immediately prior to metallization and compound growth or deposition, and can introduce subsurface damage. Other ionic gases, such as helium (He), xenon (Xe), etc., are used in material irradiation damage and thin film adhesion studies. Electron irra-diation from a TEM microscope or an electron-beam in vacuum system; lasers, for annealing or alternating materials; and nuclear particles have all been used as forms of ion etching.

Ion Etching

Surface removal by bombarding with accelerated ions (1-10 kV) in vacuum.

Irradiation Etch

The use of ionized gases or radiation particles to affect etching action. May also be used to induce damage into surfaces in the broader study of irradiation effects. Note that a single crystal, when irradiated, will revert toward the noncrystalline, amorphous state, and the reverse - an amorphous material will tend toward single crystal or the crystalline state.

Isotropic Etch

Another name for a polish etch solution. The etch attacks all crystal planes at an equal rate, producing a flat, planar surface. The opposite is anisotropic (preferential) etching. The terms anisotropic and isotropic originally applied to the propagation of light through a solid mineral. They also are now used in dry chemical etching (DCE) with regard to shaping a pit, channel or via hole with ionized gases without reference to the polish (isotropic) or preferential (anisotropic) nature of a liquid etch solution.

Jar Etch

The use of any closed vessel for etching in which parts or specimens are immersed.

Jar Etch

The use of any closed vessel for etching in which parts or specimens are immersed.

Jet Etch

A fine stream of liquid under pressure applied as an etchant, and most often for shaping the exterior of a solid specimen, structuring a material surface, etch cutting a hole through a wafer, etc. May be a single or multiple jr.t system with or without electric current. There are jet systems combining etch/cut action for slicing or dicing material with high velocity gas, steam, water, or an acid.

Junction Etch

As a semiconductor term, the development of a p-n junction by staining, etching or selective plating. In the heavy metals industry, weld joints can be developed or cleaned by etching or light sandblasting. In the plating and cladding industries, etching to develop interface joints between layers can be done with a jet.

Key Etch

A primary etch solutinn within a clean/etch sequence, or any solution applied in a processing step that is considered a fundamental solution.

Krypton Etch

The use of ionized Krypton, Kr+, as ail etch or material damaging agent by irradiation. The use of uapor pressure change to etch single crystal Krypton grown under pressure and cryogenic vacuum conditions.

Lapping

The abrasive removal of material using graded abrasive particles in a loose form as in liquid slurry on a platen.

Laser Etch

Use of electrons propagated by light at a controlled frequency and power level to effect an etching action. Several types of lasers are used to etch channels and other structures for operational Solid State devices; for material cutting, such as preparing circuit substrates; as annealing to increase crystallitc size on dendritic crystals, or in conversion of carbon to a diamond-like-compound (DLC).

Layer Etch

As applied in Solid State processing, the etch development of epitaxy multilayer structures in device fabrication or in material studies. As a more general term, the etch removal of a specific layer of material from a dissimilar material. It may be total removal as in oxide or nitride stripping from a semiconductor surface, or selective removal through a photo resist or similar surface coating to develop patterns for subsequent device fabrication.

Lift-Off Etch

A specific technique developed to remove thin film metallization from a photo-lithographically prepared wafer surface. Wafers are soaked, sprayed and/or lightly scrubbed with a plastic foam Q-tip in acetone. The acetone dissolves the photo resist layer used for patterning which loosens excess metal by lift-off, and exposes the metallized pattern for further device processing. This is a separate and distinct operation, though similar to the float-off technique.

Light Etch

The term has been used in two contexts: (1) as a physical term to differentiate between light-medium-heavy etching; or in the sense of a slow, minimum removal etch, and (2) the use of light alone, or in conjunction with solutions to enhance etching reaction or for selective plating action. White light most widely used, such as a strobe light for semiconductor p-n junction plating with copper.

Light Figure Etch

The use of preferential etchants to develop surface etch pit and dislocations on the surface of a cut single crystal ingot face or wafer and used to crystallographically orient the surface by reflected light. This is standard practice in the processing of smgle crystal ingots of all materials. It should be noted that many single crystal ingots can be cleaved into wafers on preferred fracture planes, which does not always require etching to obtained surface figures for light figure orientation (LFO), After etching face-cut silicon ingots in boiling KOH, 5 min, as an example, the ingot is mounted on a ceramic block, then on an x-y-z positioner and a pinhole light is reflected off of the surface back into a black box.

Light Figure Orientation Etch

The same as the upper item. As initially developed in Solid State semiconductor ingot processing, the etching of silicon in hot KOH solutions; germanium in KOH:I2, or acid solutions, such as CP4. The reflected surface defect pattern are used to orient the ingot for wafer slicing.

Lineation Etch

In semiconductor development the term was originally applied to distinctly aligned dislocation patterns associated with stress induced during the rotation/pull growth of Czochralski (CZ) single crystal ingots. The patterns are very distinctive on (111) and (100) oriented wafer surfaces: a series of over-lapping dislocations reducing in size from the wafer periphery toward wafer center, and disappearing before reaching the wafer center. On (111) surfaces, as three lines at 120° and associated with the bulk <211> directions; whereas on (100) surfaces, four such lines at 90° and associated with <110> bulk directions. With improved growth control, this form of defect rarely observed today.
Any crystallographically oriented line segment - representative of slip in a single crystal surface - may be termed lineation or slip. Where a series of such line segments are closely parallel - called stacking faults (SFs). This type of defect can be introduced by processing - oxidation, diffusion, epitaxy, ion implantation, and is often associated with oxide and nitride thin films. For these latter, $HF:CrO_3$ preferential etchants have been tailored for stacking fault study.

Liquid Etch

Any wet chemical etch (WCE) solution used to effect etch action. Also the use of a solid chemical molten flux etch, such as KOH pellets at 360 °C.

Long Etch

A time period for etching, as against a short or dip time period. Solution is either a slow etch on the material, or a cleaning solution with an extended soak time. This latter is common in metal processing, such as in soak etching or conditioning a surface prior to plating.

Long-Term Etching

Etching times of a few minutes to hours.

Loose Etch

Any solution used where parts are dropped free and not held in any way in the solution during the etch

period.

Low Etch

Applied with specific reference to the level of a solution above a part being etched, where insufficient covering acid will not block the in-diffusion of atmosphere over the open container. If the solution level is too shallow, it also can cause erratic etching results.

Lubricant

The liquid used for cooling and lubricating.

Macro Etch

Defects and structure observable on a surface with the unaided eye after etching. The term is widely used in the metallographic preparation of metals and alloys in the metals industries, and in geologic study of thin sections. It has occasionally been used with reference to preferential etching of semiconductor and other single crystal surfaces. Controlled etching of the surface of a metallic specimen, intended to reveal a structure which is visible at low magnification (not usually greater than 10 times).

Magnetic Etch

Not a real etching method, yet has been used in two ways: (1) magnetic powder has been brushed on wafer surfaces of ferromagnetic materials to develop domain structure, such as barium titanate and ferrites; (2) iron or carbon powder brushed on a preferentially etched wafer surface to accentuate defects and etched patterns. There are magnet-stir hot plates where a Teflon or plastic-coated magnet is dropped into the solution, then solution rotation and heat controlled by the hot plate dials. Barium titanate, being fabricated as an ultrasonic transducer, is electrically (magnetic flux) poled in water to orient domains.

Matte Etch

A surface finish etch used for decorative purposes on metal surfaces, such as copper or nickel. The surface has a low-profile grain-like structure with a dull sheen, and can be as a semi-matte finish.

Medium Etch

Used in two ways: (1) with regard to time, as a slow, medium, or fast etch, and (2) with regard to solution strength, as weak, medium, or strong.

Melt Away Etch

The separation of a thin film from a substrate for microscope study by heat liquefying and removal of the substrate. Etch removal of a metal thin film or alloyed pre-form, wire, etc., to observe the pit formed in the material surface by the metal. Semiconductor wafers with alloyed p-n junctions have been etched from the back to observe the buried junction-front in the bulk wafer. Sodium chloride, (100) substrates have been heated to the liquid state in order to remove a thin film for TEM study, rather than the usual use of water to dissolve the substrate/film interface.

Melt-Back Etch

A specialized Solid State term relative to wafer surface etching during epitaxy growth, such as using indium on indium phosphide, InP, or gallium on gallium arsenide, GaAs during liquid phase epitaxy (LPE). The method is used to clean the surfaces to reduce the growth of defects in the epitaxy film.

Mesaetch

The etching of a roughly cylindrical column or pylon on a single Crystal surface as a p-n junction device structure. In Solid State, as a single mesa, it is the mesa diode. As an array of mesas in the fabrication of SCRs, they are elements for power distribution and control in the electrical operation of the device. Mesas are commonly formed with a slightly preferential etch such that the mesa side slopes have a degree of crystallographic orientation. Using dry chemical etching techniques, the mesa sides can be more cylindrical without crystal facets.

Metal Etch

In the broadest sense, any solution used to etch any metal more commonly, the use of a liquified metal as an etch medium on high temperature, chemically inert metals, such as molybdenum, tantalum, or titanium. Occasionally refers to the metallizing of a material surface with in-diffusion to decorate defects.

Metallographic Etchig

Term primarily used in the preparation of metals and alloys as specimens for defect and structure analysis using preferential etches. Common in the metals industries and in geology, and most Solid State companies maintain a metallographic laboratory for material inspection and evaluation of processing. The etch can be macro- or micro-etch as a size definition.

Metallography

That branch of science which relates to the constitution and structure, and their relation to the properties, of metals and alloys.

Metallurgical Etch

Not common, but any etch applied to a metal or alloy specimen with reference to the field of metallurgy or metallurgical engineering as a science. Also with reference to the use of a metallurgical microscope for material observation.

Micro Etch

Any defect, figure, or structure etched on a surface that cannot be easily observed by the unaided eye, and requires a microscope for proper viewing. Both macro- and micro-etch are terms widely used in the study of metallographic specimens in general metal processing, not as often used in Solid State processing. Development of microstructure for microscopic examination. Usual magnification of more then 10 times.

Microstructure

The structure of a suitable prepared specimen as revealed by a microscope.

Milling Etch

In Solid State it refers to the use of an ion milling vacuum system where ionized argon (Ar^+) or nitrogen (N^+) is used to etch remove and pattern thin film metallization on circuit substrates or active devices. In general metal processing, it occasionally means the use of a lathe or cutting mill for a combination of cutting, etching, or shaping of a part, and may be as an electrolytic etch with the lathe head as the cathode.

Minimum Etch

An etch used for a short period of time, or one that removes little or no material during the etching period.

Mist Etch

The use of an etch as a spray of finely divided particles as from an atomizer. The method has been used for final etch cleaning of a surface; to fine-tune and optimize electrical characteristics of an exposed p-n junction semiconductor device; or to develop defects, structure, or figures in a surface for optimum clarity observation. Used in metallographic specimen etching to develop fine structure of crystallites, phases etc.

Molar Etch

Any solution mixed by its molecular weight.A Molar solution may be used as the etching solution by itself, or be only one constituent of an etch mixture.

Molten Flux Etch

Any metal or compound liquidized at or slightly above its melting point without the inclusion of water or other liquid solvent.

Multiple Etching

Treatment of microsection sequentially with specific reagents attacking distinct microconstituents.

Named Etch

Many etch solutions have a number, letter, chemical, individual's name, or a combination of such.

Native Oxide Etch

Almost all inorganic metals and compounds become surface passivated by an oxide when exposed to air. Such oxides are called native oxides as they are a normal attribute of the surface and not artificially produced. The removal of this type of oxide can be critical to metal processing, such as preparing aluminum surfaces for plating as well as copper, nickel, iron, and steels. It is of major importance in preparing semiconductor wafers for etching, metallization, diffusion, etc., as such residual oxides can affect device characteristics. Any etch solution used to remove such oxides is called a native oxide removal etch to differentiate it from a solution used to etch an oxide thin film or material, such as titanium dioxide, TiO2 or quartz, SiO_2, and called an "oxide etch". It should be realized that a "native" oxide only occurs under natural atmospheric conditions, even though it is called such in Solid State processing where residual surface oxide remains after chemical treatment.

