A black and white micrograph showing a complex metal microstructure. The image features a dense network of fine, needle-like or lath-like structures, likely martensite, which are oriented in various directions. There are also larger, more irregular regions that appear to be different phases or grain boundaries. The overall texture is highly textured and intricate.

**METALLOGRAPHY  
PRINCIPLES AND  
PROCEDURES**

**LECO** 

# TABLE OF CONTENTS

**Metallographic Sample Preparation**

**A**

**Mounting Procedures**

**B**

**Manual or Semiautomatic  
Polishing Procedures**

**C**

**Electrolytic Polishing Procedures**

**D**

**Slurry (Etch-Attack) Polishing Procedures  
Chemical Polishing Procedures**

**E**

**Microetching**

**F**

**Macroetching**

**G**



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<b>Metallographic Sample Preparation</b>	<b>Page</b>
Introduction .....	1
Requisites .....	1
Stages of Preparation (Definition) .....	1
Methods of Preparation .....	2
Surface Deformation .....	2
Pressure .....	3
Removal Rate .....	3
Abrasive Sizing .....	4
<b>Stages of Preparation</b>	
Sectioning .....	5, 6
Coarse Grinding .....	6, 7
Fine Grinding .....	6
Rough Polishing .....	7
Abrasives .....	7
Suspension Medium .....	7
Abrasive Selection .....	8
Polishing Cloths .....	8
Final Polishing .....	8
Abrasives .....	8, 9
Polishing Cloths .....	9
Polishing Vehicle .....	9
Polishing Wheel Wetness .....	9
Manipulation .....	10
Cleaning .....	11
Ultrasonic .....	11
Extractor-Condenser .....	11
Drying .....	11

# METALLOGRAPHIC SAMPLE PREPARATION

A

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 occurred. Possible damage traceable to improper sectioning techniques, see Figure 1.

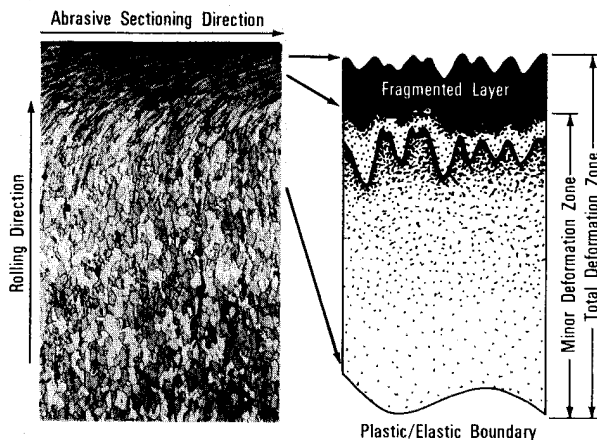


Fig. 1

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

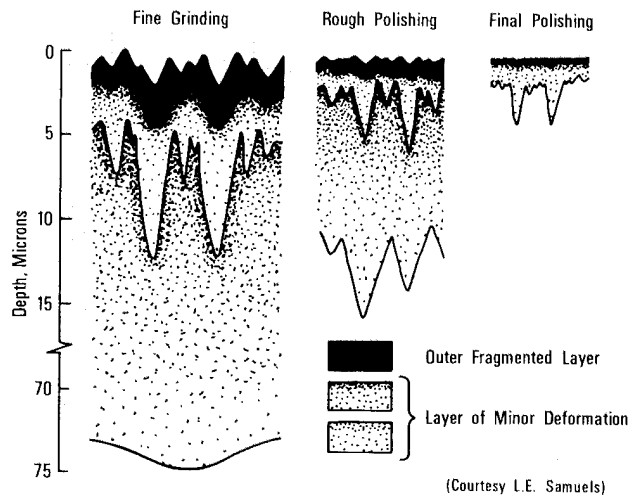


Fig. 2

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.

