Metallographic Sample Preparation

Stages of Preparation

METALLOGRAPHIC SAMPLE PREPARATION

Cornelius A. Johnson, Met.
Leco Corporation

INTRODUCTION

Metallography, the study of microstructures, has been an invaluable tool to the progress of science and industry over the past three centuries. The question could be posed, 'Why Metallography?' The first, most obvious answer would be that a part or structure cannot be designed to a critical criteria if a given lot of material is not within specification. Metallography is one major valuable tool which can be a precise aid for:

a) Quality control for material processing, material product and hardware.
b) Determining reasons for failure.
c) Advanced Technology.
d) Prediction for better demands.

REQUISITES

The key to obtaining an accurate interpretation of a microstructure is a properly prepared specimen which is truly representative of the material being examined.

The definition of a properly prepared metallographic surface states that the section must:

a) Be flat, free from scratches, stains and other imperfections which tend to mar the surface.
b) Contain all non-metallic inclusions intact.
c) Show no chipping or galling of hard and brittle intermetallic compounds.
d) Be free from all traces of disturbed metal.

To insure achievement of such true surfaces, preparation must be carried out, not only with accuracy but also, with a clear understanding of what must be accomplished during each specific stage.

STAGES OF PREPARATION

The most straight-forward approach is to divide the entire process into a logical series of stages involved and the purpose of same.

Stage 1 — Sectioning

The removal of a representative sample from the parent piece.

Stage 2 — Coarse Grinding

Producing an initial flat surface.

Stage 3 — Mounting

Embedding the sample in a plastic medium for ease in manipulation and other factors such as fragility, edge preservation, etc. This stage is sometimes omitted for certain methods of preparation or in instances where it would serve no purpose.

Stage 4 — Fine Grinding

Remove the zone of deformation caused by Sectioning and Coarse Grinding and limit the depths of deformation during this stage by proper abrasive size sequencing.

Stage 5 — Rough Polishing

Further limitation of the deformation zone produced by Fine Grinding.

Stage 6 — Final Polishing

Removal of deformation zone produced during Rough Polishing. Any zone produced at this stage should be minimal and generally will be removed during etching.

METHODS OF PREPARATION

Any material can be prepared by MECHANICAL PREPARATION — hand or semi-automatic methods. The sequence of stages previously defined are necessary in their entirety for this particular procedure.

ELECTROLYTIC POLISHING may often be used as an alternate for the Rough and Final Polishing stages or an overall improvement after Final Polishing by other methods.

SLURRY (ETCH-ATTACK) POLISHING will supplement both Rough and Final Polishing in some instances and Final Polishing in others.

CHEMICAL POLISHING is usually employed after Final Polishing.

The choice of any method is dependent upon the material to be prepared and particularly the phase relationships and distribution within the existent microstructure.

Details on these alternate and supplementary techniques are more completely detailed later.

SURFACE DEFORMATION

During Sectioning, Coarse Grinding and to a lesser extent, during Fine Grinding, a transitional surface zone of deformed metal results from abrasion. Even though this deformation zone is transitional, abrasion has caused the material to exceed the elastic limit and accordingly, permanent plastic deformation has occured. Possible damage traceable to improper sectioning techniques, see Figure 1.

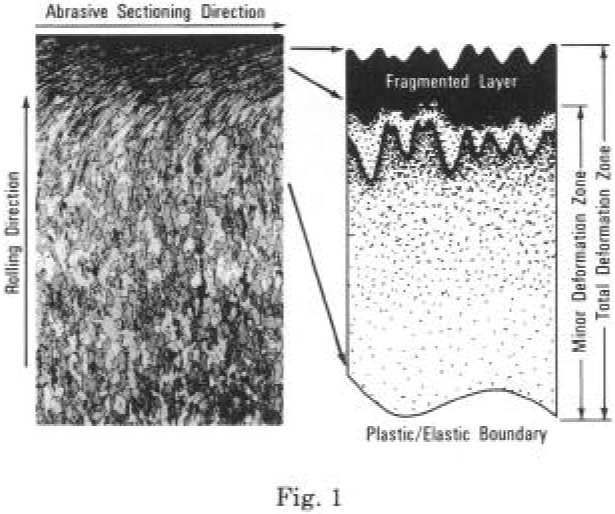
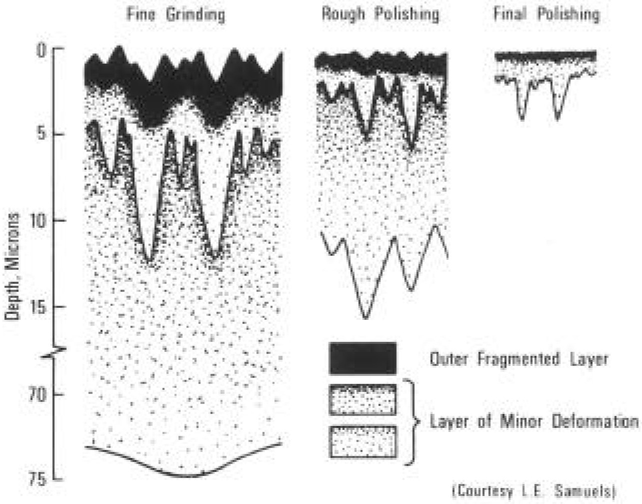


Fig. 1

According to L.E. Samuels, the abrasion effects create a fragmented layer wherein the surface grains have been broken down into sub-grains with a preferred orientation, see Figure 2. Many intermediate strains boundaries extend in rays from the "V" contours of the scratches



(Courtesy L.E. Samuels)

Fig. 2

The strain levels decrease as the plastic elastic boundary is approached. These strain boundaries are not uniformly distributed since the abrasive action and resultant scratch depth of each grain varies due to sizing, shape, hardness and dynamic strength. A conservative estimate would be that plastic deformation would never be less than fifty times the scratch depth.

At the conclusion of Rough and Final Polishing, the thickness of the induced fragmented layer and the accompanying zone of minor deformation have been slightly decreased. The plastic elastic boundaries will now contour the original scratches.

Scratch depths are dependent upon abrasive size and this effects the magnitude of the strain boundary levels. Scratch depth and total zone deformation can be considered inversely proportional to an increase in material hardness, see Figure 2A.

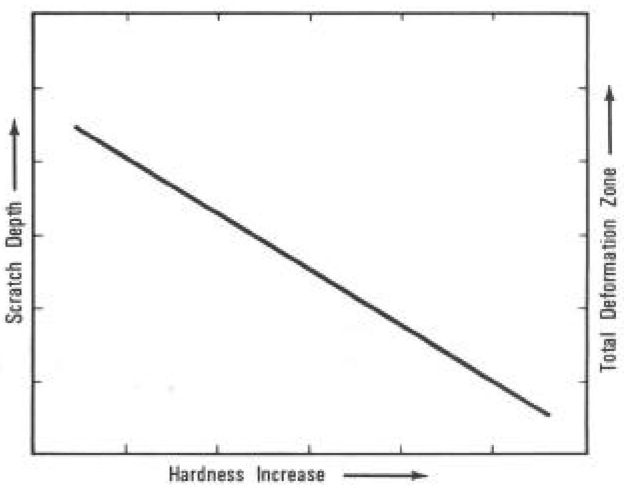


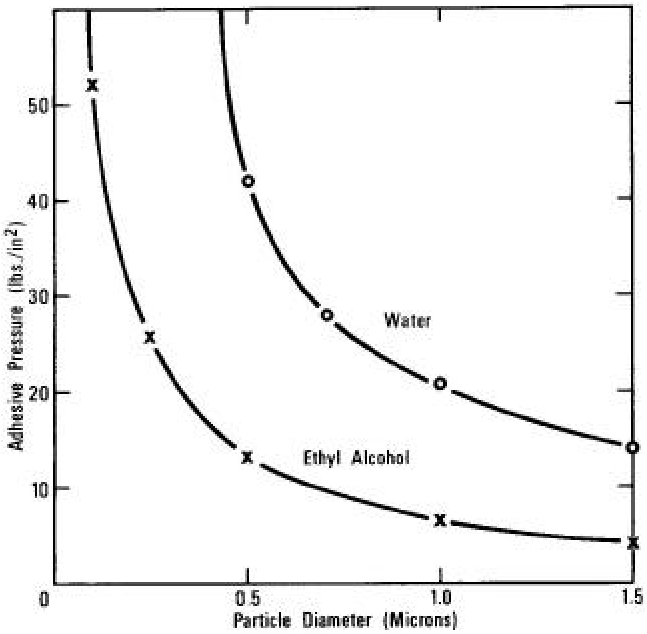
Fig. 2A

PRESSURE

There are two factors which contribute to pressure applied against a sample, the externally applied load and the adhesive pressure created by the surface tension of the vehicle (lubricant, extender). These pressures are important as they control the scratch depth and subsequently the total depth of deformation.

For the coarser abrasive sizes used in the Coarse and Fine Grinding Stages, the distance between the specimen and lap is relatively large due to the particle size. In these instances, the principle effective pressure is the specimen weight and the externally applied load.

With particle size in general use for polishing, the specimen-lap distance becomes significantly less, and this distance will approximate the particle diameter. Theoretical considerations show a sharp rise in adhesive pressure, see Figure 3.



(Courtesy of C. Johnson & J. M. Parks)

Fig.3

Each abrasive size and type must be considered by itself in regard to maximum feasible pressure and this pressure will show an increase with a decrease in particle size. Within reasonable pressure limitations, the depth of damage is slightly affected. Therefore, it is desirable to employ correspondingly high pressures to obtain maximum removal rates.

REMOVAL RATE

Material removal rate will increase linearly with pressure to a critical point and then taper off, see Figure 4.

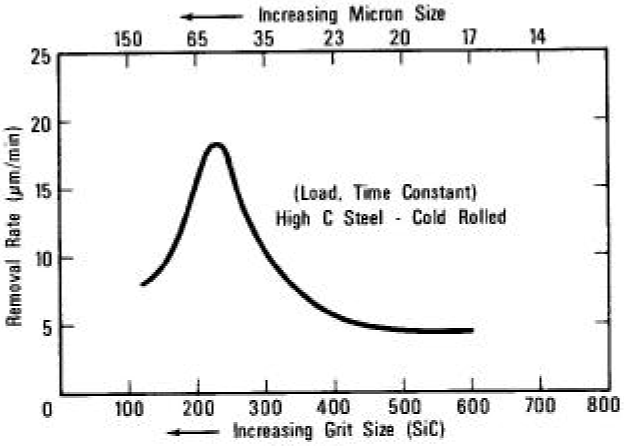


Fig. 4

The coarse size range (50-180 grit/350-70 microns) commonly used abrasive materials for Coarse Grinding do not have sufficient dynamic strength to prevent fracturing. When fracturing does occur due to excessive pressure, the surface may become impregnated with fragmented particles. Such fracturing can account for the low removal rates for 120 and 180 grit sizes. In normal metallographic sequencing, the use of such abrasive sizes is fortunately avoidable. One can readily start with a 240 grit which has a much higher removal rate, shallower scratch depth and consequently a decrease in total depth of deformation, see Figures 5 and 6.

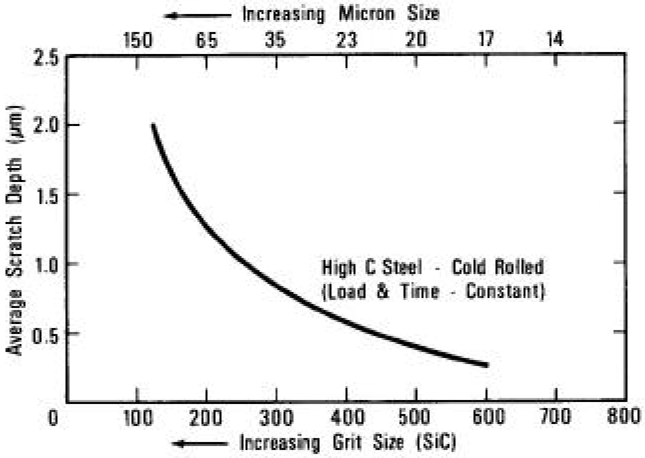


Fig. 5

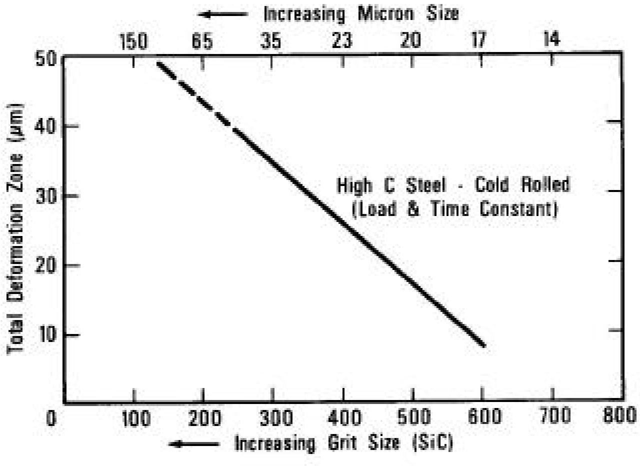


Fig. 6

ABRASIVE SIZING

The abrasive size ranges applicable to the various stages of preparation are shown in Figure 7. Comparative sizing values for the more commonly employed abrasive families appear in Table I thru IV.