Network Etching

Formation of networks, especially in mild steels, after etching in nitric acid. These networks relate to subgrain boundaries.

Neutral Etch

A very slow, nonreactive etch, when used as a cleaning solution, or the use of water as a pH 7 neutral wash or quenching solvent.

Nitride Etch

There are several artificially grown nitride compounds, such as silicon nitride, Si3N4 and aluminum nitride, A1N. The metal industries use a nitridization process to condition metal surfaces and the Solid State industry is developing and applying both oxide and nitride surface thin films in processes. In either case, any etch solution used to remove or pattern a nitride is called a nitride etch, and many also can be used on oxides.

Normal Etch

An etch solution mixed on the basis of the total valence of the metallic radical ions in solution. To obtain the number of grams of a compound for a Normal solution: divide one gram-molecular-weight (1 mole) by the total valence number of the element and radical.

Oil Etch

Petroleum base oils can act as etchants on some metals, even though they are normally only thought of as coolants in metal cutting, and similar processing. For critical materials, such as semiconductor wafers and assembly or test parts, such oil coolants with various additives for rust prevent, foaming, etc. can be severely degraded because of chemical attack, residual films, and other anomalies, such that silicones are used as replacement liquids for petroleum oils.

Optical Etching

Development of microstructure under application of special illumination techniques (dark field, phase contrast, interference contrast, polarized light).

Optical Interferometry

Is a technique that provides precise details of a materials surface topography. The simplest interferometer employs the interference between two beams of light. One beam is focused on the specimen and the second beam on an optically flat reference surface. The two reflected beams are then recombined by the beam splitter and pass through the eyepiece together. The two beams reinforce each other for those points on the specimen for which their path lengths are either the same of differ by an integral multiple of the wavelenth, n x lambda. The beams canel for path differences of n x lambda. Todays interferometers provide quantitiative 3-dimensional surface topography information.
The two most common optical interferometry techniques include - phase shifting interferometry (PSI) which uses a PZT to shift the optical path of the objective and vertical scanning interferometry (VSI) which changes the focus range of the objective. The main differences between PSI and VSI are that PSI has a higher z-axis resolution, whereas VSI has a larger scan range. PSI and VSI can also be combined to provide both high surface resolution and a larger scan range.

Orientation Etch

A preferential solution used to determine the single crystal orientation of a specimen surface by development of surface etch pits.

Oxide Cleaning Etch

A solution developed to clean an oxide surface with minimum or no removal. It can be acid, alkali, or solvent.

Oxide Etch

Any solution used to etch a metallic oxide material. See the immediately following terms for special case applications.

Oxide Removal Etch

In Solid State processing, often refers to removal of a native oxide on a material surface prior to further processing. It also is used where a deposited oxide thin film (SiO_2, Al_2O_3) is being pattern etched or removed.

Ozone Etch

Ozone; O_3, is an extremely strong oxidizing agent. In Solid State and some metal processing it is used, by itself, as a surface cleaner. Caution should be exercised as concentrations greater than 1 % in air can be hazardous to health. There are ozone producing commercial cleaning systems used in metal processing for material surface cleaning. Similar units are used in movie theaters or other commercial offices and buildings as an air freshener, and for cigarette smoke removal.

Particulate Etch

Any solution used to remove a material matrix and expose embedded particles without affecting the particles. This is common in some ore processing operations where gangue material is separated, and in some material studies. The etch may expose the particulate, only, or be used to remove the particles for separate microscope study.

Passivation Etch

The term is used in two ways: (1) a solution developed to remove a passivating thin film from a surface, such as a native oxide, or (2) a solution that will introduce a surface film passivation. An iodine solution has been used on diamonds in the latter case as pas-sivation against etching with H_3PO_4, and an I_2:Me0H rinse applied as an ionic surface contamination removal system on silicon wafers prior to diffusion.

Pattern Etch

Any etch solution that will develop structure in or on a surface and there are several different applications and methods: (1) to develop defects in surfaces; (2) to differentiate between elements, structure, or minerals in a mixture material; (3) to etch through a masking layer, such as photo resist patterned thin film oxides to remove the oxide down to the substrate in the desired pattern; (4) etching via holes, or (5) circuit pattern etching of substrates.

Phase Etch

Common to metal and metal alloy etching of steel in the etch development of alpha-, beta-, or delta-phase structure, and the recognition of martensite, carbide, and similar crystal structures.

Photo Resist Etch

Photo resist lacquers, such as the AZ- series, COP-, or PMMA types used in device and circuit fabrication of semiconductor devices, have their own solutions called developers, and used after UV exposure of the resists in fabricating patterns. The developers are designed for each type of commercial photo resist formulation, many contain hydroxide, such that caution should be observed if the material being processed in particularly vulnerable to attack by alkaline solutions.
Note that in removing photo resists before or after metallization of the specimens or wafers, the most widely used solvent is acetone (a ketone) by soaking, spraying, or light scrubbing of the specimen surfaces.

Physical Etching

Development of microstructure through removal of atoms from surface or lowering the grain-surface potential.

Pickling Etch

Term common in metal processing and plating. Metal surfaces are soak-cleaned for conditioning, such as for removal of scale, or other type contamination.

Pinhole Etch

Small, roughly circular defects in a deposited thin film are referred to as "pinholes" and may or may not go completely through the film. Any solution or method used to locate and observe such pinholes is called a pinhole etch. Such pinholes can be created by con-tamination on a substrate surface, be due to insufficient cohesion within the growing thin film, or from entrapment of particles in the film. Oxide and nitride thin films are particularly prone to pinholing in Solid State processing, and are the subject of much study. The term also is applied to the etching of a controlled pinhole, such as for thickness measurement of the film, diffusion depth profiling, or observation and study of epitaxy layer structures.

Pit Etch

A preferential etch used to develop dislocations or surface damage pits in single crystal wafers or similar specimens as wet chemical etching (WCE). This includes controlled damage pit development as a device structure. Dry chemical etching (DCE) through a photo resist, metal, or oxide/nitride thin film mask, as well as WCE, also for device fabrication. Note that dislocation pits conform to crystallographic structure and bulk plane directions, such as the sharp triangular pit on a (111) wafer surface, and do not increase in size to any extent with extended etching; whereas a surface damage pit does not conform to crystal planes and directions, expand in size with extended etching with a flat pit bottom that may be heavily terraced, and will disappear when the bulk, undamaged material surface is reached. Un-damaged bulk surfaces can be recognized by their usual high reflectivity, and common near1-mammillary and near-hexagon structure, particularly recognizable on (111) oriented surfaces. The (100) wafer bulk surface is more block-like in structure, coincident with the square out-line dislocation and surface etch pits.

Planar Etch

A polish etch that produces a very flat, highly reflective surface. Now a major surface finish for three-

dimensional layered electronic device and circuit substrate structuring.

Plane Etch

Preferential solution used to develop a crystal facet, or plane in a single crystal material. Occasionally with reference to etching a planar surface.

Plasma Etch

The use of ionized gas particles to effect cleaning or etching. Either RF or DC plasmas are used, RF more common in microwave, high frequency electronic device fabrication.

Polar Etch

Any etch used on a polar material, such as the compound semiconductors and, in particular, the (111) oriented surfaces. Gallium arsenide, as an example: the positive (111)Ga [(111)A] surface vs. the negative (111)As[(111)B] surface show different etching phenom-ena. One surface will etch preferentially with defects; whereas the opposed surface, usually the negative (111)B, will be erratic, and it is difficult to develop an etch solution that will equally polish both surfaces. Note that in compound semiconductors there also may be different electronic characteristics.

Polarity Etch

Any solution used to develop the preferential etching characteristics of polar compounds, such as GaAs, InP, AlSb, or their associated trinary and quaternary forms.

Pole Etch

A specialized term applied to the etching of magnetizable structure in materials such as barium titanate, Ba_2TiO_2, where the magnetic domains are aligned by polarization with electrolytic solutions and a magnetic flux. Garnet memory devices as a computer chip are similarly poled. The term also is applied in the etching of single crystal spheres for finite crystal form, when the solution produces only crystallographic "pole" figures at axial points, rather than developing exterior facets (planes) as a finite crystal solid form.

Polishing

A mechanical, chemical, or electrolytic process or combination therof used to prepare a smooth reflective surface suitable for microstructure examination, free of artifacts or damage introduced during prior sectioning or grinding.

Polish Etch

Any etch solution that attacks a material surface at an equal rate in all crystal plane directions without regard to their orientation. This is the opposite of preferential etching. In the Solid State single crystal etching, and much metal etching, a polished surface is required in the fabrication of devices and parts, such that polish etching is of great importance, and a major criterion in processes. It also is now called isotropic etching.

Potentiostatic Etching

Anodic development of microstructure at a constant potential. By adjusting the potential, a defined etching of singular phases is possible.

Precipitation Etching

Development of microstructure through formation of reaction products at the surface of the microsection (see also staining).

Preferential Etch

Any etch solution that will attack crystallographic planes at different rates, and produce structure as controlled by those planes. It is the opposite of polish etching. Much of the structuring and selective etching of semiconductors wafers and similar materials as devices is done with preferential etching, as well as in crystallographic study of single crystals. Such device processing includes formation of pits, channels, "V" grooves, via holes, saw-tooth structures. This was the only method of selective structuring semiconductor devices (wet chemical etching or electrolytic etching) until the fairly recent advent of dry chemical etching. Preferential etching also is now called anisotropic etching.

Pre-Plate Etch

Any acid, alkali, salt, or alcohol solution used to clean or condition a material surface prior to plating.

Pressure Etch

The forming of material, or alteration of a surface by direct pressure, alone, and may include heat or a gas atmosphere when a furnace or vacuum is used. Natural pressure produces etch figures on meteor surfaces during atmospheric entry; six-rayed star percussion figures are formed on (0001) oriented

mica sheets by striking; controlled point pressure damaging of single crystal wafer surface is used in forming etched pits or grooves in device structuring.

Printing

A method in which a carrier material is soaked with an etchant and pressed against the surface of the specimen. The etchant reacts with one of the phases. Substances form which react with the carrier material. These leave behind a life-size image. Used for exposing particular elements - for example, sulfur (sulfur prints).

Priming Etch

The term is applied with two meanings: (1) the type preparation of a surface prior to plating, such as a priming etch for conditioning, and (2) the adding of a small piece of the material to be etched to the etching solution prior to use. The latter method is used on highly reactive solutions, such as 1 HF:3 HNO_3, to obtain an even etch rate from the beginning.

Primary Etching

Development of cast structures including coring.

Profile Etch

The term is used in two ways: (1) Selective etching of any "form of structure in or on material, and (2) profile etching. The latter includes etching through thin film or metal masks; etching to observe and measure diffusion depths; to study epitaxy layer structures. Many wafers are processed with (100) surface orientation and, for profile study of structures, are cross-sectioned by cleaving in a <110> bulk plane direction.

Pylon Etch

Any etch used to form vertical, roughly cylindrical structures on a surface. A slightly preferential etch will produce facetted side-wall slopes with WCE; whereas smooth, unfa-cetted walls can be fabricated by DCE etching.

Quality Etch

Applied with regard to the purity of acids and chemicals used, such as electronic grade vs. commercial grade liquids or gases.

Quantity Etch

The etching or cleaning of a number of parts at a single time, or the use of a large volume of solution.

Rapid Etch

Any fast etching solution as against a slow etch. As an example, the 1 HF:3 HNO3 mixture is the most rapid etch solution of this two-component etching system.

Raster Etch

Electron beam (E-beam) etching using high intensity electrons; electron lithography, where a computer is used for beam positioning and exposure of photo resist patterns; and laser etching may be referred to as raster etching or annealing, as has been irradiation with ionic gases or radiation with particles in the sense of using a controlled beam of energized particles, electrons, etc., to effect an etching action. The term relates to the raster tracking element of a computer screen.

Rate Etch

Used in two ways: (1) the time period for any solution required to obtain desired results, or (2) the physical reactivity time/rate of specific mixtures. The latter most important when using exothermic solutions. Determining etch rates is a major factor in processing.