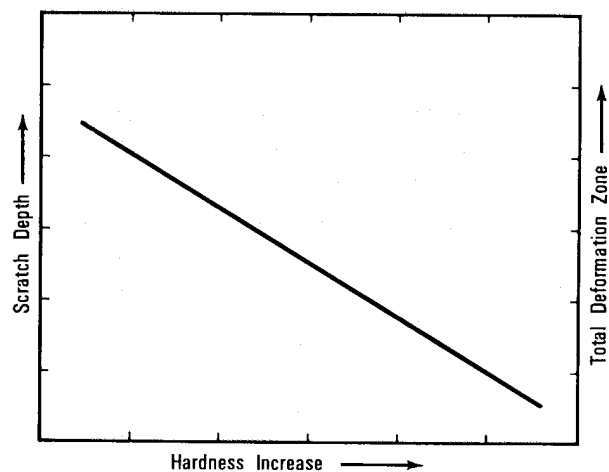


Fig. 2A

## 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.

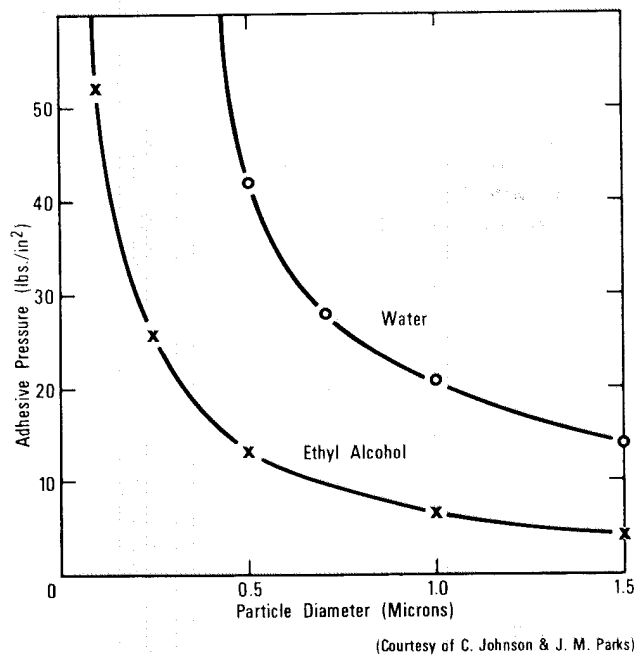


Fig. 3

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.

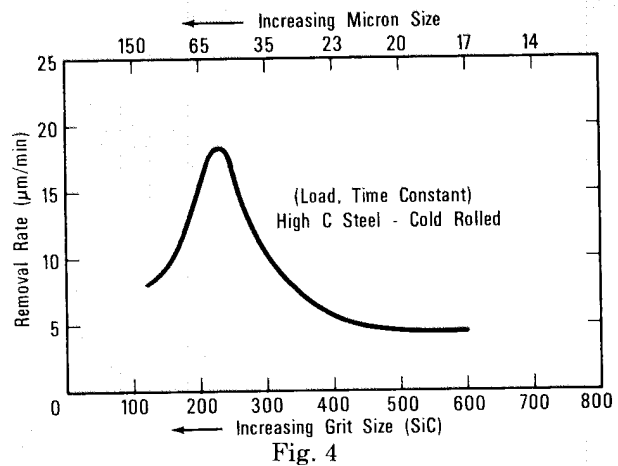


Fig. 4

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.