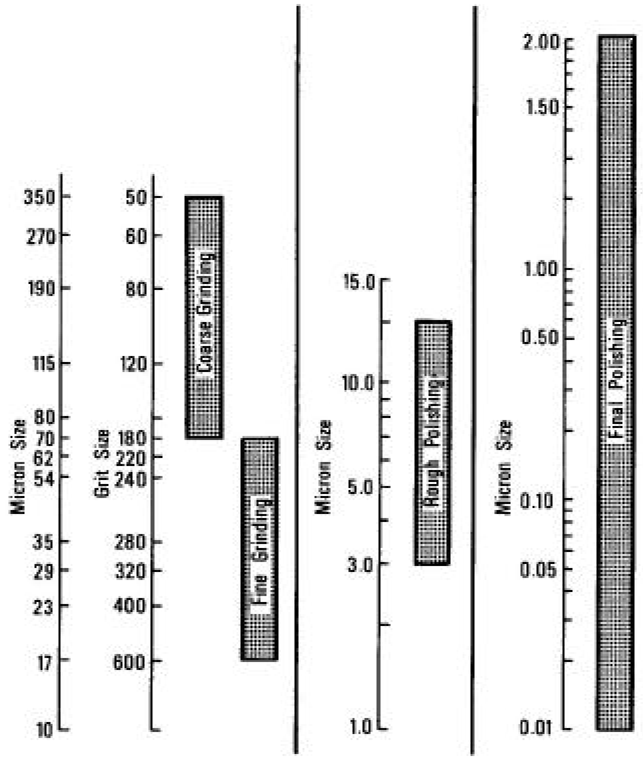


Fig. 7

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 II.
EMERY

Grit No.	Particle Size (AV. μm)
3	85
2	70
1	50
1/0	33
2/0	30
3/0	28
4/0	25

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	12-22	1,200
30	22-36	600
45	36-54	325
60	54-80	230 - 325
90***	......	170 - 230

* National Bureau Standards

** Ultra fine grades, not covered by NBS

*** ANSI B 74.16 - 1971, not covered by NBS

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	5.0	Rough Polishing
Gamma Alumina	0.05	Final Polishing
Alpha Alumina	0.3	Rough and/or Final Polishing
Chrome Oxide	1.0	Rough Polishing
Chrome Oxide	0.05	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

STAGES OF PREPARATION

STAGE 1 — SECTIONING

Sectioning is the removal of a representative area from the parent piece. The microstructure must not be altered in the process. Heat or cold working are the two most likely conditions which would quickly bring about structure changes.

A. HIGH SPEED ABRASIVE SECTIONING

Quite obviously operations such as sawing or shearing are not preferable due to the deformation produced. Abrasive cutting offers the best solution to eliminate or minimize heat and deformation.

To cut properly, a bonded abrasive wheel must be matched to the cut-off machine. Primary considerations are surface speed (SFM) for a given wheel diameter and the type of cooling system employed. Selection must be made from the proper family of abrasive wheels to meet the requirements for the vast variety of materials and hardness levels. The principle controlling and guiding variables for wheel selection can be classified as follows:

a) Abrasive Grain
    - Type Al_2O_3 or SiC
    - Grit Size

b) Bond
    - Rubber
    - Resinoid
    - Resin/Rubber

c) Hardness of Bond
    - Soft
    - Medium
    - Hard

d) Density (Structure)
    - Open
    - Dense

ABRASIVE GRAIN — Aluminum Oxide is generally recommended for sectioning steels and high tensile strength materials. Silicon Carbide abrasives are used for some grades of iron, non-ferrous materials, Titanium, Zirconium, Uranium and their alloys.

Regardless of bond hardness, the coarser grit sizes will produce a harder action. However, the cutting action will be more open as the clearance of cut will be greater. Finer grits result in a softer action and a smoother surface.

BOND — The purpose of the bonding material is to hold the abrasive grains in place. In general, rubber bonded wheels are used for wet operation and are best suited for metallographic specimens. Resinoid bonds are used for dry cutting. Resin/Rubber can be used wet or dry and may offer an economy factor.

To cut clean and fast, the bond must wear away or break down rapidly enough to expose the new abrasive grains.

Softer bond wheels are used for sectioning hard metals and alloys whereas harder bond wheels are used for softer materials. As bond hardness increases, the wheel wear is decreased.

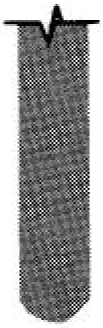
The rate of bond breakdown is related to several factors:

(a) Bond hardness.
(b) Hardness and workability of sample.
(c) Size and speed of abrasive wheel.
(d) Power of driving motor.
(e) Type, amount and method of coolant application.
(f) Amount of pressure applied to wheel.

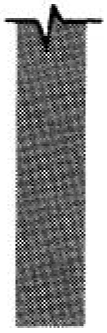
COOLING — Sufficient and proper cooling is very important. High volume jet spraying or submerged cutting are the two major techniques used. Section size, material and hardness dictate which method should be employed. Submerged cutting will tend to make a wheel bond act harder.

WHEEL SPEED — Speed (SFM) must be carefully considered both in the design of a cutter and the selection of wheels for a given cutter. In general a given wheel bond will act harder as speed is increased.

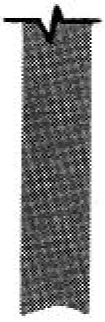
WHEEL EDGE WEAR may be used as a very good guide to indicate whether the proper wheel has been selected.



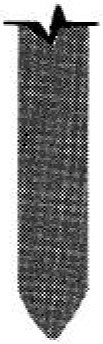
Rounded edges signify the correct wheel properly applied to the cutting of solids.



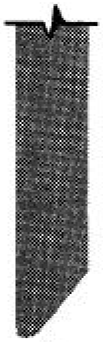
Square edges are retained on a wheel well suited to cutting both solids, sample standards and tubing of medium wall thickness.



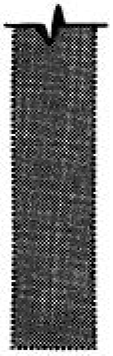
Concave edges indicate proper wheel to section light wall tubing of thin wall sections.



Pointed edges indicate wheel bond is too hard.



Tapered (chisel) edge is caused by improper application of coolant.



Glazed edges occur when the bond is not breaking down properly.

B. LOW SPEED, LOW DEFORMATION, PRECISION SECTIONING

The technique of low speed sectioning for metallographic and related type specimens is patterned after principles from the precious gem industry.

Small diameter, four to six inch diamond rimmed wheels are used. The speed range is 0-1000 RPM and the load range, 0-1000 grams. The technique is applicable to many types of materials. Response is excellent to metals and non-metallics — soft, hard, brittle, ductile, porous, simple or complex configurations, composites, etc..

The resultant surfaces are extremely smooth with very little surface deformation. Tendencies toward cracking at brittle-ductile interfaces in laminated or deposited materials is nonexistent. Brittle phases within a complex microstructure do not tend to "check" or "pluck".

STAGE 2 — COARSE GRINDING

The purpose of Coarse Grinding is to remove deformation produced during Sectioning and provide the initial flat surface. A secondary purpose may often be to remove gross amounts of surface material for microsample preparation or macroetching.

The process is performed on abrasive belts or disc covered rotating wheels. The size range is 50 to 180 grit. Water is recommended as a coolant to prevent overheating of the specimen and flush away the surface removal products, thus keeping sharp abrasive grain exposed at all times.

Abrasive belts and discs are available in silicon carbide and aluminum oxide with resin bond for wet or dry operation. Garnet coated materials are only available with glue bond and can only be used dry.

The abrasive action is very aggressive with this grit range. With higher speed Coarse Grinding, i.e. increased surface feet per minute, the resultant surface finish for a given grit size will approach that produced by a finer grit size. For example, a surface finished with 60 grit/5200 SFM would be equivalent to one produced by 120 grit/2500 SFM.

A surface which appears smooth and bright does not necessarily have the least (shallowest) amount of deformation. An apparent improved appearance can be due to rubbing or smearing of the surface by the abrasive particles not cutting cleanly. Grinding with worn or loaded abrasive surfaces will produce more extensive surface deformation.

STAGE 3 — MOUNTING (See Section B)

STAGE 4 — FINE GRINDING

The abrasives used for Fine Grinding are Silicon Carbide, Emery and Aluminum Oxide. Generally, fixed type abrasives are used, i.e. the abrasive grain is bonded to a paper or cloth backing. The bonding material may be glue, resin or resin over glue. Silicon Carbide and Aluminum Oxide materials are available with either a non waterproof paper backing with glue or resin bond for dry operation; or waterproof cloth or waterproof paper backing with resin bond for wet or dry operation. Emery coatings are only fabricated with a glue bond.

Preference is for wet operation which offers a flushing action to prevent the surface from becoming clogged with removal products. Flushing will also keep the cutting edges of the abrasive grains exposed.

Silicon Carbide abrasive grain when used wet has a removal rate twice that of a corresponding grade of Emery and provides a much shallower deformation depth.

Aluminum Oxide has a lower hardness value than Silicon Carbide which could exhibit higher dynamic strength and therefore decrease shear or fracture values; and correspondingly effect deformation depth.

STAGE 5 — ROUGH POLISHING

This stage may be considered the most important in the entire preparation sequence. The nature of the abrasive type employed should permit accurate sizing and separation by various methods into fractions of uniform particle size.

ABRASIVES

Diamond abrasives fall into the above category. Other contributing properties are high hardness, inertness and low coefficient of fricton. Diamond particles retain their shape and size during abrasion and produce a uniform and high rate of material removal with minimal induced surface damage. Removal rates may often exceed those produced during the Fine Grinding sequence, see Figures 8 and 9.

SUSPENSION MEDIUM

The suspension medium for diamond powders is very important as it provides particle suspension, contributes to lubrication, and removal rate. Some adjustments in viscosity of the medium must be made for various particle sizes to compensate for possible drastic changes in heat generation.

Oil or water soluble media promote superior lubrication and removal rates in comparison to slurry suspensions. The reason being the particles are uniformly dispersed and held in a definite suspension. The paste-like material facilitates convenient charging of the polishing cloth surface and the addition of an extender contributes to even particle distribution over same.

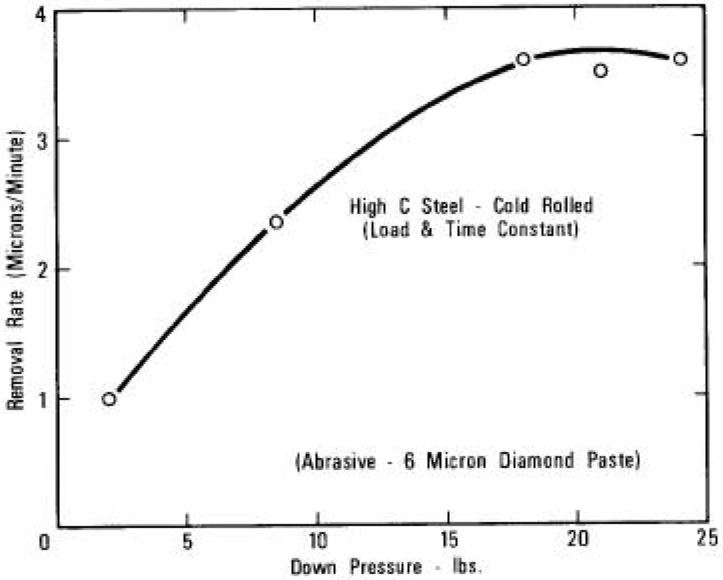
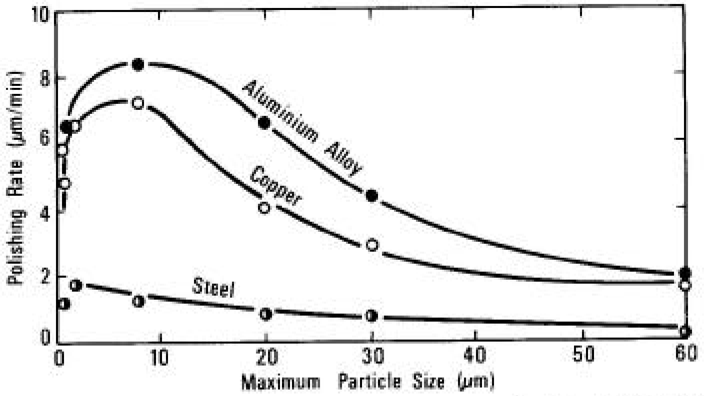


Fig. 8



(Courtesy L.E. Samuels)

Fig. 9

ABRASIVE SELECTION

A sufficiently coarse abrasive should be selected to accomplish this stage in a minimum time. The time factor will greatly influence relief effects. However, size selection is greatly dependent upon the particle sizes and material types to be used in subsequent operations.