Reactive Ion Etch

A form of dry chemical etching (DCE) where one or more of the ionized gases is a reactive gas, such as BCl3 used in etching aluminum oxide surface films. Common acronym: RIE. This form of etching is increasingly used for selective etch structuring of electronic devices that contain layered epitaxy and metallized structure. Note that there are a number of specifically designed dry ionized gas etching systems with their own special acronyms, though all operate in a similar manner.

Reproducibility

Once a preparation method of sample has been developed and adjusted, it should produce exactly the same results for the same material, every time it is a carried out.

Redoxetch

Originally referred to as oxidation-reduction reaction, where one acid is a reducing agent, and the other an oxidizer. $HF:HNO_3$ solutions are an atypical example, where HF acts as the reducer and HNO_3 the oxidizer. As a REDOX etching system, the term is applied to selective etching with pH control of the solution.

Relief Etch

Etching of any form of structure on a surface. It can be as a raised mesa, pit, channel, or via-hole completely through the material.

Removal Etch

Any solution that will dissolve and reduce the thickness or weight of a material. All etches are removal type and many are classified by their etch-rate of removal.

RF Plasma Etch

Another term for dry chemical etching (DCE), and used as a general term for any form of ionized gas etching or cleaning.

Rolling Etch

Any solution used with a rolling motion. Single crystal spheres are etch polished in a beaker held at about 45° and hand swirled to produce a slow rolling action of the spheres.
Occasionally used with reference to a boiling solution that has a rolling or roiling motion. It also has been used with regard to allowing a specimen to roll and tumble down an incline during an etching period, or the etching of an extruded, rolled metal sheet to include etching in or against the rolled direction.

Rotational Speed (rpm)

The speed with which the grinding/polishing disc is rotating.

Rough Etch

Has been applied with two meanings: (1) general reduction in thickness or size without regard to surface finish and measured in mils of depth removed from a surface or by total gram-weight loss of a specimen and (2) a controlled etch to roughen a surface. Glass microscope slides have had one side roughed with HF vapor etching to improve thin film gold adhesion for TEM study of the film growth and structure characteristics, and some metal parts arc surface finished with a named roughness, such as a matte or satin finish.

Sand Etch

The term has been applied with reference to the use of a dry abrasive to effect removal action with the abrasive under gas pressure (nitrogen) applied by spray or jet. The method also is used to clean, roughen, or condition a surface. Abrasives are usually considered as lapping and polishing compounds, although fine jets of sand have been used to fabricate screws, to drill holes through material, or form cavities (pits) in a surface. The use of S.S. White Dental Unit was one of the original methods used to dice silicon and germanium wafers, and the units are still used for sand cleaning of surfaces, e.g., bead blasting technique for cleaning metal parts.

Satin Etch

Specialized term used in the metal industry where an etch solution produces a surface finish that has the appearance of satin cloth. The surface structure contains variable length and width lines, roughly parallel with some cross-hatching, and with the reflective sheen of satin cloth. The method is called satin finishing, and is used as a decorative finish on copper, nickel, brass and other metals.

Saturation Etch

A solution containing the maximum amount of a dissolved chemical in water, alcohol or solvent at room temperature and standard pressure. If such a solution is mixed above room temperature, and under pressure^ it is called a super-saturated solution.

Saw (Acid) Etch

When wafers are cut from an ingot by a wire that is wetted with acid, it is called the acid-saw technique (AST). The wire can be of iron, SST, rayon, plastic thread, etc.
In cutting, lapping, or polishing a surface mechanically there is always a subsurface damaged zone remaining with damage depth determined by abrasive grit size and other factors. Solid State wafers, such as silicon or gallium arsenide, are often chem/mech polished with bromine-methanol (BRM) solutions to remove this damaged zone. This has been referred to as saw damage removal etching.

Scale Etch

Term used in metal processing where slow etch solutions are used to remove surface contamination - oxidation, oils, dirt - that are called "scale".

Seeded Etch

Any solution to which is added a small piece of the material to be etched. This technique is used with solutions that initially show a rapid rate of attack in the first few seconds, then a more controllable linear rate. A mixture of 1 HF:3 HNO_3 is of this type. By initially seeding such a solution, allowing the piece to completely be dissolved, the etchant becomes more controllable for linear controlled removal.

Secondary Etching

Developmnet of microstructures deviating from primary structure through transformation and heat tretment in the solid state.

Segregation (Coring) Etching

Development of segregation (coring) mainly in macrostructures and microstructures of castings.

Segregate Etch

Any etch used to remove a matrix material to expose a paniculate or segregate embedded. This may be as a contaminant included during ingot growth or a new compound due to regrowth, such as is observed in fabricating silicides as blocking layers in Solid State devices.

Selective Etch

Either wet chemical etching (WCE) or dry chemical etching (DCE), where the solution or gas is used to structure a surface, or to remove specific material layers in a heterojunction/ heterostructure device. In the latter case, the etch will attack one material layer and not another, and by suitable masking of surfaces with a thin film oxide, photo resist, or metal, then exposing a pattern, pits, channels, via-holes, or other structure can be etched selectively.

Sequence Etch

A single solution, or a series of different solutions used in a consecutive order of etching steps. Clean/etch sequences are of this type, and can include vapor degreasing, etch removal of subsurface damage, acid etching, alkali etching, with water and/or alcohol rinses following etching, or the rinses as individual steps.
The term also is applied to the etching of different layers of heterostructure devices where different etch mixtures are used again consecutively.

Series Etch

Term has been used in two ways: (1) a single etchant used two or more times in sequence, such as in dip etching or (2) different acids, alkalies, and alcohols used in a sequence.

Shaping Etch

Any solution used to etch form a solid material. May be electrolytic etching, and term has been used with reference to electroforming.

Shim Etch

Either the etching of shim-stock material, such as thin nickel sheet pattern etching for use as an evaporation mask; or the etch thinning of a material to shim thickness, e.g., at or under about 0.010".

Short Etch

Any etch solution used for a brief period of time, as against a long period, such as a soak etch.

Short-Term Etching

Etching times of seconds to a few minutes.

Shrink Etching

Precipitation on grain surfaces. Shrinkage takes place during drying, which causes a cracking of the layer formed during etching. Crack orientation depends on the underlying structure.

Sizing Etch

In Solid State material processing the term applies to the etch reduction of physical size to a particular dimension" such as in thinning of a specimen for TEM microscope study. In general material processing, there is a gelatinous substance called "size", much like a weak glue which is used as a surface coating with applications similar to those in photolithographic processing with photo resist lacquers.
In paper manufacture, size is added to the pulp to prevent ink from running, and is the more accurate meaning, where tree rosins and alums are added as the sizing compounds.

Slosh Etch

Any etch solution used with either movement of the solution or the part.

Slow Etch

The removal rate of a solution as against a rapid or fast rate. Many polish etch solutions are designed to be slow for maximum planarity of surfaces, and for the prevention of erratic surface anomalies that can occur from a too rapid etch.

Slush Etch

Etches containing ice or snow as cold solutions.

Snow Etch

Some cold etch solution use snow as one constituent to establish a specific temperature. As a group, they are sometimes referred to as the "snow etches".

Soak Etch

A slow cleaning or etching solution where the part remains immersed for an extended period of time. Common to the metal and plating industries for surface preparation.

Solid Etch

The term has been used in two ways; (1) the etching of any solid material, or (2) molten flux etching with a liquified solid chemical compound, such as KOH pellets.

Solution Etch

A term used for wet chemical etching (WCE) where the etchant is a liquid; whereas dry chemical etching (DCE) uses ionized gases.

Solvent Etch

The use of a chemical solvent as a cleaning or etching solution as against an acid, alkali, or alcohol. A few metallic compounds can only be etched in a solvent.

Spark Etch

The use of an electric spark to generate an etching action, As a cutting method, called spark erosion; as a etching method an electrically activated wire loop in alcohol is used to observe pinholes in oxide and nitride thin films by the appearance of bubbles come from the substrate surface as the wire passes over in the alcohol solution. Metal spheres, both single crystal and polycrystalline, have been formed from chips of material by electrical sparking from a copper pot under argon, and spark vaporization of material from a surface is a standard method for spectrographic analysis.

Sphere Etch

The term has been used in two ways: (1) the slow polish etching of a material sphere, or (2) the preferential etching of a single crystal sphere to finite crystal form (FCF). Single crystal spheres have been widely used to establish etch and oxidation rates on convex surfaces in metal and metallic compound development for device structuring applications.

Spin Etch

Etching of a specimen with the solution and/or part rotating.

Sputter Etch

A term for RF or DC plasma etching.

Squirt Etch

Any solution used in the form of a liquid jet or spray. Term often applied when a polyethylene bottle know as a "squirt bottle" is used, and there are many designed jet etch systems.

Stacking Fault Etch

Specialized dislocation term used in single crystal processing. Specific preferential etches have been developed to accentuate this form of defect, which can be common to oxide and nitride thin films. The defect appears as a series of short, parallel slip lines in a translucent to transparent thin section, and are a three-dimensional defect relative to x, y, z crystallo-graphic axes. They occur due to inherent stress factors from the difference between coef-ficients of expansion of oxide/nitride thin films and the substrate materials on which they are deposited. The stacking faults can be in both the oxide/nitride or in the immediate substrate surface near the interface between the two compounds (SiCVSi interface, as an example).

Stagnant Etch

The term is applied to an etch mixture that has been allowed to stand before use. The H_2SO_4:H_2O_2 mixtures have been so used, with effective depletion of the hydrogen peroxide. CP4 also has been allowed to sit 24 h before use with the effective vaporization loss of the bromine fraction. Aqua regia (1 HC1:3 HNO_3) is an example of a solution that requires aging before use, but it is not a stagnant solution.

Stain Etch

Any liquid solution or gas, such as air or oxygen, producing a coloration action on a surface with minimum etch removal. HF:HNO_3 solutions, either high in HF on high in HNO_3, will stain a surface rather than etch. Other solutions, such as those containing salts of copper, silver or gold, also will produce stains. Both HF and HNO_3, develop red/blue/yellow color; whereas metal stains are grey to black. Staining has been used for depth profiling of diffused p-n junctions; in delineating exposed planar junctions; or to observe epitaxy layer structures in cleaved (110) cross-sections, which are stained after cleaving. Spheres have been oxidized in oxide growth rate studies relative to crystal planes, and metallographic samples are heat-tinted by oxidation in air on a hot plate.

Staining

Preciptation etching that causes contrast by distinctive staining of microconstituents; different interference colors orginate from surface layers of varying thickness. Prof of inhomogeneities.

Standard Etch

The term has been used in three ways: (1) a solution developed for a particular process becomes a Standard for that process; (2) a solution used as a reference in material or chemical analysis, and (3) a solution that has become standard for a particular use, such as CP4 as a polish etch, or Sirtl etch for defects.

Steam Etch

The use of water at its boiling point as a cleaning or etching solution. Steam cleaning under pressure for cleaning buildings, clothing, and small parts in autoclaves. In material processing the steam actively getters and uses a portion of the material surface in forming the oxide, such as a silicon wafer surface atoms being used to form silicon dioxide.

Step Etch

Any solution used to form an etched step in a material surface. The method is used in device structuring; to measure a layer thickness; to profile diffused junction depths; to trace defects in a material bulk; and may be single or multiple steps. A specimen surface is successively coasted with stripes of Apiezon-W (black wax) or photo resist coated in a similar manner, being etched to a controlled depth between each coating to form the steps.

Still Etch

A solution used without movement of either the solution or the part being etched. Some very slow polishing etches have been used in this manner.

Stir Etch

Any solution used with a rotational motion. Magnetic stirring hot plates; hand swirling;a hand of electrically operated stirring rod in solution, etc. In etching wafer surfaces for planarity, rotation speed is controlled so as to prevent flow patterns being developed.

Stop Etch

Also called: "Etch-Stop". Any etch solution that will attack one material and not another in a layer-type structure, A fairly recent term as applied to the selective etching of semi-conductor heterostructure devices, although the method has been in existence for many, many years in both the metals and semiconductor industries without having a specific name applied to the process, such as the etch removal or pattern etching of an oxide with HF that does not attack the underlying substrate. The solution may not be a complete "stop", as with an Si_3N_4 thin film on SiO_2 etched with H_3PO_4, where the oxide etches at a much slower rate, effectively working as an etch-stop.