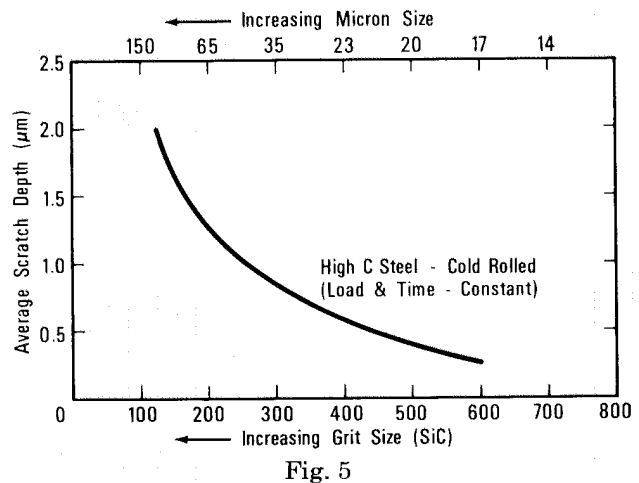


Fig. 5

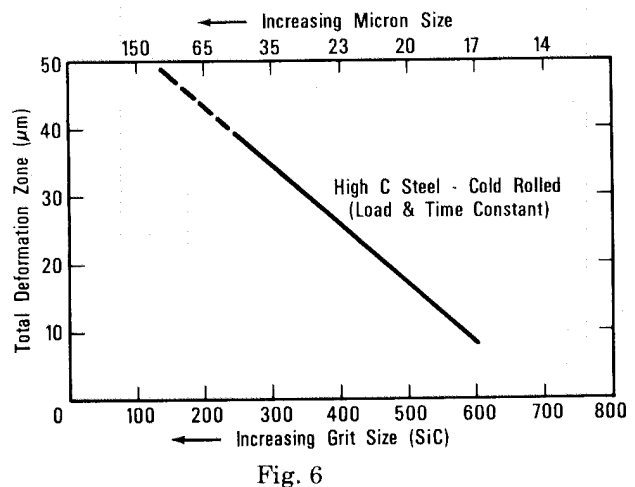


Fig. 6

# 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.

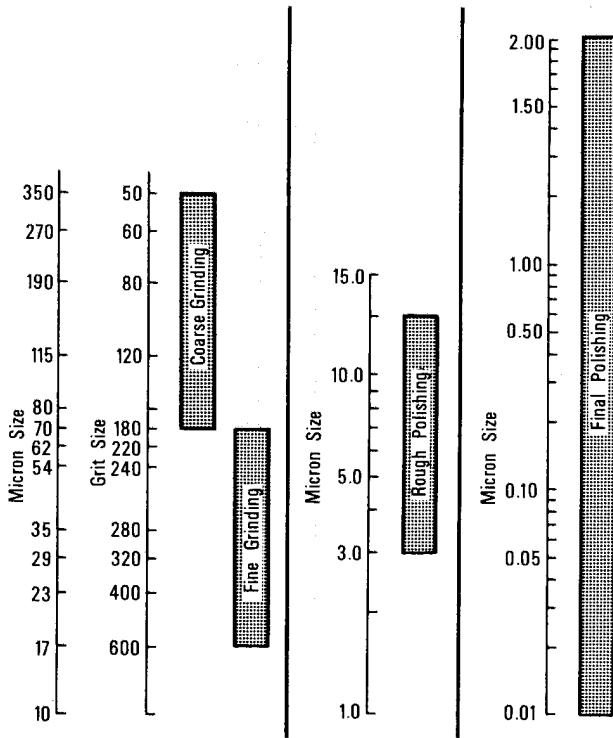


Fig. 7

Table I  
SILICON CARBIDE, ALUMINUM OXIDE, GARNET

Grit No.	Particle Size (AV. $\mu\text{m}$ )			
	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. $\mu\text{m}$ )
3	85
2	70
1	50
1/0	33
2/0	30
3/0	28
4/0	25

Table III.  
DIAMOND PASTES

Micron Size* ( $\mu\text{m}$ )	Size Range ( $\mu\text{m}$ )	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 ( $\mu\text{m}$ )	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 ( $\mu\text{m}$ )	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<br>Type $Al_2O_3$ or SiC<br>Grit Size | c) Hardness of Bond<br>Soft<br>Medium<br>Hard |
| b) Bond<br>Rubber<br>Resinoid<br>Resin/Rubber           | d) Density (Structure)<br>Open<br>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.

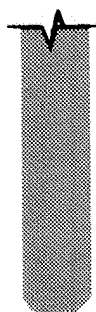
The rate of bond breakdown is related to several factors:

- Bond hardness.
- Hardness and workability of sample.
- Size and speed of abrasive wheel.
- Power of driving motor.
- Type, amount and method of coolant application.
- Amount of pressure applied to wheel.

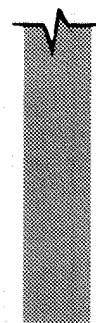
**COOLING** — Sufficient and proper cooling is very important. High volume jet spraying or submerged cutting are the two major techniques used. Section size, material and hardness dictate which method should be employed. Submerged cutting will tend to make a wheel bond act harder.

**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.

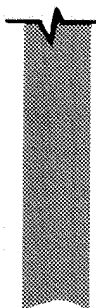
**WHEEL EDGE WEAR** may be used as a very good guide to indicate whether the proper wheel has been selected.



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

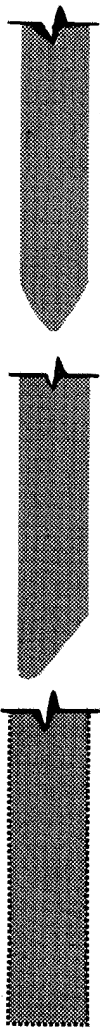


Square edges are retained on a wheel well suited to cutting both solids, sample standards and tubing of medium wall thickness.



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, in-

ertness and low coefficient of friction. 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.

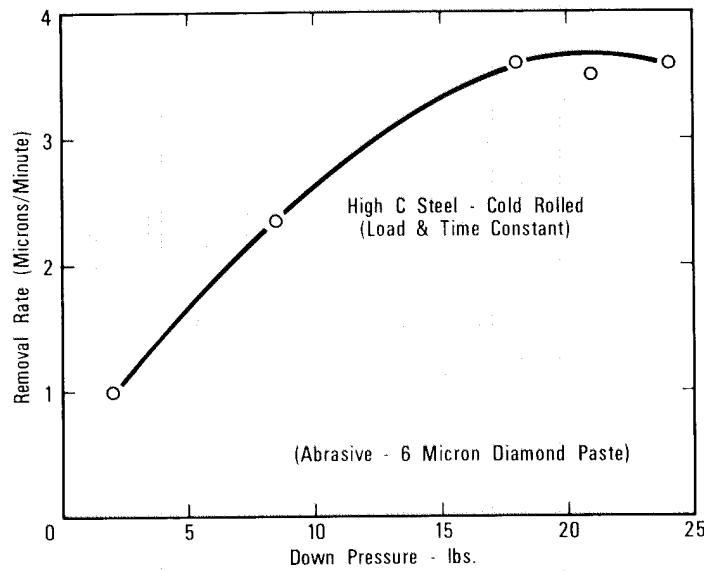
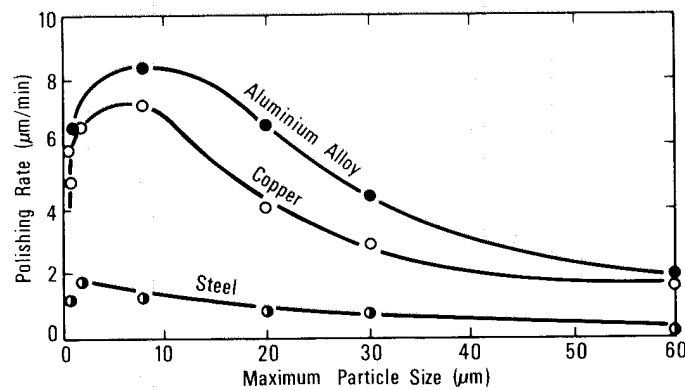


Fig. 8



(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.