Particle sizes in general use are in the overall size range of 0-10 microns. The 6 micron (range 4-8) classification will produce the highest removal rates for most materials. A sharp decrease in removal rates under similar conditions is to be expected for the lower micron and sub-micron ranges, see Figure 9.

As previously stated, removal rates will increase linearly with pressure to a critical point, see Figure 4.

At the conclusion of Rough Polishing, the sample surface will naturally show scratches of visible dimensions and there will be localized deformation associated with these scratches.

POLISHING CLOTHS

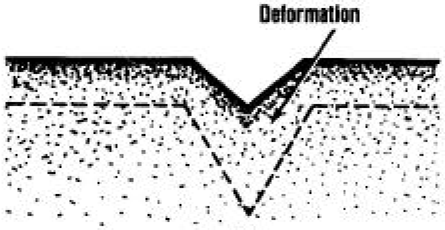
The type of cloth used for this stage has an extremely important bearing on the end result. It is imperative that relief between microconstituents of varying hardness and sample mount interfaces be held to a minimum. Napless cloths such as nylon, cotton, chemotextile materials, etc., should be used. Cloths of this nature will hold relief and undercutting at interfaces to a minimum as "pile whip" is non-existent. Selection should also be such that the cloth itself does not produce any abrasive artifacts. The hardness of the material being prepared is the guide point.

STAGE 6 — FINAL POLISHING

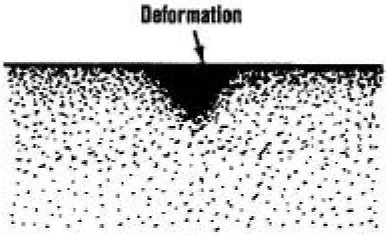
As previously stated, the Final Polishing Stage serves to remove any deformation zone resulting from Rough Polishing. Here, a uniformly polished and scratch-free surface must be produced.

Care must be taken to insure removal of any and all surface deformation. If this is not accomplished, scratches may still be apparent in the unetched state. The same artifacts will appear and to an even greater extent if any preceding steps or stages were not properly accomplished.

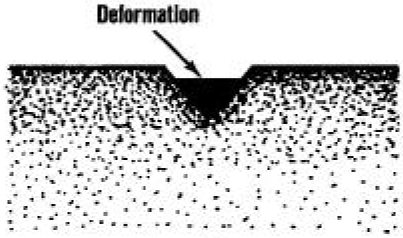
Scratches may also be evident after etching. This signifies the deformed surface was not completely removed, see Figure 10. The etchant attack will be more severe and preferential along those regions of localized deformation as they possess higher surface energy levels.



(a)
Original Scratch



(b)
Polished Surface



(c)
Etched Surface

Fig. 10

A prolonged series of alternate etching and repolishing is generally discouraged as a means to remove deformation. Relief effect tendencies can accrue with an increase in number of etch-repolish cycles. Relief can also be attributable to preferential attack of the localized deformed areas or selective attack of certain phases or grain orientations.

ABRASIVES

A wide variety of abrasive materials are used for Final Polishing. The most common are Aluminum Oxide, Chrome Oxide, Magnesium Oxide, Cerium Oxide, Silicon Dioxide and Diamond.

Aluminum Oxide is the most extensively used material. Two types are available, levigated and the high purity synthetic powders. Preference is for the synthetic materials in either powder or suspension form within the one micron and sub-micron size range. Particle size and crystalline structure are dependent upon temperature. The gamma type, low temperature form, is sized 0.05 microns. The particle sizes, 1.0 and 0.3 micron, are the high temperature alpha structure. Particles are grown to size by precisely controlling the temperature range. The alpha lattice is slightly harder than the gamma form.

Ferrous, copper, titanium, zirconium based materials and super-alloys are compatible with alumina abrasives. General preference is the gamma type. However, with some materials the alpha form may be profitably used as an intermediate step.

Other commonly used media are liquid suspension of chrome oxide and chrome oxide/cerium oxide blends. In many instances, these are unsurpassed for the graphitic irons and ferrous materials containing complex inclusions or gross amounts of inclusions.

Magnesium Oxide, even though the techniques are somewhat difficult to master, is ideally suited to many materials. Aluminum, magnesium and their alloys are best prepared with this material. The powder has uniformly well shaped particles of considerable hardness and the cutting edges are well defined. Today's high temperature calcining treatments have eliminated problems formerly associated with the subsequent formation of hard carbonates. Any trace alkalis are water soluble.

Cerium Oxide slurries are a relatively new innovation as a final polishing abrasive. The blends, particularly those with small amounts of aluminum oxide, are readily adaptable to a large group of materials. The extremely fine particle size is a definite attribute. However, these solutions have not been exploited to their fullest.

Colloidal suspensions of Silicon Dioxide have been used with remarkable success in the electronic wafer industry. The material shows promise for metallography, but again, efforts have not been sufficiently concentrated for full possible applications.

Diamond abrasives have several sizing levels below the 4-10 micron range (3 micron average, 1 micron average, 0.25 micron average). The logical choice would be the sub-micron grade as this would not be too great a step from the size used in Rough Polishing. The 3 micron or 1 micron gradings would only be used if an intermediate step were desired. The finish produced by even the finest sized diamond is generally only for routine applications. Results are more satisfactory as overall hardness of the material increases.

POLISHING CLOTHS

Napped cloths are generally preferred for this operation. Unfortunately, the fibers are compressible, and therefore tend to conform to the surface of the specimen under the slightest pressure. Due to this type contact the softer phases or grains with a certain orientation have a higher removal rate than the harder phases or grains with differing orientations. Such effects can be avoided or minimized with abrasive selection to shorten the time element.

POLISHING VEHICLE

Distilled or distilled and deionized water is generally used as the suspension or extender vehicle media for those materials which are classified under the metallic oxide category.

Metallic oxides are basic by nature. Ideal polishing conditions are present when solutions are neutral pH 7. Precautions are necessary when considerable electrochemical differences are present between individual areas or phases of a specimen. Severe etching of the anodic phase may occur if the vehicle becomes ionized.

With highly reactive materials or phase relationships, it is sometimes necessary to resort to a nonpolar vehicle, such as ethylene glycol. However, the polishing rate may be severely reduced. Therefore, careful observations are necessary whenever vehicles are altered to offset any chemical attack which may occur during polishing.

The problem is non-existent with diamond abrasives and oil vehicles as the particles are inert and the oil is non-ionizing.

POLISHING WHEEL WETNESS

The wetness or "trim" of the cloth with water-type extenders has a great bearing on the end result. If the cloth is too wet, the sample can show pits; if too dry, buffing and/or smearing can result.

To determine proper wetness, remove the sample from the wheel and check the time necessary for the polishing film to dry. In general, this should take no longer than five to eight seconds. To check for abrasive addition, note the color and consistency of the film. The film should not be opaque, but rather sufficiently transparent to reveal the sample shape and lustre.

When using diamond abrasives, improved removal rates are encouraged by low viscosity oil extenders. The cloth should always show a slight excess of vehicle to insure good lubricity and swarf removal.

MANIPULATION

FINE GRINDING: With manual processing, the sample is firmly held with the fingers. Movement is in a straight line across the abrading surface toward or away from the operator, see Figure 11. When manual dexterity has been achieved, motion in both directions may be employed.

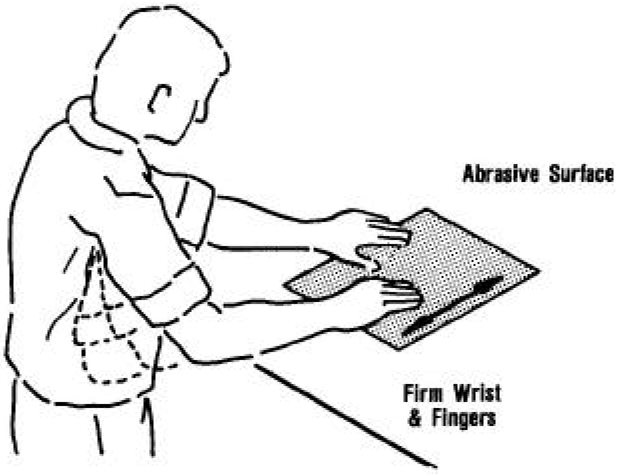


Fig. 11

The operator should be positioned to allow free passage of the elbow past the side of the body for linear coordination between the shoulder and elbow joints.

Finger and wrist joints should remain rigid and shoulder line fixed to aid in even pressure control and produce a planar surface with no faceting.

The specimen is rotated 45° between abrasive steps. The purpose is two-fold; to indicate when the abrasive scratches from the previous step have been removed and to prevent faceting, see Figure 12A.

ROUGH AND FINAL POLISHING: The rotation of the polishing wheel is normally in a counterclockwise direction. The sample should be moved in a clockwise direction around the entire polishing surface to avoid: (1) Directional traces, (2) 'Fishtailing' of certain family type inclusions, (3) "Pull-out' of phases poorly consolidated within a microstructure, such manipulation also provides equal material removal over the entire surface, see Figure 12B.

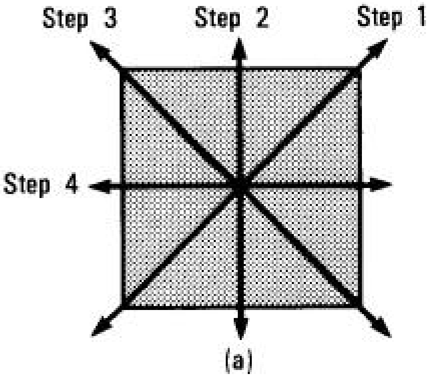


Fig. 12A

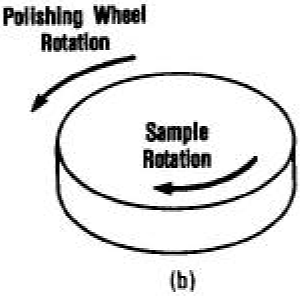


Fig. 12B

PRESSURE: Previous remarks concerning applied pressure have been made. As an overall generalization, maximum feasible pressures should be used to produce maximum removal rates.

CLEANING

GENERAL

Cleanliness is one of the most important requisites in sample preparation. Discouraging or unsatisfactory end results are more often traceable to carelessness rather than to faulty materials. Samples must be carefully cleaned between each stage of preparation to prevent contamination by coarser abrasives being carried over to a finer abrasive stage.

The role of cleanliness also includes operator's hands and equipment. The laboratory layout should be such that the extremely coarse abrasive stages are isolated from those stages involving finer abrasive material. Polishing wheels should be kept covered when not in use.

"A few minutes at the end of each working day should be set aside for general clean-up and monitoring of equipment. The results of the above routine practices are rewarding."

ULTRASONIC CLEANING

Ultrasonic Cleaning is the most effective system for the varying dirt problems encountered in sample preparation. The higher crystal frequencies produce better results.

There are many readily available water soluble detergents. Excellent, and sometimes more effective, commercial materials are also offered. The use of ammoniated solutions is discouraged as they exhibit etching tendencies with numerous materials.