Strain Etch

Similar to Stress Etch. Terms often used in combination as stress and strain studies. The bending of a thin material, such as a semiconductor wafer, will develop dislocations due to strain. Such defects have been studied as both a positive (+) or negative (-) strain direction relative to (100), (111)A or (TTT)B faces, and <110> directions.

Stress Etch

A preferential solution used to develop stress figures in a material. Stress may be from normal wear-and-tear; induced by heat treatment; physically induced by tension, compression or torque with or without heat. The latter method used in material studies.

Strong Etch

A general term applied to the use of concentrated acids or alkalies, as against a diluted solution, or a solution using weak acids.

Structure Etch

Any solution used to develop physical structure whether it is a defect in the material, a selectively etched pit, mesa, channel or the shaping of a solid.

Subtractive Etch

Term applies to the selective etching of multiple thin film layers. The removal of one layer without affecting others.

Swab Etch

Use of cotton or plastic foam type material on a stick. Medical Q-tips are used and, in Solid State processing, the cotton has been replaced with plastic foam to prevent reaction from the glue used to attach the cotton. Used with acetone for cleaning photo resist lacquers from surfaces. In processing metallographic specimens, swab etching is a common term and method of etching surface structure. Wiping of the specimen surface with a cotton ball saturated with etchant to simultaneously remove reaction products.

Swirl Etch

Any solution used with a rotating motion.

Temperature Etch

Temperature of cleaning or etching solutions vary with solution mixture, application requirements, and may be as a solid, liquid, or gas. May also refer to the use of temperature, alone, as a (heat/thermal) etch agent, under vacuum or furnace conditions. Much WCE etching is done at room temperature, but many solutions are exothermic, thus heating the solution during the etching period. To control such reactions, water or solvent cooling coils surround a water bath holding the etch solution vessel have been designed to limit such temperature rise. And temperature can be a major control factor for particular etch mix-tures... at room temperature (RT) a $1HF:8HNO_3$ solution is a good polish etch... but at 8°C becomes preferential... and at 50°C too rapid and erratic for control.

Thermal Etch

Term has been applied as (1) the use of heat in vacuum; (2) the use of heat in furnaces with a gas atmosphere. In the latter case, also called heat treatment (metals), and annealing (Solid State), though both terms have been used in all material processing areas. Pure heat alone can develop etch figures or structures on surfaces.

Thermal Etching

Annealing of the specimen in vacuum or inert atmosphere. Used primarily in high-temperature microscopy.

Thimble Etch

Any cup shaped vessel used to hold an etch solution. Specifically referenced to using small, ceramic, high temperature metal (platinum), or graphite crucibles to contain highly reactive, hot chemicals or liquid metals, such as KOH pellets at 360°C for dislocation etching of silicon wafers.

Thinning Etch

Any solution used to reduce thickness of a specimen. Widely used with reference to preparing specimens for transmission electron microscopy (TEM) studies.

Time

Preparation time, the time during which the specimen holder is rotating and pressed against the grinding/polishing disc.

Tinting

See heat tinting.

Trim Etch

Any etch used to reduce thickness, width, or length of a part, specimen, or device by small increments, only. This can include laser trimming, such as in fine-tuning paired diode devices for electrical matching

parameters.

True Microstructure

Theoretically we are interested in examining a specimen surface which shows us a precise image of the microstructure we are to analyse. Ideally, we require the following: no deformation, no scratches, no pull-outs, no introduction of foreign materials, no smearing, no relief or rounded edges and no thermal damage. This nearly perfect condition, with only superficial damage remaining, is commonly called the true microstructure.

Tumble Etch

A closed-end cylinder - called a "tumbler" - has long been used to polish rock and mineral specimens, or for surface cleaning of parts. Dry abrasive, a wet slurry or acid-slurry abrasive is added to specimens, then the tumbler rotated horizontally on a set of parallel bars. Rotation can be with a hand-operated crank but today tumblers are electrically operated. For parts clean/etch cycles, time is in minutes; whereas gem stone polishing is in days. Specimens and parts can be tumble etched in a beaker with the solution being stirred and parts floating free.

Ultrasonic Etch

Any solution used with an ultrasonic generator to develop agitation during an etching or cleaning period. There are small cup to very large basin-type systems. The latter include vapor degreasing systems. The cup types are used for cleaning and etching small parts with an etch beaker seated in a water bath that transmits the vibration frequency to the solution. Most systems are fixed frequency, but variable frequency units are available. A barium titanate, Ba_2Ti0_3, transducer is used to translate physical motion of the titanate into electrical frequency, much like piezoelectric quartz crystal radio frequency blanks.

Used Etch

Any solution applied for more than one period of time. In high volume processing where several parts are batch etched at one time, large volumes of etch solutions are used to sustain solution action without constituent depletion, and ensure good repetitive, temperature in-dependent etching without having to replenish with fresh solution after each etch period. In metal plating, the plating baths are automatically monitored, and additional solution added when plating rate reduces to a pre-set level.

Vacuum Etch

The use of pure vacuum to effect etching action by varying the vapor pressure. This method has been used to preferentially etch single crystal gases grown under cryogenic arid pressure conditions in vacuum.

Vapor Decreasing Etch

Vapor degreasing has wide use in cleaning parts with a combination of hot liquid, hot spray, and a hot vapor head in which parts are held. In the large systems, the hot liquid tank may include an ultrasonic transducer. Size ranges from a beaker on a hot plate to large tanks with an overhead crane hoist. Parts are lowered and held in the hot vapor head until they are deemed clean; they are then slowly removed and are found clean and dry. Although trichloroethylene (TCE), dichloroethylene (DCE, Perk), or trichloroethane (TCA) have been used in the past, Freon solvents are being used as replacements due to the carcinogenic nature of chlorinated solvents.

Vapor Etch

Any solution used in vapor form to effect an etching or cleaning action. HNO3, H2O, and H2O2 arc all used to oxidize a surface, then the oxide stripped with HF as one method of surface cleaning. In epitaxy system, hot HCl vapor is used to clean the quartz tube walls, the susceptor carrier and, in some cases, the material surface about to be deposited upon.

Via Hole Etch

Specialized term in semiconductor processing where a crystallographically orient hole is Selectively etched through a wafer. The side walls of the hole are then metallized through from top to bottom surface, often referred to as wrap-around plating for an electrical ground plane. See Selective Etch.

Vibration Etch

Any form of vibration used in conjunction with an etching or cleaning solution. The two most common systems are an ultrasonic transducer or a shaker table. Vibration alone is a standard assembly test vehicle, and can cause part or assembly failure. Such vibration tests include acceleration/deceleration, and g-force generators with an x, y, z spin component.

Wash Etch

Used with reference to the pouring of a solution across a surface. Not widely used for etching, as it tends to cause surface channelling. Standard water quenching of parts after etching is sometimes referred to as "Wash" clean, but is not an actual etching method.

Water Etch

Though water is considered neutral, with pH 7, the major liquid used for quenching an etch solution, and for general washing and rinsing, it can act as an acid etch on water soluble compounds. Such compounds can be polished, preferential etched, or selective etched with water only. Sodium Chloride, NaCl, is one such compound that is widely used as a (100) oriented substrate for thin film metal evaporation and epitaxy growth in morphological studies with thin films removed by the float-off technique for TEM observation. High purity water, as well as brine and atmospheric moisture, will slowly corrode many metals and alloys. The metal industry uses coupons of new materials in corrosion tests by exposure to salt atmosphere along scacoasts, and study irons and steels by dripping high purity water onto surfaces over extended periods of time. In the study of ice, it is grown as single crystals and water is used to polish or preferential etch surfaces.

Weak Etch

The use of a highly diluted etch mixture or a singular liquid; such as ammonium hydroxide, NH_4OH and ammonia, NH_3, which are both weak bases.

Wet Chemical Etching (WCE)

One of the three major divisions in chemical etching. It is the use of any liquid to effect cleaning or etching action and still the most widely used method of etching.

Wet Etching

Developmnet of microstructure with liquids (acids, bases, neutral solutions, mixtures of solutions).

Wipe Etching

See swabbing.

White Etch

The term has been applied to mixtures of HF:HNO3 with or without HAc/H_2O. It is not recommended, as these solutions are clear and transparent, not an opaque white.

Work Damage Etch

Any polish etch used to remove residual sub-surface damage remaining after cutting and mechanical lap and polish in preparing a wafer or similar specimen. The bromine:methanol (Br_2:Me0H) or BRM solutions are currently used on many metals and metallic compounds for this purpose, as they not only remove such damage, but act as slow polishing solutions.

X-Ray Etch

Though X-rays are a photographic technique used in both medicine and material studies, X-rays, as well as other particles, have been used in material surface studies. Fluorite, CaF_2, will show color changes when subjected to X-rays, such that here it is referred to as a method of etching. Radiation or irradiation using ionized gases or atomic particles can produce similar effects in material, as well as cause internal, bulk damage. Again, not a true etching phenomenon, but of major importance in material processing. All space hardware is subjected to radiation evaluation as one high reliability test, and X-ray may be included.

Yellow Room Etch

Any solution used in a Yellow Room for the processing of material by photolithographic techniques. Photo resist lacquers are affected by exposure to white light, such that all processing is done under yellow light with humidity controlled at 40 % ± 5 RH, and temperature between 70-72 °F for- optimum results. Too much humidity and photo resist will not harden properly; whereas too low a humidity, the lacquer will harden too rapidly and crack. If the temperature approaches 80°F, the resist also will not cure or harden properly even under controlled oven bake out conditions.

Common Chemicals

Name	Formula	Note
1-butanol	$CH_3(CH_2)3OH$	Flammable, liquid
1, 2-ethanediol (dihydroxy, ethane, ethylene glycol, glycol)	$C_2H_6O_2$ ($HOCH_2CH_2OH$)	Liquid
1, 3-dymethyl 2-thiourea	$C_3H_8N_2S$ ($CH_3NHCSNHCH_3$)	Crystalline
2-butoxyethanol	C_4H_9 x O x CH_2 x CH_2)H	Liquid
n-butyl alcohol	Normal butyl alcohol	Liquid
Acetic acid (HAc, HOAc)	CH_3COOH	Toxic (custic)
Acetone	CH_3COCH_3	-
Acetic anhydride	$(CH_3CO)_2O$	Toxic
Acetone sodium bisulfite	CH_3COCH_3 x $NaHSO_3$	Toxic
Acetylacetone	$C_5H_8O_2$	Flammable, liquid
Acetylene	C_2H_2	-
Aluminium chloride	$AlCl_3$	Crystalline
Alum (Potassium)	$K2Al_3(SO_4)_4$ x $24H_2O$	-
Ammonia	NH_3	Toxic, gas

Ammonium bifluoride	NH_4F x HF	Toxic
Ammyl alcohol	$CH_3(CH_2)_3$ x CH_2OH	Flammable, liquid
Ammonium fluoride	NH_4F	-
Ammonia water	$NH_3 + H_2O$	Toxic, liquid
Ammonium molybdate	$(NH_4)6Mo_7O_2$x 4 x $4H_2O$	Crystals
Ammonium acetate	CH_3COONH_4	Crystalline
Ammonium chloride	NH_4Cl	Crystalline
Ammonium dicitrate	$C_6H_{14}N_2O_7$	Crystalline
Ammonium ditartrate	$(NH_4)_2C_2H_4O_6$	Crystalline
Ammonia hydoxide	NH_4OH	Toxic, liquid
Ammonium hydrogen fluoride	$(NH_4)HF_2$	Crystalline
Ammonium paramolybdate	$(NH_4)6Mo_7O_24$ x $4H_2O$	Crystalline (molybdic acid)
Ammonium peroxydisulfate	$(NH_4)2S_2O_8$	Crystalline
Ammonium polysulfide	$(NH_4)2Sx$	Toxic, liquid