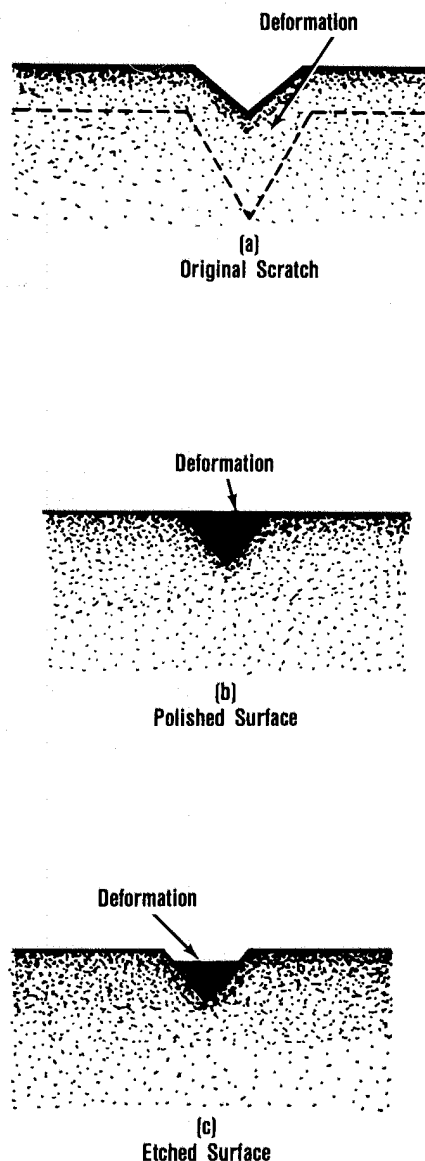


Fig. 10

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 non-polar 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.

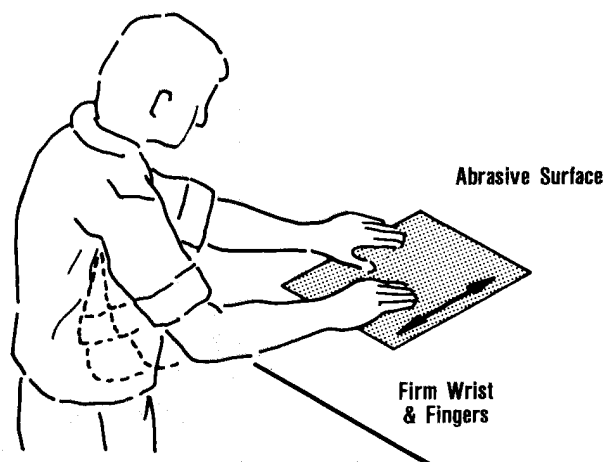


Fig. 11

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.

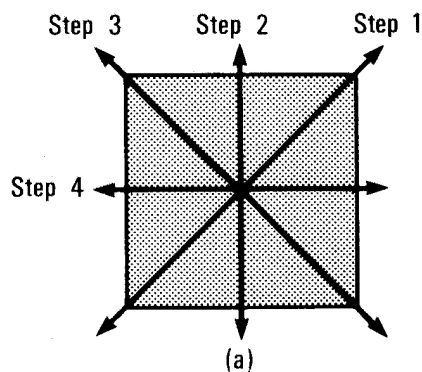


Fig. 12A

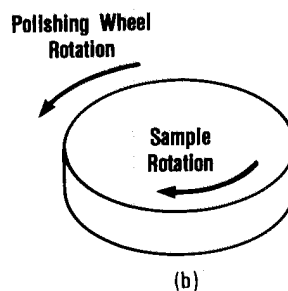


Fig. 12B

**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.

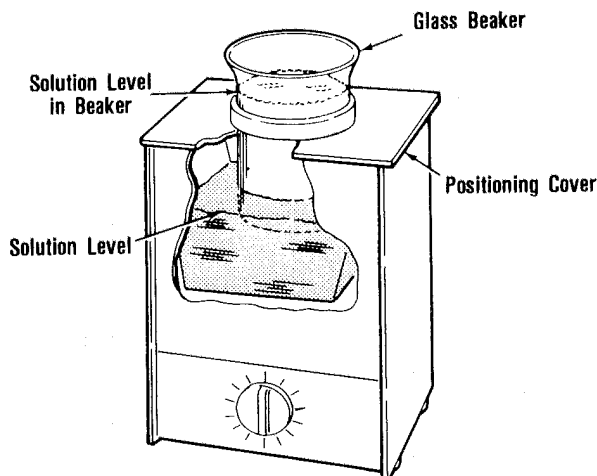


Fig. 13

## EXTRACTOR-CONDENSER

The system consists of an Erlenmeyer 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.