Superior end results are obtained if more than one sample cleaning step is used. This may be very simply accomplished through use of a glass beaker and positioning cover to fit the top of the master tank. Since the glass beaker is acoustically transparent, the ultrasonic energy is transmitted through the tank solution (coupling agent) to the cleaning solution in the beaker, see Figure 13.

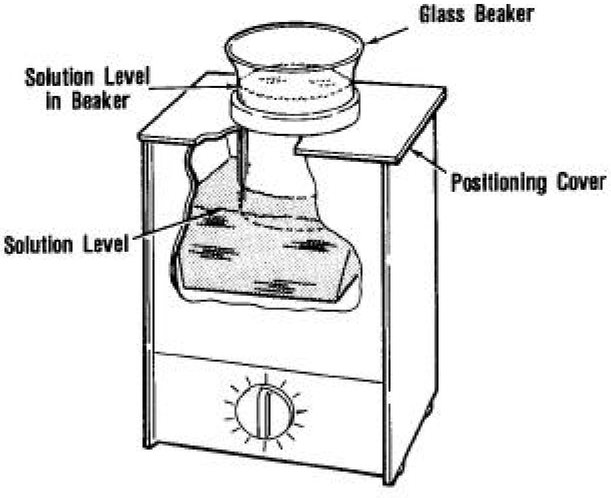


Fig. 13

EXTRACTOR-CONDENSER

The system consists of an Ehrlenmeyer flask, a siphon cup and a glass condensing coil that fits into the top of the flask. The flask is filled with solvent to a level slightly below the overflow tube of the siphon cup. The specimens are placed in the siphon cup suspended from the condensing coil which is connected to a cold water line, see Figure 14.

The flask is heated to the boiling point of the solvent. As evaporation occurs the vapors contact the chilled coil. Condensation drips the solvent into the siphon cup. As the cup fills to a predetermined level, the solvent returns to the flask carrying the oils and dirt removed from the surface and pores of the sample. Recycling continuously provides clean solvent.

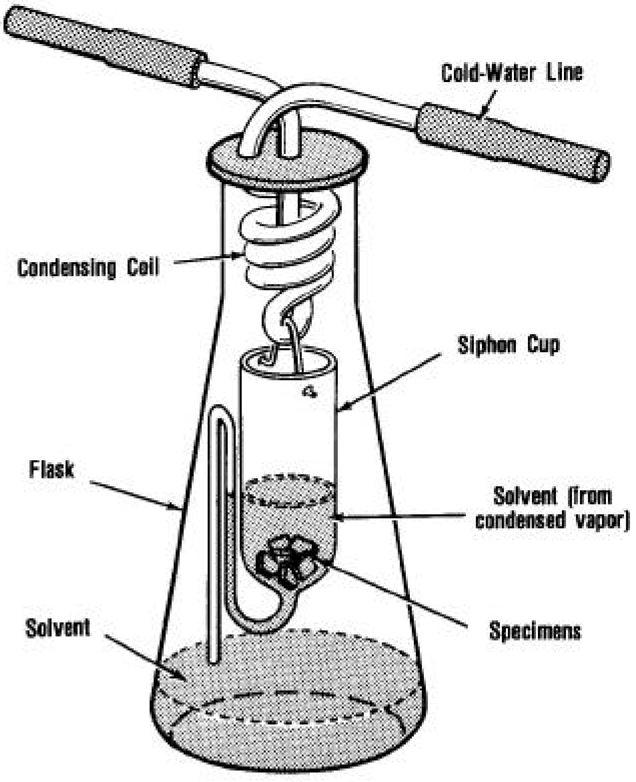


Fig. 14

DRYING

Proper drying of a surface after cleaning or etching is very important. The specimen must be dried quickly to prevent staining or corrosion. After rinsing, the sample is flooded with a high quality alcohol and dried in a stream of warm dry air. With porous materials, an additional rinse in high purity acetone after an alcohol rinse will be very beneficial.

Specimens are generally rinsed with warm water, even after Ultrasonic Cleaning. However, some materials may stain or corrode when rinsed in warm water. In such cases cooler water is recommended.

Page

Mounting Procedures

MOUNTING PROCEDURES

SPECIMEN MOUNTING

Metallographic samples are mounted primarily for ease in manipulation and for edge protection during preparation.

COMPRESSION MOUNTING

Compression molding techniques are used to produce hard mounts in a minimum of time. The materials used are classified as: (1) Thermosetting and (2) Thermoplastic. Thermosetting media requires heat and pressure during the molding cycle and can be ejected at maximum molding temperature. Thermoplastic materials remain fluid at maximum molding temperatures and become dense and transparent with a decrease in temperature and an increase in pressure, see Figures 15 and 16.

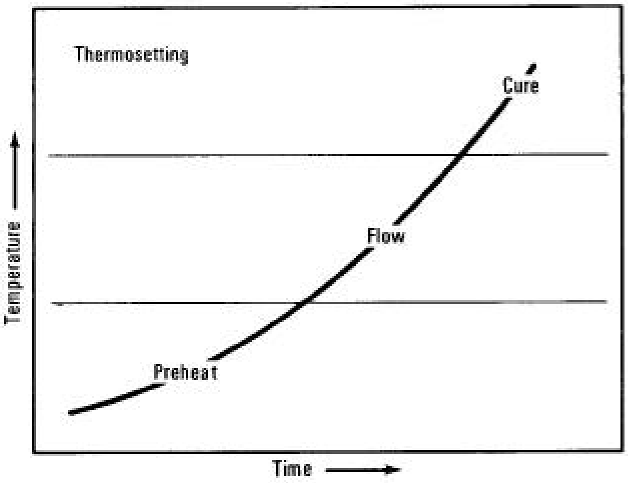


Fig. 15

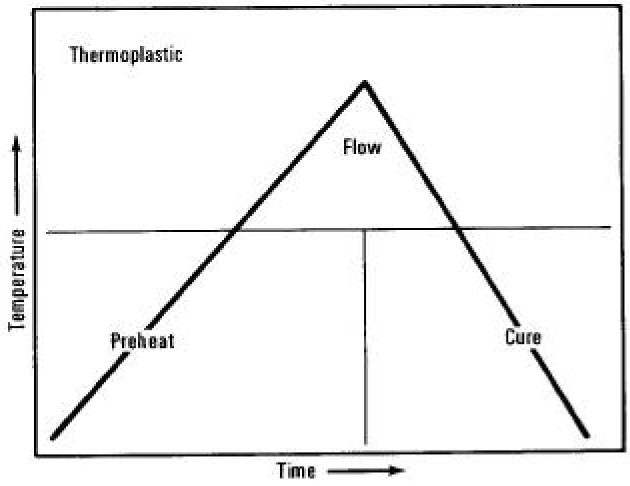


Fig. 16

The variables in compression molding are pressure, temperature and time. By mounting press design the temperature and pressure factors can be held constant.

With more fragile sections powdered material should be used. Normally powdered media should be initiated with the molds at room temperature. This practice is recommended as powdered material has an extremely large exposed surface area and consequently the individual grains, upon contact with heated molds, have a marked tendency to immediately cure without fusion.

Pre-molded thermoset preforms can be used when a section will not be damaged as it is forced into the mounting material by the initial application of pressure.

When transparency is needed for locating a particular area Lucite® is the best mounting medium to use. Very light pressures are used during the preheating and flow cycles. Even though high pressures are normally recommended for the cure cycle, lower pressures may be used with no undesirable effects.

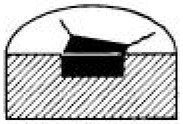
The material readily flows into small areas. This factor plus the allowable pressure variances make the material very desirable for small, fragile pieces. These possibilities very often offset the longer times involved in molding transparent mounts.

Table V.
Molding Temperatures and Pressures

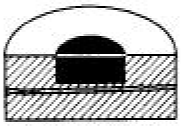
Material	Form	Classification	Molding Temp (°F)	Molding Pressure (psi)
Bakelite®	Powder	Thermosetting	270°-300°	4200
Bakelite®	Preform	Thermosetting	270°-300°	4200
Diallyl-Phthalate	Powder	Thermosetting	280°-290°	2500-3000
Epoxy	Powder	Thermosetting	280°-300°	1000-3000
Lucite® (transparent)	Powder	Thermoplastic	280°-300°	4200*

*Cure cycle only.

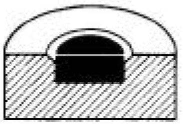
THERMOSETTING MOLDING DEFECTS



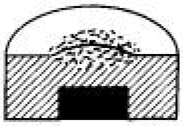
Split (Radial): Section too large for mold area. Sharp corners on specimen. Use larger mold size. Reduce specimen size and eliminate sharp corners if possible.



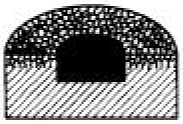
Split (Circumferential): Material has absorbed moisture. Gases released by chemical reaction. Use preheated powders or premolds. Momentarily release pressure during flow stage.



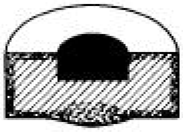
Shrinkage (Edge): Poor adhesion to sample surface with excessive shrinkage at interface. Use lower molding temperature.



Burst (Front Surface): Insufficient pressure and/or insufficient cure time. Adjust molding pressure. Increase cure time.



Woody (Unfused): Insufficient pressure and/or insufficient cure time. Curing of powder particles prior to flow stage. Adjust pressure and/or cure time. Rapidly seal mold closure and apply pressure to eliminate localized curing.



Case Hardening & Blister: Excessive mold temperature. Decrease mold temperature. Momentarily release pressure during flow stage.

General:

If mold temperature is too high, the following difficulties may also be encountered:

(1) Mounts sticking to mold surface regardless of finish or application of release agent.

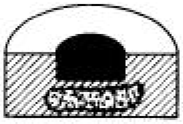
(2) Dull surfaces on mounts.

(3) Case hardening of outer mount surfaces.

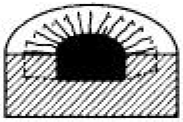
(4) Excessive flash.

(5) Mold staining..

THERMOPLASTIC MOLDING DEFECTS



Cottonball: Center portion of medium did not reach maximum temperature prior to cure stage. Increase holding time at maximum temperature.



Crazing: Inherent stresses relieved upon or after ejection of mount. Cool to a lower temperature prior to ejection. Decrease pressure during cure stage. Stress relieve mounts in boiling water.

COLD MOUNTING

Cold mounting techniques offer particular advantages when a specimen may be too delicate to withstand the pressures and heat involved in compression molding. Large groups of samples may also be readily mounted when work flow can be properly scheduled. However, the time necessary to process small groups of samples far exceeds that for compression mounting.

The three most common types of materials are: (1) Epoxides, (2) Polyesters, and (3) Acrylics. These systems are all two component types consisting of a resin and a hardener. Since an exothermic reaction during polymerization is involved, the mixing by volume or weight ratios of each system is critical. The epoxides are pale yellow and transparent. The polyesters are also transparent and available in water clear or a slight pink hue. The acrylics are opaque.

The characteristics of the common family types are compared in Figure 17.

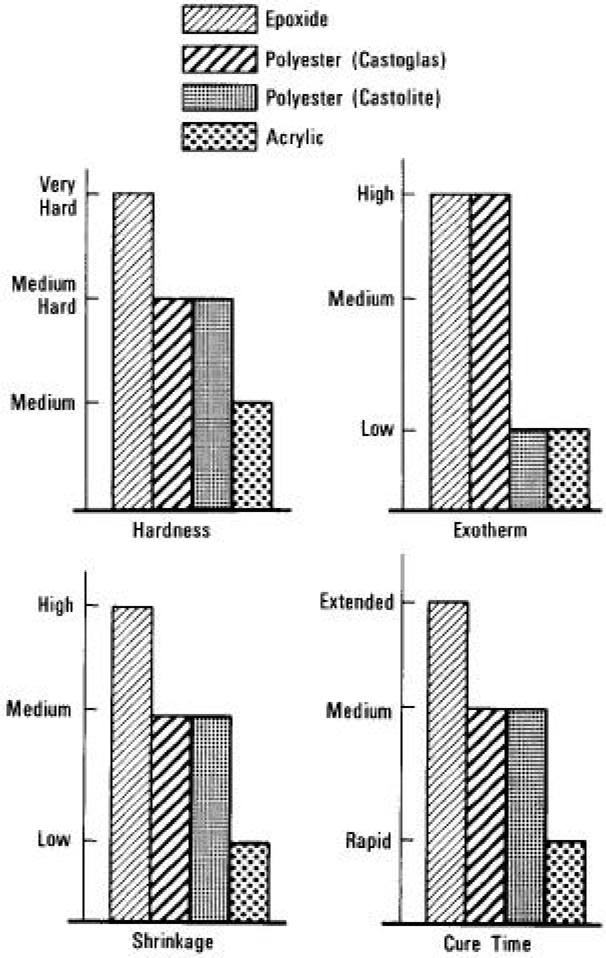
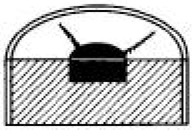
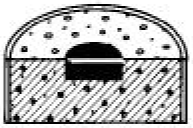


Fig. 17

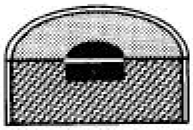
EPOXIDE MOLDING DEFECTS



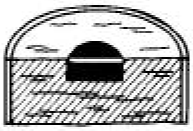
Cracking: Resin to hardener ratio incorrect. Exotherm too extreme. Correct resin to hardener ratio. Use forced cool air to control rate of exotherm.