Ammonium thiosulfate	$(NH_4)2S_2O_3$	Crystalline
Ammonium nitrate	NH_4NO_3	-
Ammonium sulfate	$(NH_4)2SO_4$	-
Antimony trichloride	$SbCl_3$	-
Antimony trisulfide	Sb_2S_3	-
Arsenic trioxide	As_2O_3	-
Argon	Ar	Gas
Arsine	AsH_4	-
Barium hydroxide	$Ba(OH)_2$	-

Barium oxide	BaO	-
Beetsugar	$C_6H_{12}O_6$	Glucose
Benzene	C_6H_6	-
Bismuth chloride	$BiCl_4$	-
Bismuth trioxide	Bi_3O_3	-
Bleaching powder	$CaCl(OCl)$	Bleach
Borax	$Na_2B_4O_7 \times 10\ H_2O$	-
Boric acid	$B(OH)_2$	-
Bromine	Br_2	Toxic, liquid
Benzalkonium chloride	Essentially alkyl-dimethyl-benzyl-ammonium chloride.	Crystals
Butyl carbitol	Diethylene glycol mono-butyl ether	Liquid
Butyl cellosolve	2-butoxyethanol	Liquid
Calcium carbonate	$CaCO_3$	-
Calcium fluoride	CaF_2	-
Calcium hydroxide	$Ca(OH)_2$	-
Calcium oxide	CaO	Lime

Cane sugar	$C_{12}H_{22}O_{11}$	Sucrose
Cadmium chloride	$CdCl_2$ x H_2O	Toxic, Crystalline
Carbolic acid	C_6H_5OH	-
Carbon disulfide	CS_2	-
Carbon tetrachloride	CCl_4	-
Chloroform	CHC_{12}	-
Carbitol	Diethylene glycol monoethyl ether	Liquid
Cadmium acetate	$Cd(C_2H_3O_2)_2$	Toxic, Crystalline
Cerium (IV) nitrate	$Ce(NO_3)_4$	Crystalline
Cellosolve	ethylene glycol monoethyl ether	Liquid
Chromium anhydride	See chromic acid	-
Chromium (III) oxide	Cr_2O_3	Crystalline
Chromium (VI) oxide (Chromic acid)	CrO_3	Toxic, crystalline
Citric acid	$C_6H_8O_7$ x H_2O	Crystalline

Copper (II) ammonium chloride	$(NH_4)_2[CuCl_4]$ x $2H_2O$	Toxic, crystalline
Copper ammonium persulfate	$[Cu(NH_3)4]S_2O_8$	Crystalline
Copper (II) chloride	$CuCl_2$ x H_2O	Toxic, crystalline
Copper (II) nitrate	$Cu(NO_3)_2$ x $6H_2O$	Toxic, crystalline
Copper nitrate	$Cu(No_3)_2$	-
Copper (II) sulfate	$CuSO_4$ x $5H_5O$	Toxic, crystalline
Copper sulfate	$CuSO_4$	-
Cupric chloride	$CuCl_2$	Crystalline
Copper oxide	CuO	-
Diborane	B_2H_6	-
Diethylene glycol	$(HOCH_2CH_2)_2O$	Liquid
Diethylene glycol monobutyl ether	See butyl carbitol	-
Diethylene glycol monoethyl ether	See ether	-
Diethyl ether	See ether	-

Ethane	C_2H_6	-
Ethanethiol	$C_6H_{14}O_2$	Flammable, liquid
Ethanol, ethyl alcohol (EOH)	C_2H_5OH	Flammable, liquid
Ethylene glycol	$C_2H_6O_2$ ($HOCH_2CH_2OH$)	fFlammable, liquid
Ethly ether (ether)	$C_2H_5OC_2H_6$	Flammable, liquid
Ethylene glycol monobutyl ether	2-butoxyethanol	Liquid
Ethylene glycol (EG)	CH_2OHCH_2OH	-
Ethylene	C_2H_4	-
Ethylene glycol monoethyl ether	See cellosolve	-
Ethylenediamine (ED)	$NH_2(CH_2)NH_2$	-
Ferrous acetate	$Fe(C_2H_3O_2)2 \times 4H_2O$	Liquid
Ferric chloride	$FeCl_3$	Crystalline
Ferric oxide	Fe_2O_3	Red ocher (rouge)
Ferric oxide	Fe(OH)	Yellow/tan roughe
Ferric oxide	Fe_3O_4	Black rouge

Ferric nitrate	$Fe(NO_3)_3 \times 9H_2O$	Crystals
Ferric sulphide	FeS	-
Fluoboric acid	HBF_4	Toxic, liquid
Formic acid	HCOOH	Fructose, liquid
Fruit sugar	$C_6H_{12}O_6$	-
Glycerol (glycerine)	$C_3H_8O_3$ ($HOCH_2CHOHCH_2OH$)	Liquid
Glucose	$C_6H_{12}O_6$	-
Gold (III) chloride	$AuCl_3 \times H_2O$	Crystalline
Hydrochloric acid	HCl (95 %)	Muriatic acid, Toxic, liquid
Hydrofluoric acid (HF)	HF (49 %)	Toxic, liquid
Hydrogen	H_2	Explosive, gas, flammable
Hydrogen peroxide	H_2O_2 (30 %)	Peroxide, liquid
Hydrogen sulfide	H_2S	Toxic, gas

Hypochlorous acid	$HClO_4$	-
Iodic acid	HIO_3	-
Iodoform	CHI_3	-
Iron (III) chloride	$FeCl_3$ x $6H_2O$	Crystalline
Iron (III) nitrate	$Fe(NO_3)_3$ x $9H_2O$	Crystalline
Iron (II) sulfate	$FeSO_4$ x $7H_2O$	Crystalline
Lactic acid	$C_3H_6O_3$	Liquid
Lactose	$C1_2H_2O_{11}$	-
Lead acetate	$Pb(CH_3COO)_2$	Toxic, crystalline
Lead sulphite	$PbSO_4$	Crystalline
Lead chloride	$PbCl_3$	-
Lead chromate	$PbCrO_4$	-
Lead nitrate	$Pb(NO_3)_2$	-
Lead oxide	PbO	-
Lime	CaO	-

Litharge	PbO	-
Lithium chloride	$LiCl$	-
Magnesium oxide (Magnesia)	MgO	Crystalline
Magnesium chloride	$MgCl_2$	Toxic, crystalline
Magnesium hydroxide	$Mg(OH)_2$	Milk of magnesia
Maltose	$C1_2H_{22}O_{11}$	-
Manganese dioxide	MnO_2	-
Mercury (II) nitrate	$Hg(NO_3)_2 \times 8H_2O$	Toxic, crystalline
Mercury chloride	$HgCl_2$	Toxic, crystalline
Mercury nitrate	$Hg_2(NO_3)_2$	-
Methane	CH_4	-
Methyl alcohol, methanol (MeOH)	CH_3OH	Toxic, liquid
Methyl ether	CH_3OCH_3	-
Methyl ethyl ketone (MEK)	-	-
Meta-Nitrobenzene sulphonic	$NO_2 \times C_6H_4 \times SO_3H$	Toxic, liquid
Molybdic acid	See ammonium molybdate	Crystals
Muriatic acid	Technical grade HCl	Liquid

Nitric acid	HNO_3	Toxic, liquid
Nitric acid	HNO_3 (70 %)	White fuming
Nitric acid	HNO_3 (72 %)	Yellow fuming
Nitric acid	HNO_3 (74 %)	Red fuming
Nitrogen	N_2	Gas
Oxalic acid	$C_2H_2O_4 \times 2H_2O$	Toxic, crystalline
Perchloric acid	$HClO_4$	Toxic, liquid
Perchloroethylene (PCE)	C_2Cl_4	Perk
Phosphoric acid	H_3PO_4	Toxic, liquid
Phosphine	PH_3	-
Phosphorous pentoxide	P_2O_5	-
Plaster of Paris	$CaSO_4$ (+H_2O)	-
Picric acid	$C_6H_3N_3O_7$	Toxic, explosive, crystalline

Potassium bicarbonate	$KHCO_3$	Crystalline
Potassium bromide	KBr	-
Potassium carbonate	K_2CO_3	Crystalline
Potassium chloride	KCl	Crystalline
Potassium chlorate	$KClO_3$	-
Potassium cyanide	KCN	Toxic, crystalline
Potassium dichromate	$K_2Cr_2O_7$	Toxic, crystalline
Potassium ferricyanide	$K_3[Fe(CN)_6]$	Crystalline
Potassium ferrocyanide	$K_4[Fe(CN)_6]$	Crystalline
Potassium hydrate solution	$KOH + H_2O$	Toxic, liquid
Potassium hydrogen fluoride	KHF_2	Crystalline
Potassium hydrogen sulfate	$KHSO_4$	Crystalline
Potassium hydroxide	KOH	Toxic, crystalline
Potassium iodide	KI	Crystalline

Potassium metabisulfite	$K_2S_2O_5$	Crystalline
Potassium nitrate	KNO_3	Crystalline
Potassium nitrite	KNO_2	-
Potassium phthalate (di-)	$C_8H_4K_2O_4$	Crystalline
Potassium permanganate	$KMnO_4$	Crystalline
Potassium sulfate	K_2SO_4	-
Potassium thiocyanate	KSCN	Toxic, crystalline
Propane	C_3H_6	-
Propionic acid	CH_3CH_2COOH	-
(Iso)propyl alcohol (ISO, ISO4)	C_3H_7OH	-
Pyrocatechol (P)	$C_6H_4(OH)_2$	-
Pyrophosphoric acid	H_4PO_4; anhydrous	Crystals or viscous liquid
Quinone	C6H4O2	-
Selenic acid	H_2SeO_4	Liquid

Silver bromide	$AgBr$	-
Silver chloride	$AgCl$	-
Silver cyanide	$AgCN$	Toxic, crystalline
Silver iodide	AgI	-
Silver nitrate	$AgNO_3$	Crystalline
Soap	$3C_{17}H_{35}O$	-
Sodium bisulphite	$Na_2S_3O_5$	Crystalline
Sodium bicarbonate	$NaCHO_3$	Crystalline
Sodium carbonate	$Na_2CO_3 \times 10H_2O$	Crystalline
Sodium chloride	$NaCl$	Crystalline
Sodium chromate	Na_2CrO_4	Crystalline
Sodium cyanide	$NaCN$	Toxic, crystalline
Sodium dichromate	$Na_2Cr_2O_7 \times 2H_2O$	Crystalline
Sodium fluoride	NaF	Crystalline
Sodium hydrogen phosphate	$Na_2HPO_4 \times 12H_2O$	Crystalline

Sodium hydroxide	$NaOH$	Toxic, crystalline
Sodium iodide	NaI	-
Sodium molybdate	$Na_2MoO_4 \times 2H_2O$	Liquid
Sodium nitrate	$NaNO_3$	Crystalline
Sodium sulfate	Na_2SO_4	Crystalline
Sodium sulfate, anhydrous	Na_2SO_4	Crystalline
Sodium sulfide	Na_2S	Crystalline
Sodium tetraborate	$Na_2B_4O_7$	Crystalline
Sodium thiocyanate	$NaSCN$	Crystalline
Sodium thiosulfate (fixer)	$Na_2S_2O_3 \times 5H_2O$	Crystalline
Spirits of ammonia	$NH_3 + H_2O$	Toxic, liquid
Stannic chloride	$SnCl_5$	-
Stannous chloride	$SnCl_2$	Crystalline
Stearic acid	$C_{17}H_3 \times 5COOH$	-
Strontium chloride	$SrCl_3$	-
Strontium nitrate	$Sr(NO_3)_2$	-
Sucrose	$C_{12}H_{22}O_{11}$	-

Sulfuric acid	H_2SO_4 (95 %)	Toxic, liquid
Sulfamic acid	NH_2SO_3H	Liquid
Sulfur dioxide	SO_2	-
Sulfur trioxide	SO_3	-
Tartaric acid	$C_4H_6O_6$	Liquid
Thioglycolic acid	$HSCH_2COOH$	Liquid
Thiourea	$CS(NH_2)_2$	Crystalline
Tin (II) chloride	$SnCl_2 \times 2H_2O$	Toxic, crystalline
Toluene	$C_6HC_5H_3$	-
Turpentine	$C_{10}H_{16}$	-
Vogel's special reagent	Mixture of tar and (stainless steel etchant) sulfurous acid, boiled and filtered; protected	Liquid