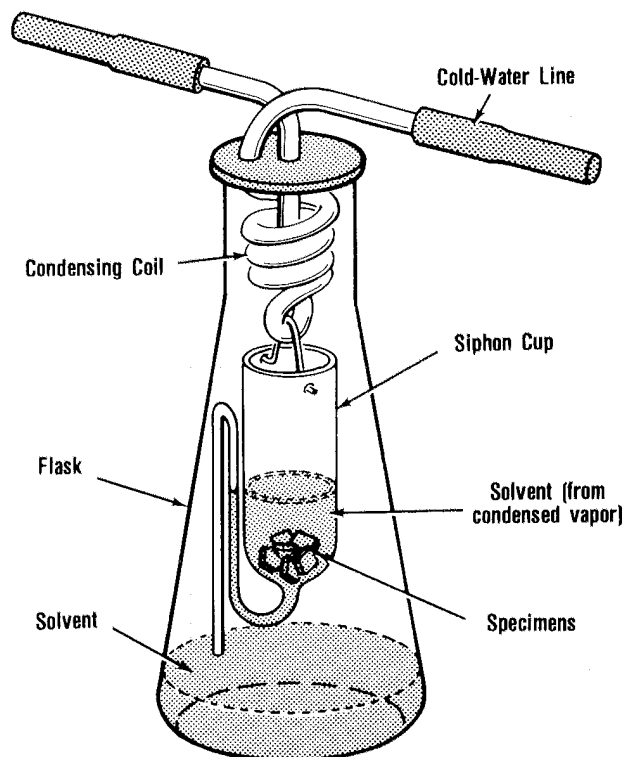


Fig. 14

## 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.

<b>Mounting Procedures</b>	<b>Page</b>
Specimen Mounting (Purposes).....	13
Compression Mounting.....	13
Thermosetting Molding Defects.....	13, 14
Thermoplastic Molding Defects.....	14
Cold Mounting.....	14
Epoxide Molding Defects.....	14, 15
Polyester Molding Defects.....	15
Acrylic Molding Defects.....	15
Vacuum Impregnation.....	15
Procedure.....	15
Care.....	15, 16
Edge Protection.....	16
Steel Shot.....	16
Electro Or Electroless Deposition.....	16
Glass Filled Mounting Media.....	16
Support Strips.....	16
Ceramic Filler.....	16
Fluid Bed Coating.....	17



# 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.

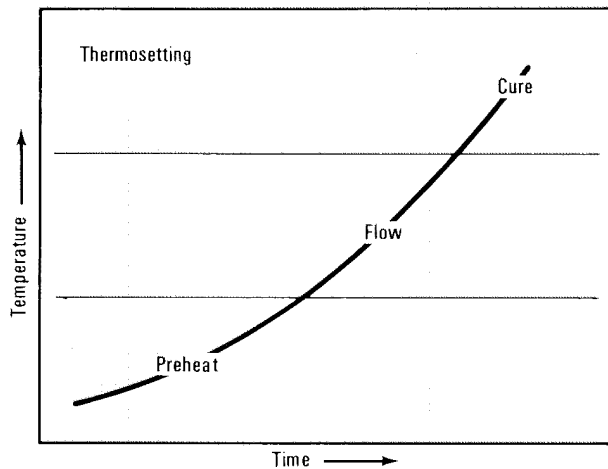


Fig. 15

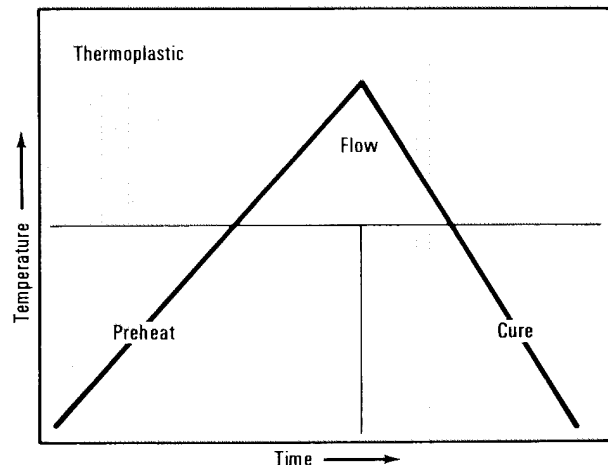


Fig. 16

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.

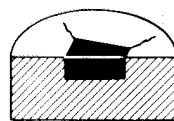
The material readily flows into small areas. This factor plus the allowable pressure variances make the material very desirable for small, fragile pieces. These possibilities very often offset the longer times involved in molding transparent mounts.