Entrapped Air: Too violent agitation while blending resin and hardener mixture. Blend mixture more gently or remove air with vacuum.

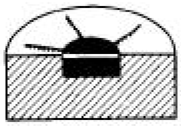


Discoloring: Hardener has oxidized. Resin to hardener ratio incorrect. Keep containers tightly sealed. Correct resin to hardener ratio.

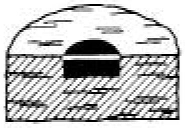


Soft Mounts: Resin to hardener ratio incorrect. Incomplete blending of resin and hardener mixture. Correct resin to hardener ratio. Completely blend mixture.

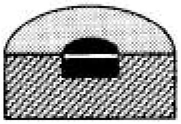
POLYESTER MOLDING DEFECTS



Cracking: Resin to hardener ratio incorrect. Exotherm too extreme. Correct resin to hardener ratio.

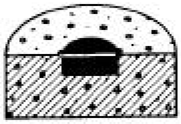


Discoloration: Resin has oxidized. Resin to hardener ratio incorrect. Keep container tightly sealed. Correct resin to hardener ratio.



Soft Mounts: Resin to hardener ratio incorrect. Incomplete blending of resin and hardener mixture. Correct resin to hardener ratio. Completely blend mixture.

ACRYLIC MOLDING DEFECTS



Entrapped Air: Too violent agitation while blending resin and hardener mixture. Blend mixture more gently or remove air with vacuum.

VACUUM IMPREGNATION

Many materials, both organic and inorganic, may be porous, friable, poorly consolidated, have hard and/or soft phase relationships or other extremes.

Vacuum impregnation with a suitable liquid epoxy system will produce a sample which is non-porous with excellent consolidation and rigidity. Penetration is generally sufficient for sectioning or resectioning.

The resultant high density permits preparation without plucking, tearing, fracturing or introducing other forms of sub-surface damage.

Suggested equipment is shown in Figure 18 for using Bakelite® ring forms of standardized mount diameters. Aluminum or tin coated forms may be used for larger sections.

PROCEDURE

Prior to impregnation, samples should be thoroughly cleaned and if necessary, oven dried.

Applying a thin film of release agent to the epoxy contacting surfaces of the specimen forms will be helpful in removing finished mounts from same. Do not coat inside diameter of Bakelite® ring forms as adhesion of epoxy is highly desirable and necessary.

The sample is placed in the ring form and the resin-hardener mixture poured to a level slightly below the top surface. With bell jar in place the system is evacuated to 22 inches of mercury for at least ten minutes total holding time. Active bubbling will occur as air is removed from both epoxy and sample. Intermittent release and reactivation of vacuum will indicate when all air has been removed. Releasing the vacuum will force the epoxy into any continuous void areas. Evacuation below 22 inches of mercury may produce vaporization in an epoxy system due to exceeding the boiling point of the mixture.

CURING

The ratio of epoxy resin to hardener is extremely critical to promote proper curing as this is dependent upon the necessary exothermic reaction for proper

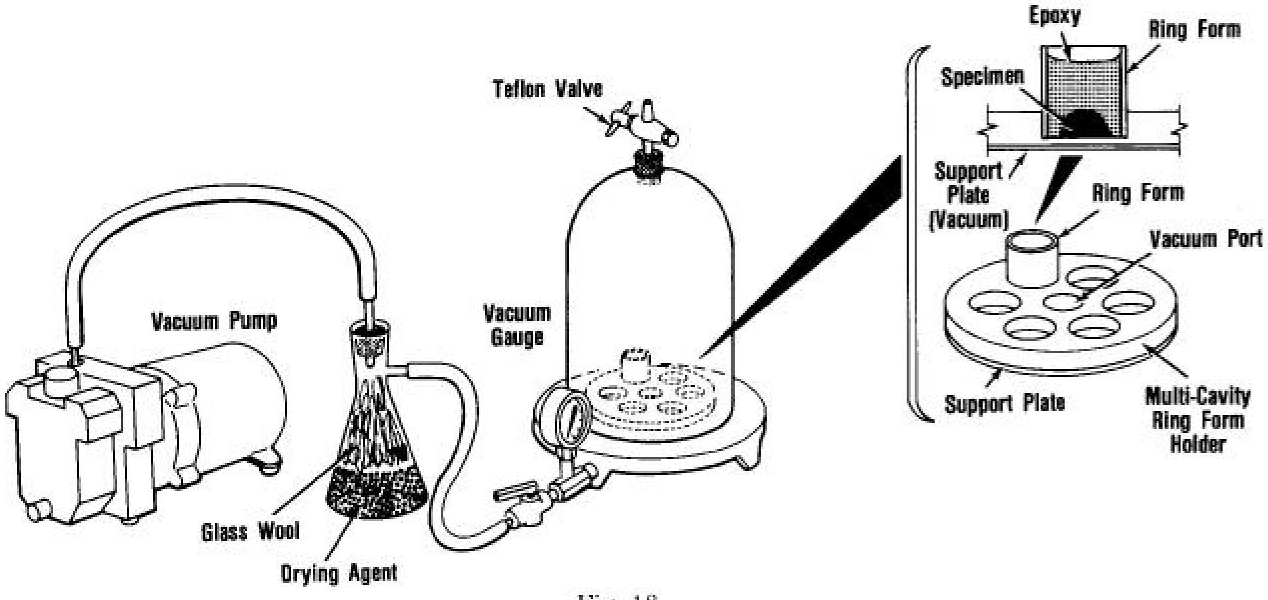


Fig. 18

polymerization. The supplier's recommendation should be strictly followed and never varied.

The choice of system is closely related to the volume of material being cast. Mounts for generalized metallographic sample preparation have dimensions of 1.0, 1.25, 1.50 inches in diameter and are ½ to 3/4 inches high. With these dimensions, a low exotherm system with an air cure can be successfully used.

With larger sections higher polymerization exothermic reactions are involved to promote proper curing. A controlled curing cycle which may be programmed with an automatic timer is shown in Figure 19.

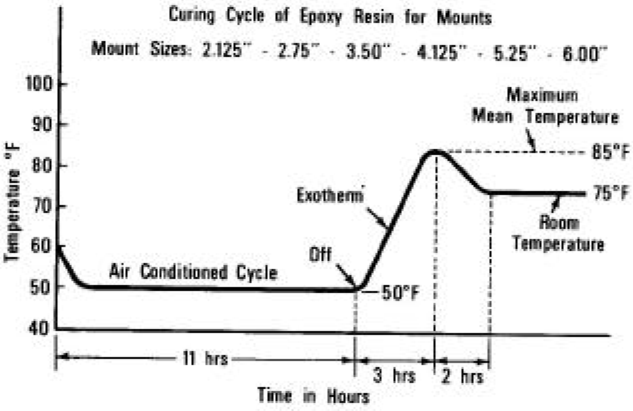


Fig. 19

The cast samples are placed in front of a small air conditioner. The reduction in temperature retards the surface exothermic reaction, preventing shrinkage and stress formation.

A post cure of 1½ to 2 hours at 150° F. will fully develop the physical properties of epoxy. This is applicable to either air or force-cure mounts.

EDGE PROTECTION

Specimen surfaces must be flat to the very edges for microscopic observation and proper photomicrography. Unless special techniques are used prior to mounting or in mounting media selection edge, rounding will occur at the sample-mount interface, see Figure 20.

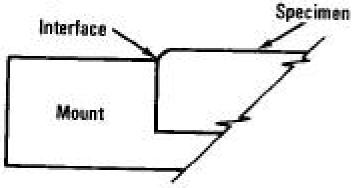
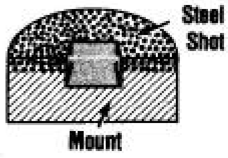
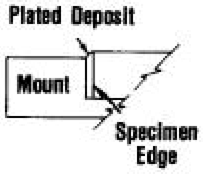


Fig. 20

The degree of rounding is dependent upon the hardness and abrasion differential between the specimen and the mounting material.

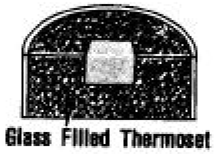


Steel Shot: This technique serves the same purpose as the following suggestion.

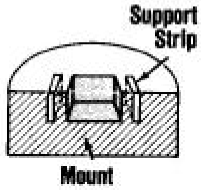


Electro or Electroless Deposition: The section is plated with a coating sufficiently thick to compensate for edge rounding during preparation. The more common materials are Ni and Cu. Nickel will sometimes peel away from a surface due to its stressed condition. Copper is generally used for post-plating electronic gear to preserve edges on single or multi-coated depositions.

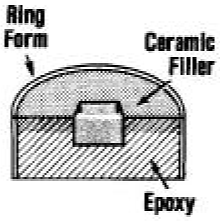
Commercially available electroless coatings are generally stress free. Selection of plating material should be given prior thought regarding etchant rate and reaction with the different metals involved.



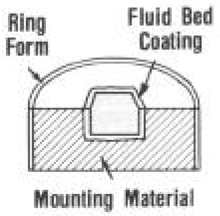
Glass Filled Mounting Media: Thermosetting mounting media with special filler additives will often offer sufficient edge support to prevent rounding. Straight mineral filled epoxies are also helpful if allowed to post cure at room temperature to increase the hardness level.



Support Strips: Small support strips of a similar hardness material are spaced to permit flow of the mounting material between section and spacer. Linen bonded micarta is also excellent as support strips. Configurations, to encircle the entire section, are rather easy to fabricate from this material. Screening and using the "fines" from compression mounting material will increase flow and improve hardness and density of the finished mount.



Ceramic Fillers: Particles of a fine mesh ceramic material (pelletized alumina) are mixed with a liquid epoxy. The pelletized material is available in several mesh sizes and hardness. Selection can be somewhat adjusted to match sample hardness. A small amount of ceramic material is mixed with the resin-hardener mixture. Due to density differences, the filler will settle and form a layer along the bottom surface.



Fluid Bed Coating: A process wherein a fine epoxy powder mixed with a ceramic filler (pelletized alumina) is used to coat a sample. The coating will adhere to practically all surfaces and there is no necessity to electroclean or etch for adherence. Suggested equipment is shown in Figure 21.

The sample is oven pre-heated to 300° F. for 20 minutes. Next, it is quickly immersed in a fluid bed apparatus which contains the epoxy-filler mixture. As the system is aerated the mixture will deposit a coating as long as the specimen surface temperature remains at 300° F. Replacement in the oven at the 300° F. temperature level will bake and harden the coating. Standard compression mounting procedures of 285° F. do not effect the coating.

If it is desirable to coat a material which will not maintain a 300° F. surface temperature, a dip or spray with high temperature aluminum paint will permit fluid bed coating.