Wetting agents	Additives for lowering surface tension	Surface tension
Water	H_2O	Liquid
Xylene	$C_6H_4(CH_3)_2$	Flammable, liquid
Zinc chloride	$ZnCl_2$	Toxic, crystalline
Zinc oxide	ZnO	-
Zinc sulfate	$ZnSO_4$	-
Zinc dichloride	$ZrCl_2$	-
Zirconium iodide	ZrI_4	-
Zephiran chloride	A proprietary material produced in grades containing about 12 % and 17 % benzalkonium chloride as active constituent, plus some ammonium acetate; also called sephiran chloride. Available from pharmacies.	Aqueous solution

Common Hardness of Materials

Material	Knoop Hardness
Talc	20
Silver	20
Cooper	163
Aluminium	80-250
Annealed Steel	200
Martensite	500-800
Glass	530
Carburized Steel	730
Chromium Plating	740
Quarz (Silicon Dioxide)	820
White Cast Iron	840
Nitrided Steel	970
Cementite (Iron Carbide)	1025
Zirconia	1160
Zirconium Boride	1550
Chromium Carbide	1735
Molybdenum Carbide	1800

Titanium Carbide	1800
Tungsten Carbide	1880
Tantalum Carbide	2000
Alumina	2100
Zirconium Carbide	2100
Beryllium Carbide	2410
Titanium Carbide	2470
Silicon Carbide	2480
Aluminium Boride	2500
Vanadium Carbide	2660
Boron Carbide	2750
Titanium Diboride	4400
Diamond	7000

Grit Size Table

Grit Number	Size (μm)	Grit Number	Size (μm)	Emery Grit
P60	269.0	60	268.0	-
P80	201.0	80	188.0	-
P100	162.0	100	148.0	-
P120	127.0	120	116.0	-
P180	78.0	180	78.0	3
P240	58.5	220	66.0	2
P280	52.2	240	51.8	-
P320	46.2	-	-	-
P360	40.5	280	42.3	1
P400	35.0	320	34.3	0
P500	30.2	360	27.3	-
P600	25.8	400	22.1	00
P800	21.8	-	-	-
P1000	18.3	500	18.2	000
P1200	15.3	600	14.5	-
P1500	12.6	800	12.2	0000
P2000	10.3	1000	9.2	-
P2500	8.4	1200	6.5	-
P4000*	5.0*	-	-	-

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

* FEPA grits finer then P2500 are not standardized and are graded at the discretion of the manufacturer.

Mohs Hardness Scale

Material	Value
Talc-$Mg_3Si_4(OH)_2$	1
Gypsum-$CaSO_4$-2(H_2O)	2
Calcite-$CaCO_3$	3
Fluorite-CaF_2	4
Apatite-$Ca_5(PO_4)_3(OH,F,Cl)$	5
Orthoclase-$KAlSi_3O_8$	6
Quartz-SiO_2	7
Topaz-$Al_2SiO_4(F,OH)_3$	8
Corundum-Al_2O_3	9
Diamond-C	10

Table 1 General comparison of compression mounting resins

Factor	Phenolics	Acrylics	Epoxy	Diallyl phthalates
Cost	Low	Moderate	Moderate	Moderate
Ease of use	Excellent	Moderate	Excellent	Excellent
Availability of colors	Yes	Clear	Black only	Blue only
Cycle times	Excellent	Poor	Excellent	Excellent
Edge retention[a]	Fair	Poor	Excellent	Moderate
Clarity	Opaque	Excellent	Opaque	Opaque
Hardness[a]	Low	Good	High	High
Form	Granular	Powder	Granular or powder	Granular
Specific gravity, g/cm^2	1.4	0.95	1.75–2.05[b]	1.7–1.9
Colors	Black, red, green	Clear	Black	Blue
Shrinkage (compression), in./in.	0.006	...	0.001–0.003[b]	0.001–0.003
Coefficient of linear thermal expansion, (in./in.)°C×10^{-6}	50	...	28[b]	19
Heat (boiling) etchant	Seriously degraded	Soften	Holds up in heated etchant	Degrades but not as badly as phenolics
Molding temperature, °C (°F)	150–165 (300–330)	...	143–177 (290–350)	160–177 (320–350)
Molding pressure, MPa (psi)	21–28 (3050–4000)	...	17–28 (2500–4000)	24–41 (3500–6000)
Curing time (at temperature and pressure) for 12 mm (0.5 in.) mount	90–120 s	2–4 min	90–120 s	90–120 s

(a) No polymer is hard compared to metal, except for lead. Abrasion/polishing rate relative to metals is the important comparison. Epoxies are very good, because fillers make their grinding/polishing rates similar to metals.

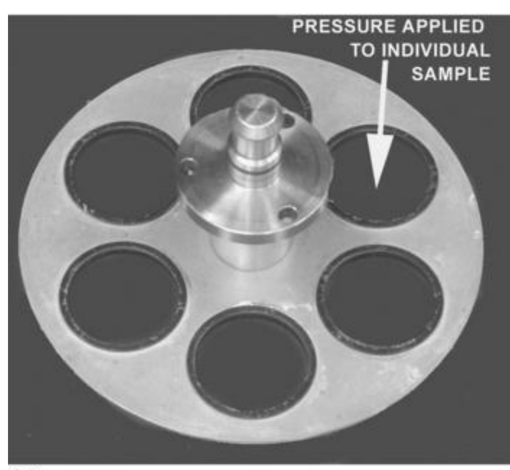
(b) Glass-filled epoxy

General comparison of castable (cold-mounting) resins

Factor	Epoxy	Acrylic	Polyester resin
Type	Epoxy resin and hardener	Acrylic resin and powder	Polyester resin and hardener
Peak temperature, °C (°F)	28 (82)	27 (80)	38 (100)
Shore D hardness	82	80	76
Cure time	6–8 h. Some cure in 45 min.	5–8 min	6–8 h
Comments	Moderate hardness, low shrinkage, transparent	Very fast cure, translucent, some shrinkage, inexpensive, and widely used on printed circuit boards; high exotherm during polymerization	Transparent, clear, rarely used

Selection of a mounting plastic for a specific application requires detailed consideration of key performance factors that sometimes can be quite demanding. Some key factors are (Ref 3):

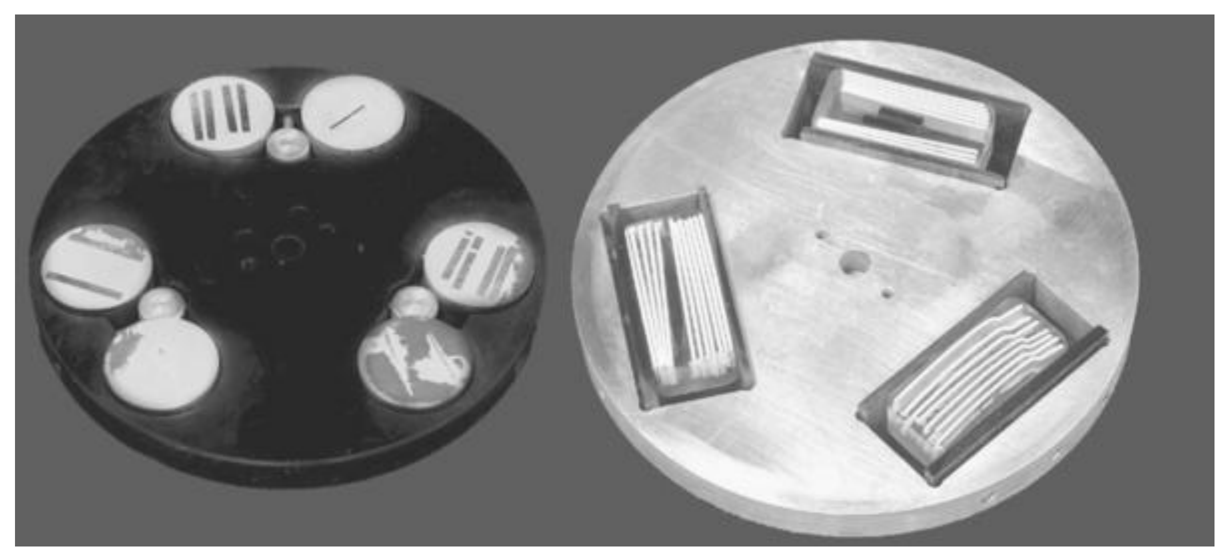
Sample holders for semiautomatic polishing machines. (a) Nonfixed holder. (b) Fixed (rigid) holder



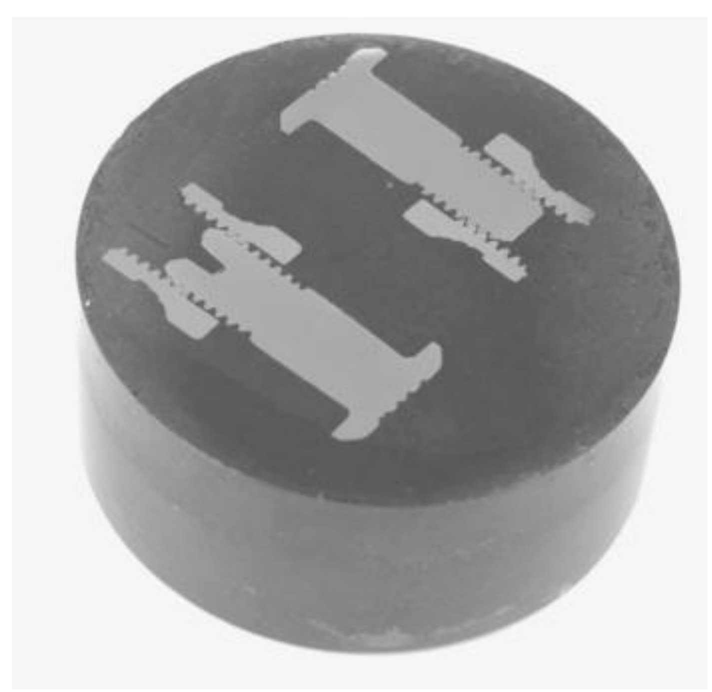
(a)

(b)

Arrangement of specimen mounts in a rigid (fixed) sample holder



Dual-specimen mount for a nonfixed sample holder



Typical problems of compression mounting materials

Problem	Cause	Solution
Thermosetting resins		
Radial split	Too large a section in the given mold area; sharp-cornered specimens	Increase mold size; reduce specimen size.
Edge shrinkage	Excessive shrinkage of plastic away from sample	Decrease molding temperature; cool mold slightly prior to ejection.
Circumferential splits	Absorbed moisture; entrapped gasses during molding	Preheat powder or premold; momentarily release pressure during fluid state.
Burst	Too short a cure period; insufficient pressure	Lengthen cure period; apply sufficient pressure during transition from fluid state to solid state.
Unfused (woody)	Insufficient molding pressure; insufficient time at cure temperature; increased surface area of powdered materials	Use proper molding pressure; increase cure time. With powders, quickly seal mold closure and apply pressure to eliminate localized curing.
	Excessive mold temperature	Decrease mold temperature. Momentarily release pressure during flow stage.

Typical problems of compression mounting materials

Case hardening and blister		
Thermoplastic resins		
Cottonball	Powdered media did not reach maximum temperature; insufficient time at maximum temperature	Increase holding time at maximum temperature.
Crazing	Inherent stresses relieved on or after ejection	Allow cooling to a lower temperature prior to ejection; temper mounts in boiling water.