Table V.  
Molding Temperatures and Pressures

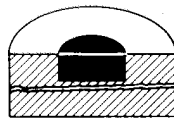
Material	Form	Classification	Molding Temp (°F)	Molding Pressure (psi)
Bakelite®	Powder	Thermosetting	270°-300°	4200
Bakelite®	Preform	Thermosetting	270°-300°	4200
Diallyl-Phthalate	Powder	Thermosetting	280°-290°	2500-3000
Epoxy	Powder	Thermosetting	280°-300°	1000-3000
Lucite® (transparent)	Powder	Thermoplastic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

\*Cure cycle only.

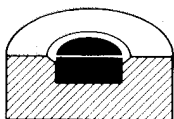
## THERMOSETTING MOLDING DEFECTS



**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.



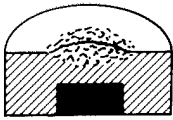
**Split (Circumferential):** Material has absorbed moisture. Gases released by chemical reaction. Use preheated powders or premolds. Momentarily release pressure during flow stage.



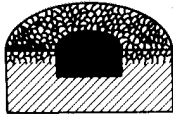
**Shrinkage (Edge):** Poor adhesion to sample surface with excessive shrinkage at interface. Use lower molding temperature.



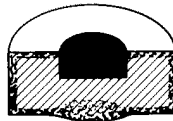




**Burst (Front Surface):** Insufficient pressure and/or insufficient cure time. Adjust molding pressure. Increase cure time.



**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:**

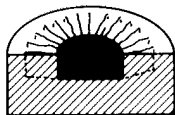
If mold temperature is too high, the following difficulties may also be encountered:

- (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.

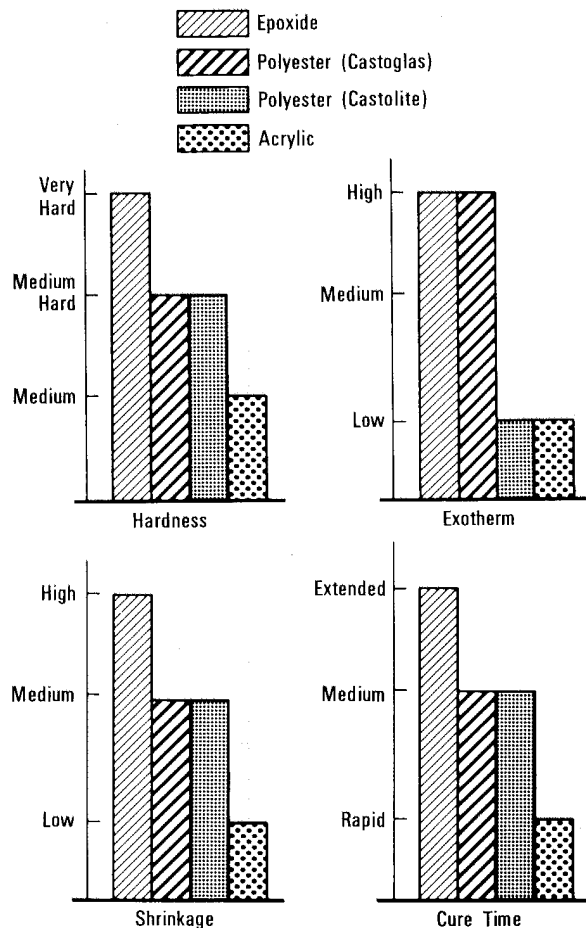
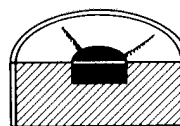
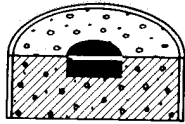


Fig. 17

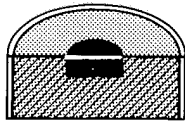
**EPOXIDE MOLDING DEFECTS**



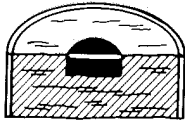
**Cracking:** Resin to hardener ratio incorrect. Exotherm too extreme. Correct resin to hardener ratio. Use forced cool air to control rate of exotherm.



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

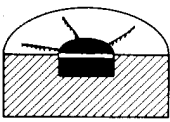


**Discoloring:** Hardener has oxidized. Resin to hardener ratio incorrect. Keep containers tightly sealed. Correct resin to hardener ratio.

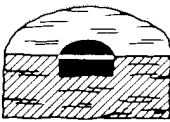


**Soft Mounts:** Resin to hardener ratio incorrect. Incomplete blending of resin and hardener mixture. Correct resin to hardener ratio. Completely blend mixture.

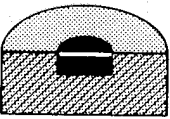
### POLYESTER MOLDING DEFECTS



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

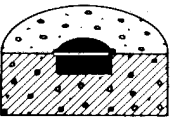


**Discoloration:** Resin has oxidized. Resin to hardener ratio incorrect. Keep container tightly sealed. Correct resin to hardener ratio.