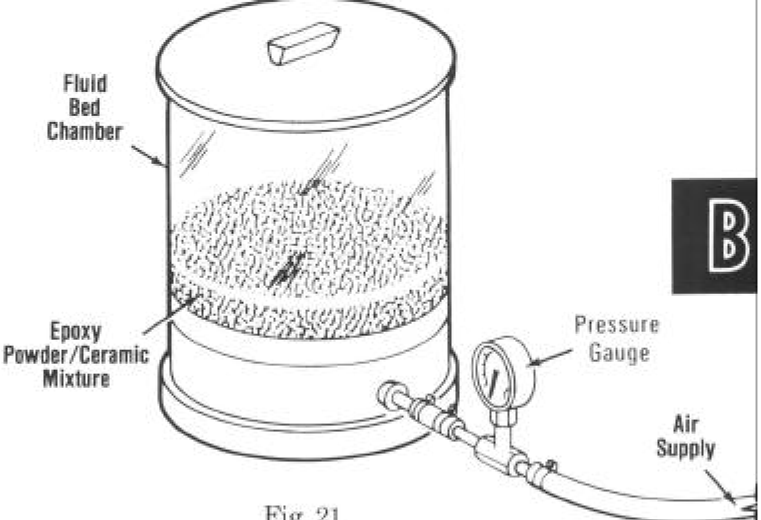
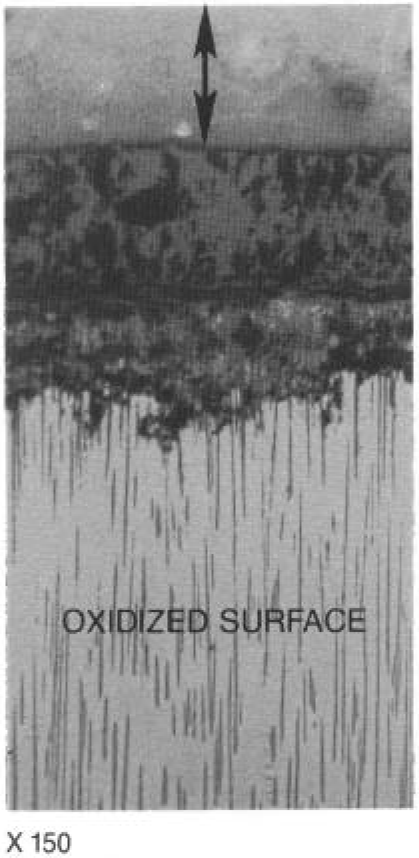


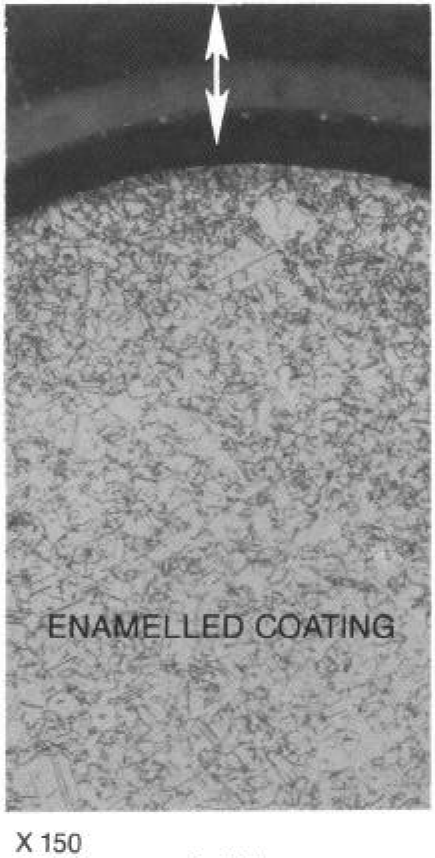
Fig. 21

FLUID BED COATING



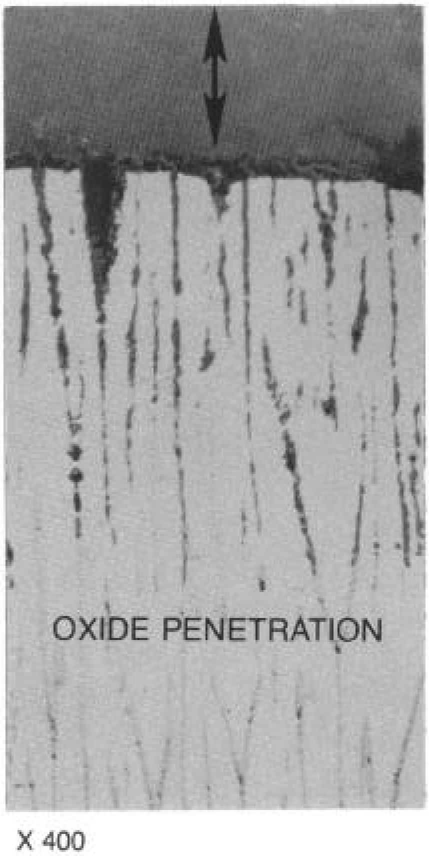
X 150

Co-Ta-Carbide



X 150

Cu Wire



X 400

Co-Ta-Carbide

Manual or Semi-Automatic Polishing Procedure

I. POLISHING PROCEDURES FOR FERROUS MATERIALS

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
SiC 320 grit	Paper or Disc (waterproof)	Water
SiC 400 grit	Paper or Disc (waterproof)	Water
SiC 600 grit	Paper or Disc (waterproof)	Water

REMARKS:

(a) Al_2O_3-Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.

(b) Emery-Coated Products — Abrasive sequence would be 1/0, 2/0, 3/0, 4/0. These materials are for dry operation. Material removal rates are much less than either SiC or Al_2O_3.

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 6 (μm)	Nylon Cloth	Lapping Oil

FINAL-POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Gamma Alumina 0.05 (μm)	Lecloth® (flocked cotton sateen)	Distilled or Deionized Water

SUGGESTED DEVIATIONS:

GRAPHITIC CAST IRONS
(Courtesy J.C. Moore, LECO CORPORATION.)

FINE GRINDING:

(a) 240, 320, 400, 600 grit SicC or Al_2O_3 paper or disc with water as lubricant. Clean thoroughly and dry.

(b) Etch rather heavily.Use 4% Picric Acid Solution for pearlitic matrix materials and 4% Nital Solution for ferritic matrix materials.

(c) Abrade on 4/0 Emery Coated paper dry. After a short period of abrasion clean abrasive surface with cotton swab saturated with alcohol. Repeat etching, abrasion and cleaning until the graphite flakes, nodules or temper carbon show definite retention and uniform matrix finish. Clean samples thoroughly.

ROUGH POLISHING: Etch sample lightly as recommended above. Precondition synthetic velvet cloth with one micron diamond paste. A water soluble extender is recommended as oils may penetrate and stain graphite particles. Carefully observe condition of graphite. Use alternate etch and repolish if necessary. Clean sample thoroughly.

FINAL POLISHING: Etch sample lightly. Precondition synthetic velvet cloth with 0.25 micron diamond paste, use water soluble extender. Repeat alternate etch and repolish as necessary. Polarized light will clearly reveal the condition of the graphite as it is anisotropic. Staining can occur during etching as graphite can absorb Nital.

GALVANIZED COATINGS

(a) Water should not be used as a lubricant at any stage due to staining effect or corrosion effect of the coating. Kerosene or lapping oil are good alternates.

(b) Rough Polishing: Silk cloth as lap covering, 0.3 micron Alpha Alumina as abrasive and filtered kerosene as lubricant.

(c) Final Polishing: Synthetic Velvet as lap covering, 0.05 micron Gamma Alumina as abrasive and a mixture of alcohol and glycerine as lubricant.

STAINLESS STEELS, STAINLESS STEEL CASTING ALLOYS, HEAT RESISTING ALLOYS

(a) Rough Polishing: Superior results can often be obtained by sequencing through 9 micron and 3 micron diamond paste with nylon cloth as lap covering and lapping oil as lubricant.

(b) Final Polishing: Sequencing through 0.3 micron Alpha Alumina and 0.05 Gamma Alumina, Lecloth® as lap covering and distilled or deionized water as lubricant.

(c) Check the possibilities of Electropolishing, particularly with solid solution alloys and transformed structures.

(d) Check the possibilities of Slurry (Etch-Attack) Polishing particularly with wrought heat resisting alloys.

II. POLISHING PROCEDURES FOR COPPER BASED MATERIALS

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
SiC 320 grit	Paper or Disc (waterproof)	Water
SiC 400 grit	Paper or Disc (waterproof)	Water
SiC 600 grit	Paper or Disc (waterproof)	Water

REMARKS:

(a) Al_2O_3-Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.

(b) Emery-Coated Products. Abrasive sequence would be 1/0, 2/0, 3/0, 4/0. These materials are for dry operation. Material removal rates are much less than either SiC or Al_2O_3.

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 6 (μm)	Nylon Cloth	Lapping Oil

FINAL POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Gamma Alumina 0.05 (μm)	Lecloth® (flocked cotton sateen)	Distilled or Deionized Water

SUGGESTED DEVIATIONS

(a) Check the possibilities of Electropolishing particularly with microstructure amenable to same.

(b) Electropolishing may also be used to distinct advantages with many materials which may have been processed through the Rough and Final Polishing Stages by Mechanical Methods. Response to extremely short cycles is many times advantageous even with those structures containing finely dispersed intermetallic compounds and complex phase relationships.

(c) Check the possibilities of Slurry (Etch-Attack) Polishing as a means to remove deformation or to process multi-material sections.

III. POLISHING PROCEDURES FOR ALUMINUM AND MAGNESIUM BASED MATERIALS

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
SiC 320 grit	Paper or Disc (waterproof)	Water
SiC 400 grit	Paper or Disc (waterproof)	Water
SiC 600 grit	Paper or Disc (waterproof)	Water

REMARKS:

(a) Al_2O_3-Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.

(b) Emery-Coated Products. Abrasive sequence would be 1/0, 2/0, 3/0, 4/0. These materials are for dry operation, however, kerosene may be used for lubricant with resultant decreased paper life. Material removal rates are much less than either SiC or Al_2O_3.

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 6 (μm)	Nylon Cloth	Lapping Oil

FINAL POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Magnesium Oxide 2.0 (μm)	Lecloth® (flocked cotton sateen)	Distilled or Deionized Water

REMARKS:

(a) Aluminum polishing wheels are recommended to eliminate electrochemical reaction between the sample and wheel. A thin insulating plastic material or aluminum foil between the bronze wheel and wheel covering would accomplish the same.

(b) The conditioning of wheel covering differs from standard procedures. The entire surface should be premoistened with distilled or deionized water. The MgO powder is dispensed in the center of the wheel, moistened and worked into a heavy, creamy consistency.

(c) The sample is skidded over the surface and the abrasive is moved outward. The sample edges are slightly beveled to aid hand manipulation.

(d) Light pressure must be used as many optical identifications of intermetallic compounds are dependent on standardized oxide film colors. These colors are not reproducible under heavy pressure.

IV. POLISHING PROCEDURES FOR TITANIUM, ZIRCONIUM, HAFNIUM AND ALLOYS

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
SiC 320 grit	Paper or Disc (waterproof)	Water
SiC 400 grit	Paper or Disc (waterproof)	Water
SiC 600 grit	Paper or Disc (waterproof)	Water

REMARKS:

(a) Al_2O_3-Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.

(b) Emery-Coated Products. Abrasive sequence would be 1/0, 2/0, 3/0, 4/0. Material removal rates are much less than either SiC or Al_2O_3.

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 9 (μm)	Nylon Cloth	Lapping Oil

FINAL POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Alpha Alumina 0.3 (μm)	Lecloth® (flocked cotton sateen)	Distilled or Deionized Water

REMARKS:

(a) This family of materials is extremely susceptible to surface deformation.

The following etchant also functions as a Chemical Polish.

COMPOSITION:

60 cc Glycerine

20 cc Nitric Acid

20 cc Hydrofluoric Acid (48%)

TECHNIQUE:

Swab vigorously with saturated cotton. Reaction is very active at outset, but diminishes as deformation is removed. Staining effects on various phases are time dependent. Reaction rate may be varied by heating (increase) or chilling (decrease) the sample or etchant.

CAUTION:

Etchant must be fresh each time, stability decreases in a few hours. Protect hands.

SUGGESTED DEVIATIONS

(a) Check the possibilities of Electropolishing, Slurry (Etch-Attack) Polishing or Chemical Polishing.

V. POLISHING PROCEDURES FOR CEMENTED CARBIDES

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond 45 (μm) (280 mesh)	Resin-Bonded Diamond Disc	Water
Diamond 15 (μm)	Resin-Bonded Diamond Disc	Water

REMARKS:

For gross surface removal, employ a 63-74 μm(220 Mesh) Resin–Bonded Diamond Disc to decrease wear on 45 (μm) (280 Mesh) Disc.

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 6 (μm)	Pan K (PVC Chemotextile)	Lapping Oil
Diamond Paste 3 (μm)	Pan K (PVC Chemotextile)	Lapping Oil

REMARKS:

Very often the 3 micron Diamond Paste step may be omitted.