Radial cracks in thermosetting plastics usually result from attempts to mold a specimen whose dimensions are

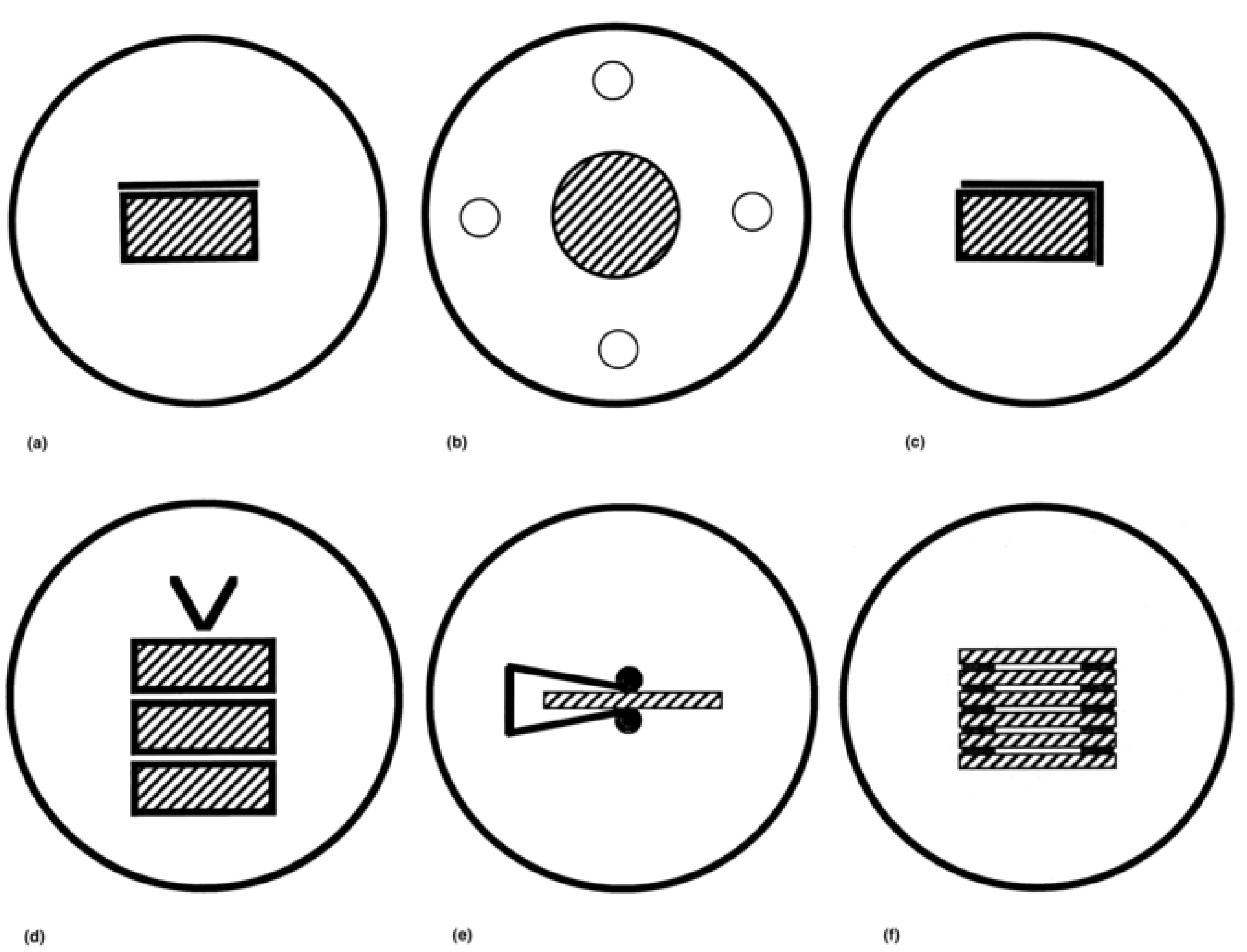
Typical problems of castable mounting materials

Problem	Cause	Solution
Acrylics		
Bubbles	Too violent agitation while blending resin and hardener	Blend mixture gently to avoid air entrapment.
Polyesters		
Cracking	Insufficient air cure prior to oven cure; oven cure temperature too high; resin-to-hardener ratio incorrect	Increase air cure time; decrease oven cure temperature; correct resin-to-hardener ratio.
Discoloration	Resin-to-hardener ratio incorrect; resin has oxidized	Correct resin-to-hardener ratio; keep containers tightly sealed.
Soft mounts	Resin-to-hardener ratio incorrect; incomplete blending of resin-hardener mixture	Correct resin-to-hardener ratio; blend mixture completely.

Typical problems of castable mounting materials

Defect	Cause	Solution
Tacky tops	Resin-to-hardener ratio incorrect; incomplete blending of resin-hardener mixture	Correct resin-to-hardener ratio; blend mixture completely.
Epoxies		
Cracking	Insufficient air cure prior to oven cure; oven cure temperature too high; resin-to-hardener ratio incorrect	Increase air cure time; decrease oven cure temperature; correct resin-to-hardener ratio.
Bubbles	Too violent agitation while blending resin and hardener mixture	Blend mixture gently to avoid air entrapment.
Discoloration	Resin-to-hardener ratio incorrect; oxidized hardener	Correct resin-to-hardener ratio; keep containers tightly sealed.
Soft mounts	Resin-to-hardener ratio incorrect; incorrect blending of resin-hardener mixture	Correct resin-to-hardener ratio; blend mixture completely.

Examples of special mount arrangements. (a) Sheet placed next to the specimen in an epoxy mount for edge retention. (b) Epoxy mount with rod at each quadrant for specimen flatness and edge retention. (c) Mount with an L-shaped strip to indicate specimen orientation. (d) V-shaped metal strip in a mount to indicate orientation. (e) Epoxy mounts with binder clips to hold the specimen perpendicular to the polished surface. (f) Mount with sheet specimens separated by double-sided tape at the ends.



Heat Tinting

- Oxide films can be formed by heat tinting. The polished specimen is heated in an oxidizing atmosphere.
- Coloration of the surface takes place at different rates according to the reaction characteristics of different microstructural elements under the given conditions of atmosphere and temperature. The thickness of the film is influenced by differences in chemical composition and crystallographic orientation, and the observed interference colors allow the distinction of different phases and grains.

Heat Tinting

- Different metals require different oxidation durations and temperatures. High temperatures may induce phase transformations on the surface, an effect that sometimes limits application of this technique. Some specimens may oxidize after exposure to ambient atmospheres. This was demonstrated during research on uraniumzirconium alloys (Ref 10). A U-14Zr (at.%) alloy was oxidized 40 min at 900 °C (1650 °F). Several conventional etching techniques were used without success to reveal the characteristics of the oxide/metallic interface. However, after exposing the specimen to ambient atmosphere for 48 h, a thin zirconium-rich layer with slender fingerlike penetrations into the bulk oxide was visible at 2000×.

Heat Tinting

- Heat tinting can also be performed using a more sophisticated procedure in which temperature and oxidation are closely monitored in an enclosed system. This procedure has been used in studies of surface reactions of single crystals (Ref 11). Heat tinting is also be preceded by chemical etching to reveal grain and phase boundaries. This has proved successful with uranium alloys, uranium carbides (Ref 12, 13), zirconium and its alloys, high-speed tool steels, and austenitic stainless steel weldments.

Color Etching

- Color etching, also commonly referred to as tint etching, has been used to color many metals and alloys, such as cast irons, steels, stainless steels, nickel-base alloys, copper-base alloys, molybdenum, tungsten, lead, tin, and zinc. Satisfactory color, or tint, etchants are balanced chemically to produce a stable film on the specimen surface. This is contrary to ordinary chemical etching (discussed in the section "Etching" of this article), when the corrosion products produced during etching are redissolved into the etchant.

Color Etching

- Immersion color etchants that produce color contrast are associated with Klemm and Beraha, whose work is described in Ref 14 and 15. Color etchants work by immersion, never by swabbing, which would prevent film formation. Externally applied potentials are not used. Color etchants have been classified as anodic, cathodic, or complex systems, depending on the nature of the film precipitation (Ref 2). Tint etchants generally color one anodic phase. Some success has been attained in developing color etchants for steels that are selective to the phases that are normally cathodic. However, most tint etchants color the anodic phases. Color etchants are usually acidic solutions, using water or alcohol as the solvent. They have been developed to deposit a 0.04 to 0.5 μm (1.6 to 19.7 μin.) thick film of an oxide, sulfide, complex molybdate, elemental selenium, or chromate on the specimen surface.

Color Etching

- The colors produced by color (tint) etchants are visible under bright-field illumination, and in many cases further enhancement is attained using polarized light. Colors are developed by interference in the same manner as with heat tinting or vacuum deposition. As noted, color is determined by the thickness of the film, usually in the sequence of yellow, red, violet, blue, and green when viewed using white light. With anodic systems, the film forms only over the anodic phase, but its thickness can vary with the crystallographic orientation of the phase. For cathodic systems, because the film thickness over the cathodic phase is generally consistent, only one color is produced, which will vary as the film grows during etching. Therefore, to obtain the same color each time, the etching duration must be constant. This can be accomplished by timing the etch and observing the macroscopic color of the specimen during staining.

Color Etching

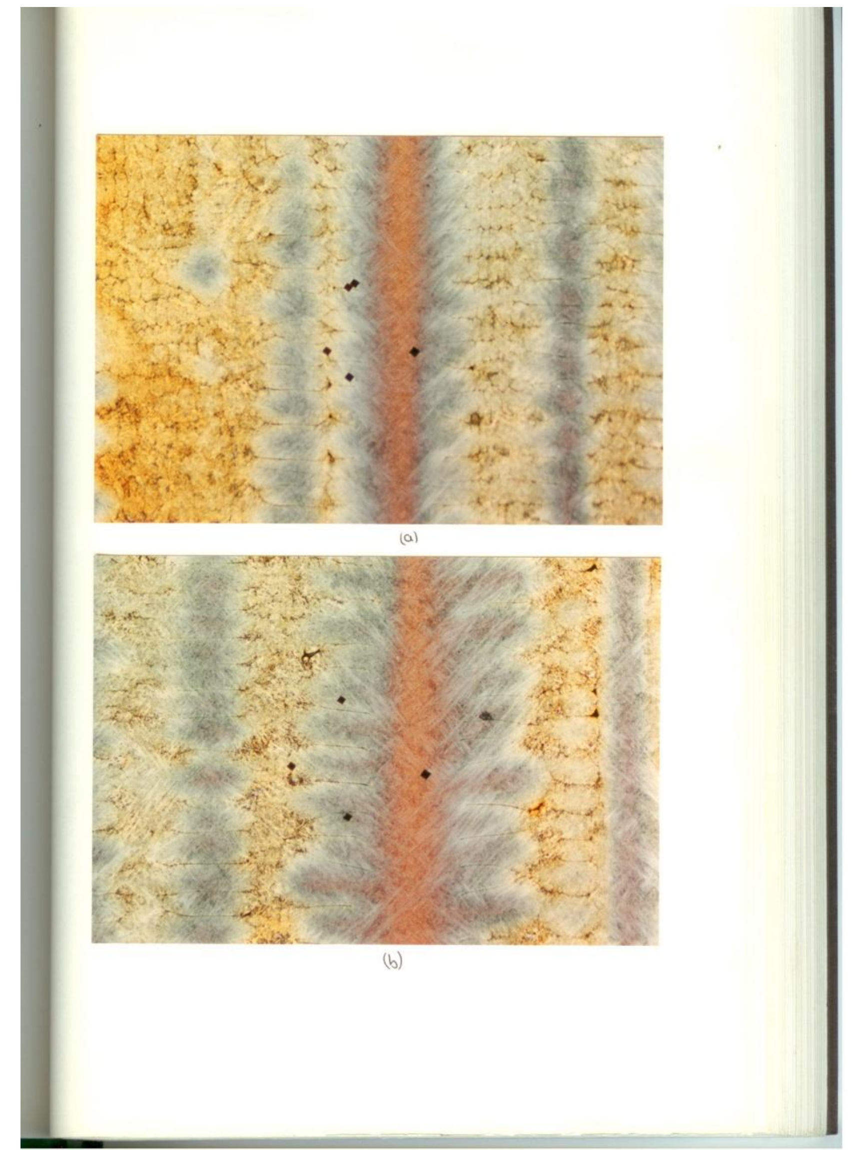
- Specimens for color etching must be carefully prepared during polishing. Control of scratches is the most challenging difficulty, particularly for alloys such as brass. Scratches are often observed after color etching, even if the specimen appeared to be free of scratches before polishing. This is a common problem with techniques that use interference effects to produce an image. However, preparation is carried out in virtually the same way as for specimens that would be chemically etched, but greater attention must be given to fine scratch removal.