**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

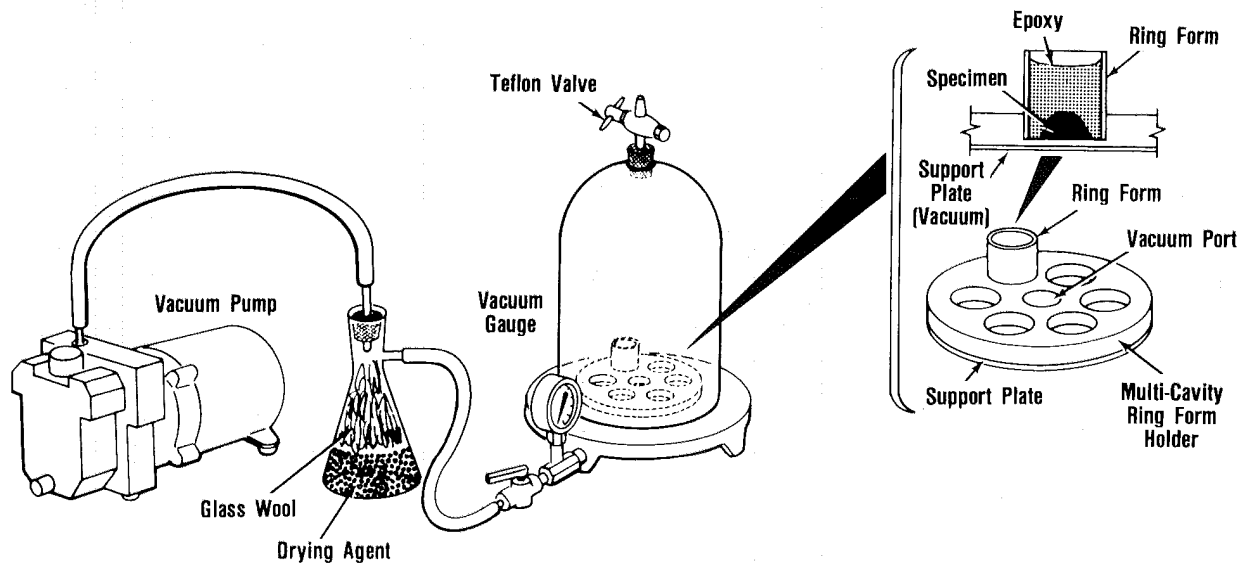


Fig. 18

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 1/2 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.

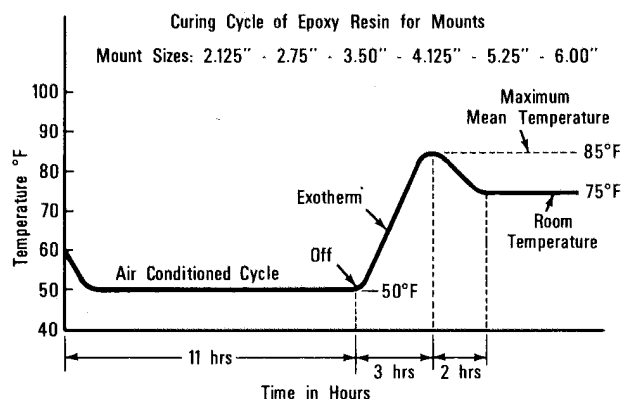


Fig. 19

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 1 1/2 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.

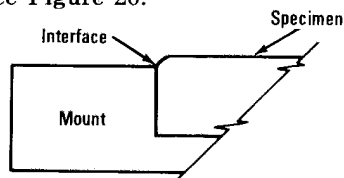
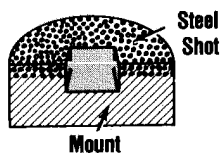
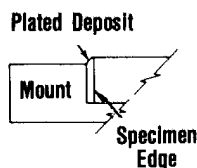


Fig. 20

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

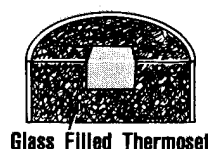


**Steel Shot:** This technique serves the same purpose as the following suggestion.

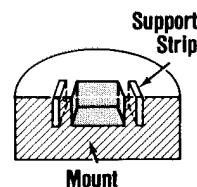


**Electro or Electroless Deposition:** The section is plated with a coating sufficiently thick to compensate for edge rounding during preparation. The more common materials are Ni and Cu. Nickel will sometimes peel away from a surface due to its stressed condition. Copper is generally used for post-plating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

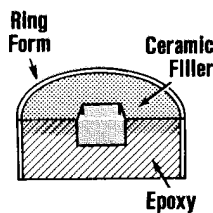
Commercially available electroless coatings are generally stress free. Selection of plating material should be given prior thought regarding etchant rate and reaction with the different metals involved.



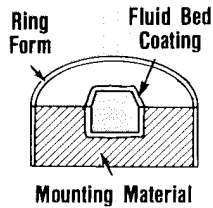
**Glass Filled Mounting Media:** Thermosetting mounting media with special filler additives will often offer sufficient edge support to prevent rounding. Straight mineral filled epoxies are also helpful if allowed to post cure at room temperature to increase the hardness level.