FINAL POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 0.1 (μm)	Pan K (PVC Chemotextile)	Lapping Oil

REMARKS:

If extremely fine scratches are visible in the binder material, a short cycle on Lecloth® with Gamma Alumina (0.05 Micron) will remove same.

VI. POLISHING PROCEDURES FOR LEAD ALLOYS, TIN ALLOYS AND ZINC BASED DIE CASTINGS

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
SiC 320 grit	Paper or Disc (waterproof)	Water
SiC 400 grit	Paper or Disc (waterproof)	Water
SiC 600 grit	Paper or Disc (waterproof)	Water

REMARKS:

(a) Al_2O_3 Coated Products (waterproof) — Same abrasive sequence and lubricant may be used. Abrasive action is less severe.

(b) Emery Coated Products — Abrasive sequence would be 1/0, 2/0, 3/0. These materials are for dry operation, however kerosene may be used for lubricant with resultant decreased paper life. Material removal rates are much less than either SiC or Al_2O_3.

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 6 Micron	Nylon Cloth	Lapping Oil

REMARKS:

There may be some advantage to extend Rough Polishing into two steps by incorporating a 0.5 Micron Diamond with Lecloth® step. If Lapping Oil should attack any micronconstituents alcohol or ethylene-glycol may be substituted.

FINAL POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Gamma Alumina 0.05 Micron	Lecloth® (flocked cotton sateen)	Distilled or Deionized water

REMARKS:

(a) The lead alloys lend themselves to mechanical preparation rather than electrolytic polishing since many lead alloys undergo Eutectic formation during solidification. Very often Eutectic structures will show supercooling tendencies and instability in the solid solution zones.

(b) Both tin and lead alloys are inherently soft and very susceptible to gross surface flow and accompanying deformation during preparation. Careful etching and repolishing will remove the disturbed metal.

(c) One should be careful to observe the melting point of the material being prepared and select mounting methods accordingly.

VII. POLISHING PROCEDURES FOR REFRACTORY ALLOYS AND METALS (Nb, Mo, W, V, Ta)

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
SiC 320 grit	Paper or Disc (waterproof)	Water
SiC 400 grit	Paper or Disc (waterproof)	Water
SiC 600 grit	Paper or Disc (waterproof)	Water

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 6 (μm)	Nylon Cloth	Lapping Oil

REMARKS:

(a) Extreme caution should be exercised in all preceding stages to avoid or, at least, certainly minimize surface deformation.

(b) In the early portion of Rough Polishing more scratches seem to appear than are being removed. The scratches from Fine Grinding are being "opened-up." Extending the polishing time will remove these effects.

FINAL POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Gamma Alumina 0.05 (μm)	Lecloth® (flocked cotton sateen)	Distilled or Deionized water

SUGGESTED DEVIATIONS:

(a) Check the possibility of Electropolishing.

(b) Check the possibility of Slurry (Etch-Attack) Polishing.

VIII. POLISHING PROCEDURES FOR PLATED SECTIONS

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
SiC 320 grit	Paper or Disc (waterproof)	Water
SiC 400 grit	Paper or Disc (waterproof)	Water
SiC 600 grit	Paper or Disc (waterproof)	Water

REMARKS:

(a) Al_2O_3-Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action may be less severe.

(b) Emery-Coated Products. Abrasive sequence would be 1/0, 2/0, 3/0, 4/0. Material removal rates are much less than either SiC or Al_2O_3.

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 6 (μm)	Nylon Cloth	Lapping Oil

FINAL POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Gamma Alumina 0.05 (μm)	Lecloth® (flocked cotton sateen)	Distilled or Deionezed water

REMARKS:

(a) During preparation softer electrodeposits may tend to flow and the interfaces between the various layers will not be clearly delineated.

(b) Edge Protection — Suggestions for Post Plating and Fluid Bedding are described under Mounting Procedures.

(c) Etching — The interfaces can be clearly delineated by etching. Specific details are given in Table 4 under Microetching.

IX. POLISHING PROCEDURES FOR POWDER METALS AND ALLOYS

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
SiC 320 grit	Paper or Disc (waterproof)	Water
SiC 400 grit	Paper or Disc (waterproof)	Water
SiC 600 grit	Paper or Disc (waterproof)	Water

REMARKS:

(a) Al_2O_3–Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.

(b) Emery-Coated Products. Abrasive sequence would be 1/0, 2/0, 3/0, 4/0. These materials are for dry operation only. Material removal rates are much less than either SiC or Al_2O_3.

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 6 (μm)	Nylon Cloth	Lapping Oil

FINAL POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Gamma Alumina 0.05 (μm)	Lecloth® (flocked cotton sateen)	Distilled or Deionized water

GENERAL:

(a) Cleaning: Porosity is generally associated with powder metal sections. The porous area can become filled with foreign products during sectioning and preparation.

The samples should be very thoroughly cleaned in either an Extractor-Condenser Apparatus or several stages in an Ultrasonic Cleaner.

(b) Impregnation: Porous sections should be either impregnated with a high temperature wax (350° F.) or vacuum impregnated with epoxy. Such practice will prevent contamination during preparation and also improve consolidation for mounting and preparation.

Procedures for both Cleaning and Impregnation are detailed under Mounting Procedures.

X. POLISHING PROCEDURES FOR CERAMICS

FINE GRINDING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond 45 (μm) (280 Mesh)	Resin-Bonded Diamond Disc	Water
Diamond 15 (μm)	Resin-Bonded Diamond Disc	Water

REMARKS: For gross surface removal, employ a 63-74 micron (220 Mesh) Resin ,Bonded Diamond Disc to decrease wear on 45 micron(280 Mesh) Disc.

ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 9 (μm)	Pan K (PVC Chemotextile)	Lapping Oil
Diamond Paste 6 (μm)	Pan K (PVC Chemotextile)	Lapping Oil

REMARKS:

With some materials the 9 micron Diamond Paste Step may be omitted. If any microconstituents or the mounting media are stained or attacked by an oil extender, ethylene-glycol or alcohol may be used as a lubricant.

FINAL POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 3 (μm)	Pan K (PVC Chemotextile)	Lapping Oil
Alpha Alumina 0.3 (μm)	Silk	Distilled Deionized Water

REMARKS: Polishing times should be as short as possible to avoid relief polishing.

Microetching

Metal Progress Data Book

MICROETCHING

A metallographic specimen in the "as polished and unetched" state will reveal inclusions, porosity, cracks, intergranular corrosion, surface conditions, etc.

Etching is defined as the process to reveal structural details by preferential attack of a metal surface with an acid or basic chemical solution.

The most commonly used etching technique is termed Solution Etching. This may be further classified into two categories:

A. Acid and Basic Reagents
  1. Immersion
  2. Swabbing

B. Electrolytic
  1. Direct Current
  2. Alternating Current

For Immersion Etching, the sample is held with tongs and immersed in a suitable etching solution. The specimen is gently agitated to eliminate adherent air bubbles and continually supply fresh reagent to the surface. Swab etching implies the surface is gently wiped with a soft cotton swab saturated with etchant. The swab should be replenished with fresh reagent if etching times are comparatively long.

With Electrolytic Etching, direct current electrolysis is usually used. The specimen is made the anode and a suitable insoluble material is the cathode. For a few materials (platinum, palladium and their alloys) alternating current electrolysis is used.

STANDARD METHOD FOR MICROETCHING METALS & ALLOYS[2]

1. Scope

1.1 These methods cover chemical solutions and procedures to be used in etching metals and alloys for microscopical examination. Safety precautions and miscellaneous information are also included.

2. Summary of Methods

2.1 Table 1 is an alphabetical listing of the metals (including rare earths) and their alloys for which etching information is available. For each metal and alloy, one or more etchant numbers and their corresponding use is indicated. Alloys are listed as a group or series when one or more etchants are common to the group or series. Specific alloys are listed only when necessary. When more than one etchant number is given for a particular use, they are usually given in order of preference. The numbers of electrolytic etchants are underlined to differentiate them from nonelectrolytic etchants.

2.2 Table 2 is a numerical listing of all the etchants referenced in Table 1 and includes the composition and general procedure to be followed for each etchant.

2.3 To use the tables, look up the metal or alloy of interest in Table 1 and note the etchant numbers corresponding to the results desired. The etchant composition and procedure is then located in Table 2 corresponding to the etchant number.

2.4 If the common name of an etchant is known (Marble's, Vilella's, etc.), and it is desired to know the composition, Table 3 contains an alphabetical listing of etchant names, each coded with a number corresponding to the same etchant given in Table 2.

3. Safety Precautions

3.1 Special safety precautions are mentioned in Table 2 only for extremely hazardous mixtures such as the cyanides and those that can form nitrogen dioxide gas. However, ALL CHEMICALS ARE POTENTIALLY DANGEROUS and it is assumed that the person using any of the etchants is thoroughly familiar with all of the chemicals involved and the proper procedure for handling and mixing these chemicals.

3.2 Some basic suggestions for the handling of etching chemicals are as follows:

3.2.1 When pouring, mixing, or etching, always use the proper protective garb (glasses, gloves, apron, etc.).

3.2.2 Use proper devices (glass or plastic) for weighing, mixing, containing, and storage of solutions.

3.2.3 Wipe or flush any and all spills, no matter how minute in nature.

3.2.4 Dispose of any and all solutions that are not properly identified by composition and concentration (when in doubt, throw it out).

3.2.5 Store and handle chemicals, according to the manufacturer's recommendations. Observe printed cautions on reagent bottles.

3.2.6 If not sure about a chemical or its proper use, contact your Chemical or Safety Department.

---

[2] American Society for Testing & Materials. Designation: E 407-70 (Reprinted with Permission)

3.2.7 Have available and use quick references as to toxicity and working precautions of various chemicals. Some of the many excellent references are (1) "Handbook of Dangerous Materials" by N. Irving Sax, (2) "The Chemistry of Industrial Toxicology" by H. B. Elkins, and (3) "Safety in the Metallography Laboratory" by R. L. Anderson, Scientific Paper 65-1P30-METLL-P2, Westinghouse Research Lab., Pittsburgh, PA.

4. Miscellaneous information

4.1 If you know the trade name of an alloy and need to know the composition to facilitate the use of Table 1, refer to "Engineering Alloys" by Norman E. Woldman (Reinhold Publishing Corp.).

4.2 Chemicals used should meet USP and NF specifications or better.

4.3 When mixing etchants, always add reagents to the solvent unless specific instructions indicate otherwise.

4.4 Where water is given as the solvent, distilled water is preferred because of the great variance of the purity of tap water.

4.5 Methanol is usually available only as absolute methanol. When using this alcohol it is imperative that approximately 5 volume percent of water is added whenever an etchant composition calls for 95 percent methanol. Most of these etchants will not work at all if water is not present.

4.6 For conversion of small liquid measurements, there are approximately 20 drops/ml.

4.7 Etching should be carried out on a freshly polished specimen.

4.8 Gentle agitation of the specimen or solution during etching will result in a more uniform etch.

4.9 The etching times given are only suggested starting ranges and not absolute limits.

4.10 In electrolytic etching, d-c current is implied unless indicated otherwise.

4.11 A good economical source of d-c current for small-scale electrolytic etching is the standard 6-V lantern battery.

4.12 In electrolytic etching, the specimen is the anode unless indicated otherwise.

4.13 Do not overlook the possibility of multiple etching, that is, etching with more than one solution in order to fully develop the structure of the specimen.

4.14 Microscope objectives can be ruined by exposure to hydrofluoric acid fumes from etchant residue inadvertently left on the specimen. This problem is very common when the specimen or mounting media contain porosity and when the mounting material (such as bakelite) does not bond tightly to the specimen resulting in seepage along the edges of the sample. In all cases, extreme care should be taken to remove all traces of the etchant by thorough washing and *complete drying* of the specimen before placing it on a microscope stage.