Color Etching

- Color etchants have been developed that deposit a thin sulfide film over a wide range of metals, such as cast irons, steels, stainless steels, nickel-base alloys, copper, and copper alloys. These films are produced in two ways. For reagents containing potassium metabisulfite (K2S2O5) or sodium metabisulfite (Na2S2O5), the iron, nickel, or cobalt cation in the sulfide film originates from the specimen, and the sulfide anion derives from the reagent after decomposition.

Color Etching

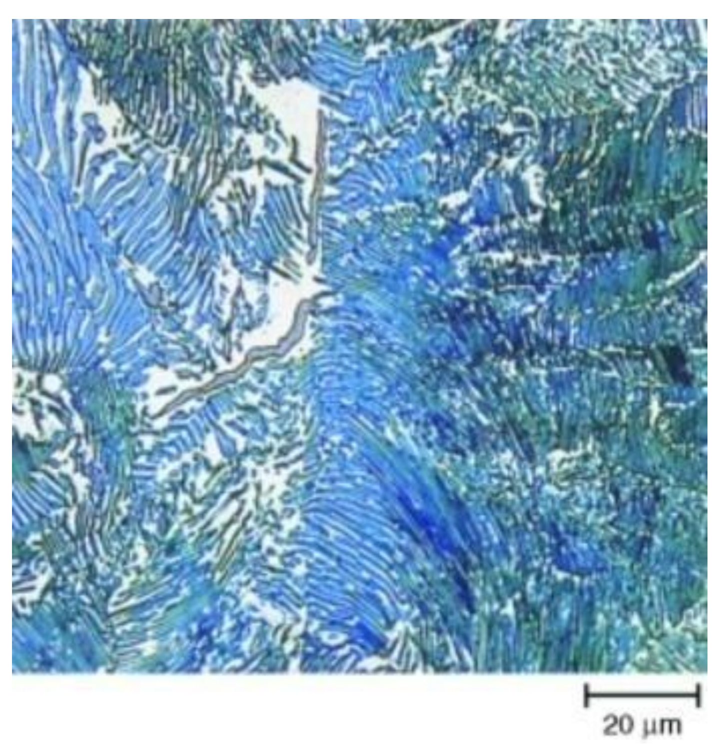
Color Etching

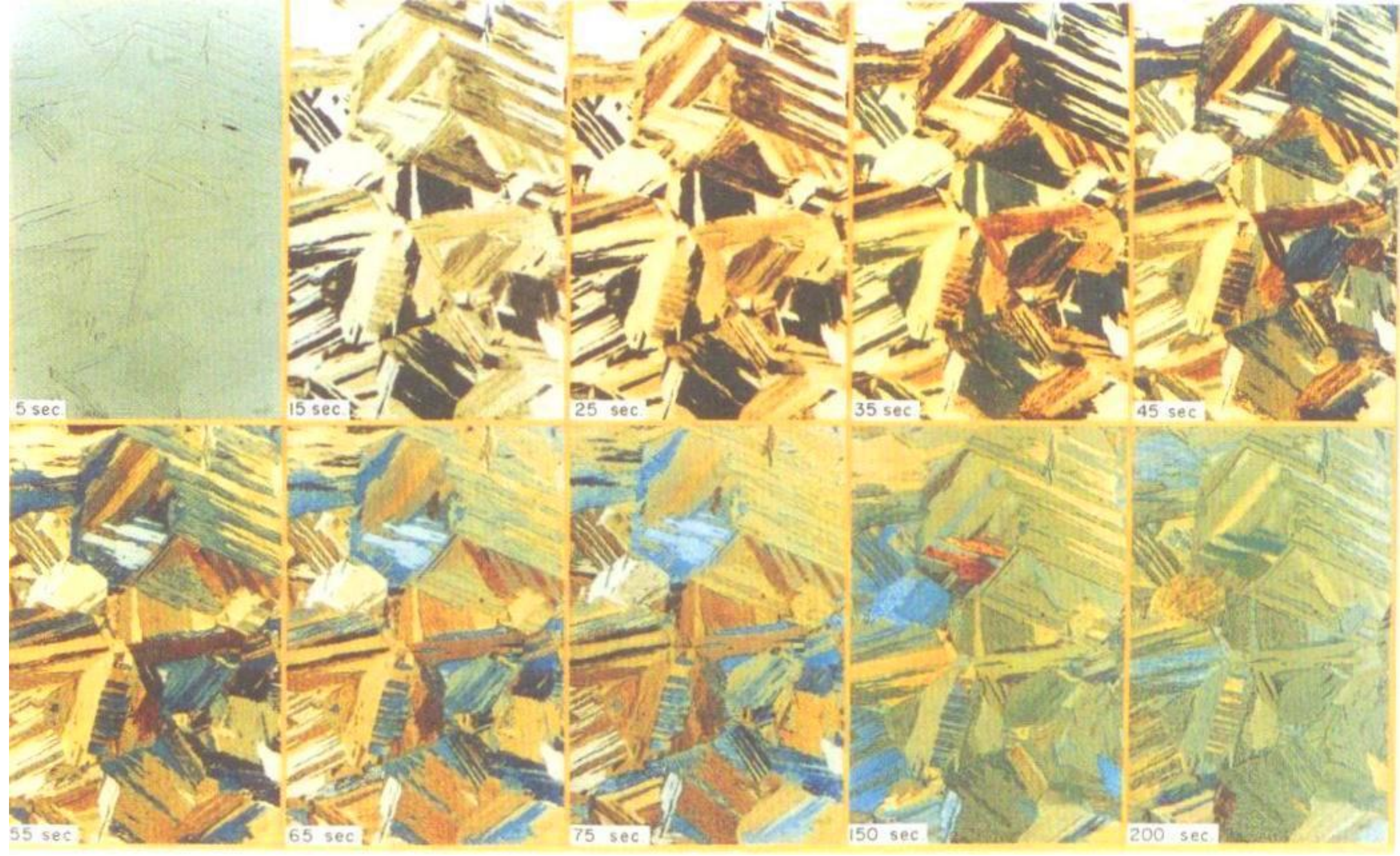


Color Etching

- The second type of film is produced by a metal-thiosulfate complex in the reagent that consists of an aqueous solution of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$), citric acid (organic acid), and lead acetate ($Pb(C_2H_3O_2)_2$) or cadmium chloride ($CdCl_2$) (metal salt). In such etchants, the specimen acts as the catalyst, and the film formed is lead sulfide (PbS) or cadium sulfide (CdS). These reagents color only the anodic constituents; the film is not formed over the cathodic features. Color etchants that use reduction of the molybdate ion have also been developed (Ref 17). Sodium molybdate ($Na_2MoO_4 \cdot 2H_2O$) is used. Molybdenum in the molybdate ion, Mo has a valence of +6. In the presence of suitable reducing compounds, it can be partially reduced to +4. A dilute (1%) aqueous solution of $Na_2MoO_4 \cdot 2H_2O$ is made acidic by the addition of a small amount of nitric acid (HNO_3). This produces molybdic acid (H_2MoO_4). Addition of a strong reducing agent, such as iron sulfate ($FeSO_4$), colors the solution brown.

Fe-1C alloy etched with acidified 1 g Na2MoO4 in 100 mL H2O to color the cathodic cementite. The cementite in the pearlite is blue; grain-boundary cementite is violet. 500×. (G.F. Vander Voort)





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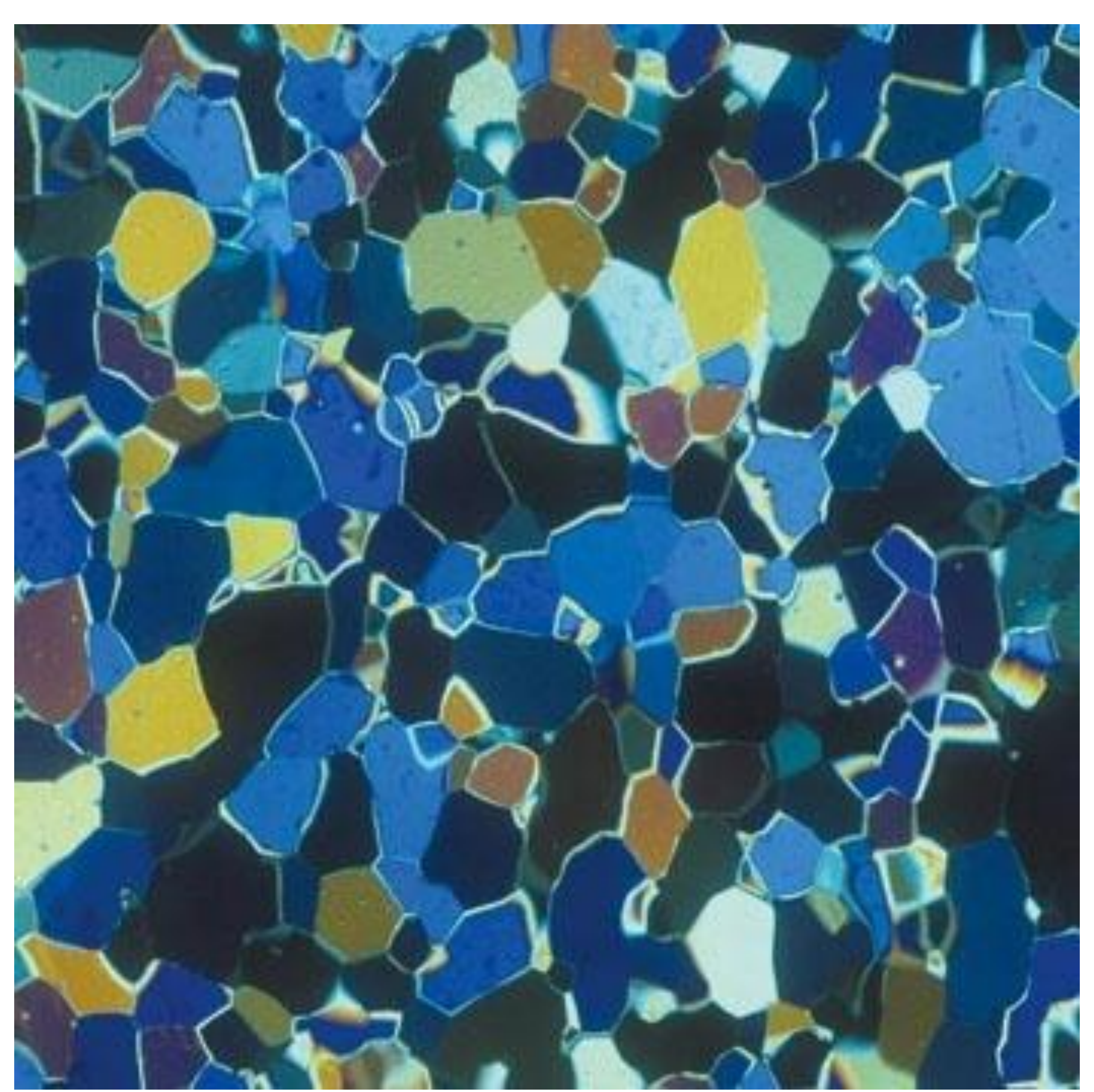

Plasma Etching

- Plasma etching is a process similar to sputter coating - commonly employed for depositing conductive films for SEM inspection. However the process is working in reverse with the specimen forming the cathode in the vacuum reaction cell. High voltage is applied between the anode and cathode which oppose each other with a small gap between. The cathode is the 'target' on which the sample is placed. Gas, usually an inert gas such as argon is leaked under controlled conditions into the vacuum. The gas atoms become positively ionized in the high electrostatic field between the electrodes.

Plasma Etching

- The positively charged gas ions accelerate toward the cathode and bombard the sample, eroding the surface in the process. There are many different suppliers and models of plasma etching equipment on the market. Those with low power rating are generally referred to as cleaners, whereas those with higher power or wattage are termed etchers. Plasma etching should generally be used as a means of cleaning or improving a mechanically or electrolytically prepared surface.