TABLE I —
ETCHANTS FOR METALS & ALLOYS*

NOTE — Electrolytic etchants are underlined

Metal	Etchants	Uses
Aluminum base:		
Pure Al	1a, 2, 3	general structure
	4, 5	grain structure under polarized light
	1b	grain boundaries and slip lines
1000 series	1a, 3, 2	general structure
	4, 5	grain structure under polarized light
	6, 7	phase identifications
2000 series	3, 2, 1a	general structure
	8a, 6, 7	phase identifications
3000 series	3, 1a	general structure
	4, 5	grain structure under polarized light
	8a, 6, 7	phase identifications
4000 series	3, 1a	general structure
5000 series	3, 1a, 2, 6, 8a	general structure
	4, 5	grain structure under polarized light
6000 series	3, 1a, 2, 6, 8a	general structure
	4, 5	grain structure under polarized light
	1a, 2, 7, 6, 8a	phase identifications
7000 series	3, 1a, 2	general structure
	4, 5	grain structure under polarized light
	3b, 6	phase identifications

---

* Etchants are listed in order of preference when more than one is given for a specific use.
Table II gives composition and procedure for individual etchants

TABLE I — continued

Metal	Etchants	Uses
Beryllium base:		
Pure Be	9, 10	general structure via polarized light
Be alloys	11	general structure
Chromium base:	12, 13c	general structure
Cobalt base:		
Pure Co	14, 15, 16, 17,	general structure
Hard facing and tool metals	18, 19, 20	general structure
High-temperature alloys	20, 18, 16, 21, 22b, 24, 25	general structure
	19	phase identification
Columbium base (see niobium base)		
Copper base:		
Pure Cu	26, 27, 28, 29, 30, 31d, 32, 33 34b, 35, 36, 37, 38, 39, 40, 41, 42, 8b	general structure
	43, 28	chemical polish and etch
Cu-Al (Aluminum bronze)	44, 31d, 34b, 35, 36, 37, 38, 39, 40, 45, 28	general structure
Cu-Be	46, 41, 45	general structure
Cu-Cr	41	general structure
Cu-Mn	41	general structure
Cu-Ni	34, 47, 48, 40, 49, 50	general structure
Cu-Si	41	general structure
Cu-Sn (tin bronze)	51, 52	general structure
Admiralty metal	8b	general structure
Gilding metal		
Cartridge brass		
Free-cutting brass		
Nickel silver	31d, 32, 33, 41, 42, 49	general structure
Cu alloys	26, 27, 28, 29, 30, 44, 41, 31d, 32, 33, 34b, 35, 36, 37, 38, 39	general structure
	53, 43, 28, 49	chemical polish and etch
	42, 49	darkens beta in alpha-beta brass
	54	etching of cold worked brass
Dysprosium base:	55, 56	general structure
Erbium base:	55, 56	general structure
Gadolinium base:	55, 56, 57	general structure
Germanium base:	58, 59, 60	general structure
Gold base:		
Pure Au	61, 62	general structure
	63	chemical polish and etch
Au alloys	64b, 62	general structure
	63	chemical polish and etch
> 90% noble metals	61	general structure
< 90% noble metals	65	general structure
Hafnium base:	66, 67, 68, 69, 70	general structure
	71	grain structure under polarized light
	72	chemical polish and etch
Holmium base:	55, 56	general structure
Iridium base:	73c	general structure
Iron base:		
Pure Fe	74a	grain boundaries
	75	substructure
Fe + C	76, 74a, 77, 78, 79	general structure
and	74a, 77, 31a	ferrite grain boundaries
Fe + < 1C + < 4% additions	80, 81, 82	prior austenite grain boundaries in martensitic and bainitic steels
	78	untempered martensite
	31b, 78	carbides and phosphides (matrix darkened, carbides and phosphides remain bright)
	83	cementite attacked rapidly, austenite less, ferrite and iron phosphide least
	84	overheating and burning
	85	stains carbides
	86	chemical polish — etch
Fe + 4-12 Cr	80, 87, 88, 89, 90, 91, 79	general structure
	86	chemical polish — etch

TABLE I — continued

Metal	Etchants	Uses
Fe + 12-30 Cr + < 6 Ni (400 Series)	80, 87, 88, 89, 34, 40, 92, 93, 94, 95, 91	general structure
	96, 97, 98	sigma phase
	31c	carbides
	86	chemical polish — etch
Fe + 12-20 Cr + 4-10 Ni + < 7% other elements (controlled transformation, precipitation hardening, stainless maraging alloys)	80, 31c, 89, 99, 100, 91	general structure
	31c	carbides
	86	chemical polish — etch
Fe + 15-30 Cr + 6-40 Ni + < 5% other elements (300 Series)	13b, 89, 87, 88, 83a, 80, 94, 95, 91, 101	general structure
	13a, 102	carbides and sensitization
and	48, 96, 97, 98	stains sigma phase
Fe + 16-25 Cr + 3-6 Ni + 5-10 Mn (200 Series)	103, 104, 98	delineates sigma phase and ferrite
	103, 104	welds of dissimilar metals
	86	chemical polish — etch
High temperature	89, 25, 105, 106, 97	general structure
	107, 108	γ' precipitate
	86	chemical polish — etch
Nonstainless maraging steels	109, 89, 99, 100	general structure
	83b	grain boundaries
	86	chemical polish — etch
Tool steels	74a, 80, 14	general structure
	110	grain boundaries in tempered tool steel
	86	chemical polish — etch
Superalloys	111	general structure
	111	γ' depletion
Lead base:		
Pure Pb	57, 112	general structure
	113	for alternate polishing and etching
Pb + < 2 Sb	114, 115, 57, 74b	general structure
	113	for alternate polishing and etching
Pb + > 2 Sb	114, 57, 74b	general structure
	113	for alternate polishing and etching
Pb + Ca	112	general structure
	113	for alternate polishing and etching
Pb alloys	116, 117b	general structure
Babbitt	74b	general structure
Magnesium base:		
Pure Mg	118, 119, 74a, 120, 121, 122	general structure
	123	strain-free polish — etch
Mg-Mn	119, 74a, 124, 122	general structure
Mg-Al, Mg-Al-Zn (Al + Zn < 5%)	118, 119, 74a, 125, 124, 123, 122	general structure
	120, 125, 126, 127	phase identification
	124, 126, 127	grain structure
Mg-Al, Mg-Al-Zn (Al + Zn > 5%)	118, 119, 74a, 125, 124, 121, 122	general structure
	120, 125, 126, 127	phase identification
Mg-Zn-Zr and	118, 119, 74a, 1d, 128, 124, 126 127, 121, 122	general structure
Mg-Zn-Th-Zr	120, 121	phase identification
Mg-Th-Zr and	118, 119, 74a, 1d, 124, 127, 121, 122	general structure
Mg-Rare Earth-Zr	120, 121	phase identification
Molybdenum base:	98c, 129, 130, 131	general structure
As cast	132a	chemical polish prior to etching
Nickel base:		
Pure Ni and high Ni alloys	133, 134, 47, 135, 136, 25, 108 31c	general structure
	137	grain boundary sulfidation
Ni-Ag	38, 138, 50, 139	general structure
Ni-Al	50, 140, 141, 142, 89, 143	general structure
Ni-Cr	144, 50, 83, 134, 145, 98, 146, 147, 13a	general structure
Ni-Cu	38, 138, 50, 133, 140, 25, 134, 47, 48b, 94, 108, 34	general structure
Ni-Fe	50, 140, 141, 83, 134, 148, 40, 107, 149	general structure
	74e, 25, 150	orientation pitting

TABLE I — continued

Metal	Etchants	Uses
Ni-Mn	74e	general structure
Ni-Mo	143	general structure
Ni-Ti	143, 151, 50, 133	general structure
Ni-Zn	152	general structure
Superalloys	94, 105, 138, 153, 12	general structure
	25	grain size
	107, 111, 13a	reveals microstructural inhomogeneity
	133	grain boundary sulfidation
	154	fine precipitation structure
	19b, 155, 156	differential matrix and nonmetallic staining
	22a	for passive alloys (for example Inconel 625)
	157	specific for Hastelloy W
	107	submicroscopic structure in aged superalloys particularly for electron microscopy. Stains the matrix when γ' precipitates are present
	154	γ' banding
	18	pre-etch activation for passive samples
Niobium (Columbium) base:	129, 66, 158, 159, 160, 161, 162, 163	general structure
	164, 129, 160	grain boundaries
Osmium base:	165a	general structure
	165a	etch — polishing for viewing grains with polarized light
Palladium base:		
Pure Pd	61, 166, 62, 165a	general structure
Pd alloys	166, 64a, 62, 165a	general structure
> 90% noble metals	61	general structure
< 90% noble metals	65	general structure
Platinum base:		
Pure Pt	64a, 73a	general structure
	167	electrolytic polish and etch
Pt alloys	64b, 73a	general structure
	167	electrolytic polish and etch
> 90% noble metals	61	general structure
< 90% noble metals	65	general structure
Pt-10% Rh	168	general structure
Plutonium base:	169	general structure
Rhenium base:	13b, 98c, 132b, 170a	general structure
Rhodium base:	171	general structure
Ruthenium base:	73b	general structure
	73b	etch — polishing for viewing grains with polarized light
Silver base:		
Pure Ag	172, 173, 62	general structure
Ag alloys	65, 61, 174, 175, 62	general structure
Ag-Cu	30	general structure
Ag-Pd alloys	173	general structure
Ag solders	173, 176	general structure
Tantalum base:		
Pure Ta	177	general structure
Ta alloys	159, 66, 178, 163, 161, 179	general structure
	164	grain boundaries and inclusions
	158	grain boundaries — retains carbide precipitate
Thorium base:		
Pure Th	185	general structure
Th alloys	185	general structure
Tin base:		
Pure Sn	74d, 180, 51	general structure
	181	grain boundaries
Sn-Cd	74d	general structure
Sn-Fe	74d, 117a	general structure
Sn-Pb	182, 183, 74b	general structure
	116	darkens Pb in Sn-Pb eutectic
Sn coatings (on steel)	183	general structure
Babbitts	184	general structure

TABLE I — continued

Metal	Etchants	Uses
Sn-Sb-Cu	74b	general structure
Titanium base:		
Pure Ti	186, 187, 67, 68, 69	general structure
	188	removes stain
	72	chemical polish and etch
Ti-5 Al-2.5 Sn	189	reveals hydrides
Ti-6 Al-6V-2 Sn	190	stains alpha and transformed beta, retained beta remains white
Ti-Al-Zr	191	general structure
Ti-8 Mn	192	general structure
Ti-13V-11Cr-3Al (aged)	192	general structure
Ti-Si	193	general structure
Ti alloys	186, 187, 192, 194, 158, 132b, 1c, 67, 68, 69, 3a	general structure
	11, 1c	reveals alpha case
	72, 192, 178	chemical polish and etch
	170a	outlines and darkens hydride in some alloys
	188	removes stain
Tungsten base:		
Pure W	98c, 131	general structure
As cast	132a	chemical polish prior to etching
W-Th	209	general structure
Uranium base:		
Pure U	67, 69, 195, 196	general structure
U + Zr	68	general structure
U beryllides	170a	general structure
U alloys	67, 69, 195, 196	general structure
	207	carbides
Vandaium base:		
Pure V	170b, 165b	general structure
	197, 198	grain boundaries
V alloys	199, 198	general structure
Zinc base:		
Pure Zn	200a	general structure
Zn-Co	177	general structure
Zn-Cu	201	general structure
	203	distinguishes gamma and epsilon
Zn-Fe	74a	structure of galvanized sheet
Die castings	202	general structure
Zirconium base:	66, 67, 204, 68, 69, 205	general structure
	206	electrolytic polish and etch
	71	grain structure under polarized light
	72	chemical polish and etch

TABLE 2
NUMERICAL LIST OF ETCHANTS

Etchant	Composition	Procedure
1	1 ml HF 200 ml water	*(a)* Swab with cotton for 15 seconds. *(b)* Alternately immerse and polish several minutes. *(c)* Immerse 3 to 5 seconds. *(d)* Immerse 10 to 120 seconds.
2	1 g NaOH 100 ml water	*(a)* Swab 10 seconds to reveal general structure. *(b)* Immerse 15 min. wash 10 min. in water to form film with hatching which varies with grain orientation.
3	2 ml HF 3 ml HCl 5 ml HNO_3 190 ml water	*(a)* Immerse 10 to 20 sec. Wash in stream of warm water. Reveals general structure. *(b)* Dilute with 4 parts water — colors constituents — *Mix fresh.*
4	25 ml H_3PO_4 50 ml Carbitol (diethylene glycol monoethyl ether) 4 g boric acid 2 g oxalic acid 10 ml HF 32 ml water	Electrolytic: Use carbon cathode raising d-c voltage from 0 to 30 V in 30 sec. Total etching time 3 min. with agitation. Wash and cool. Repeat if necessary.