**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.

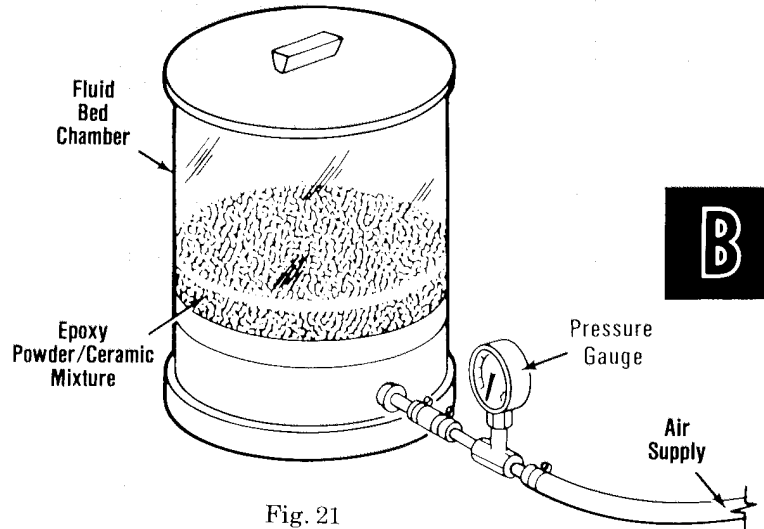
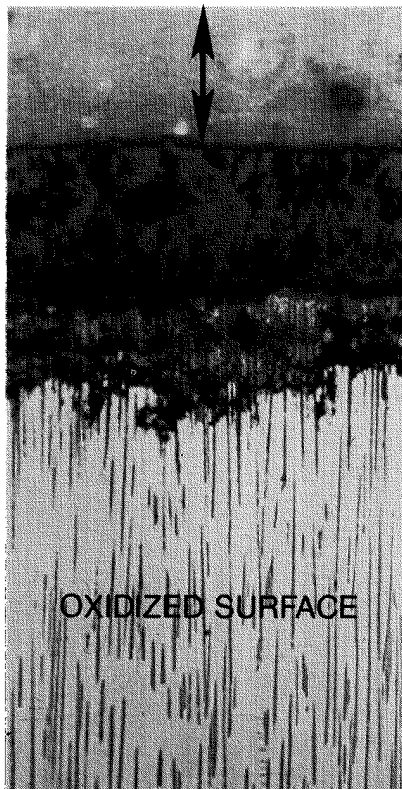


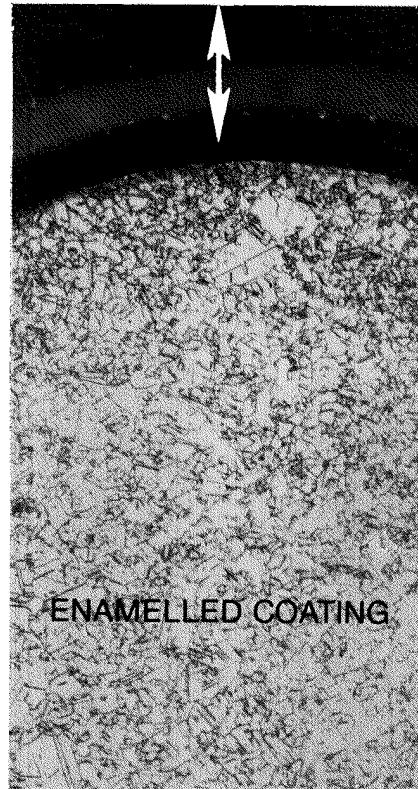
Fig. 21



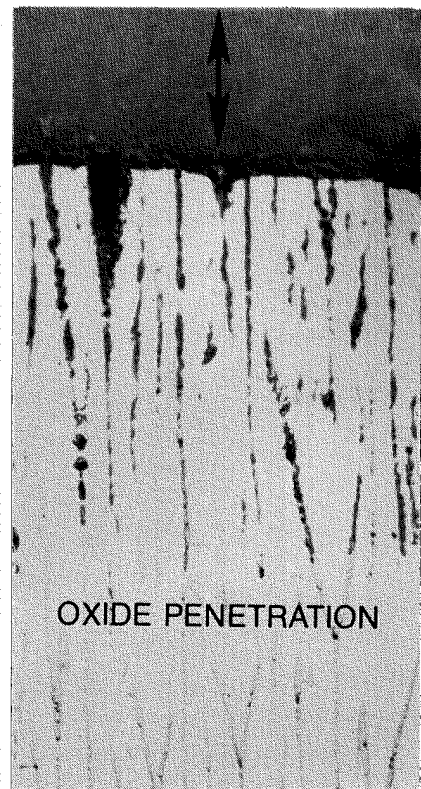
### FLUID BED COATING



X 150  
Co-Ta-Carbide



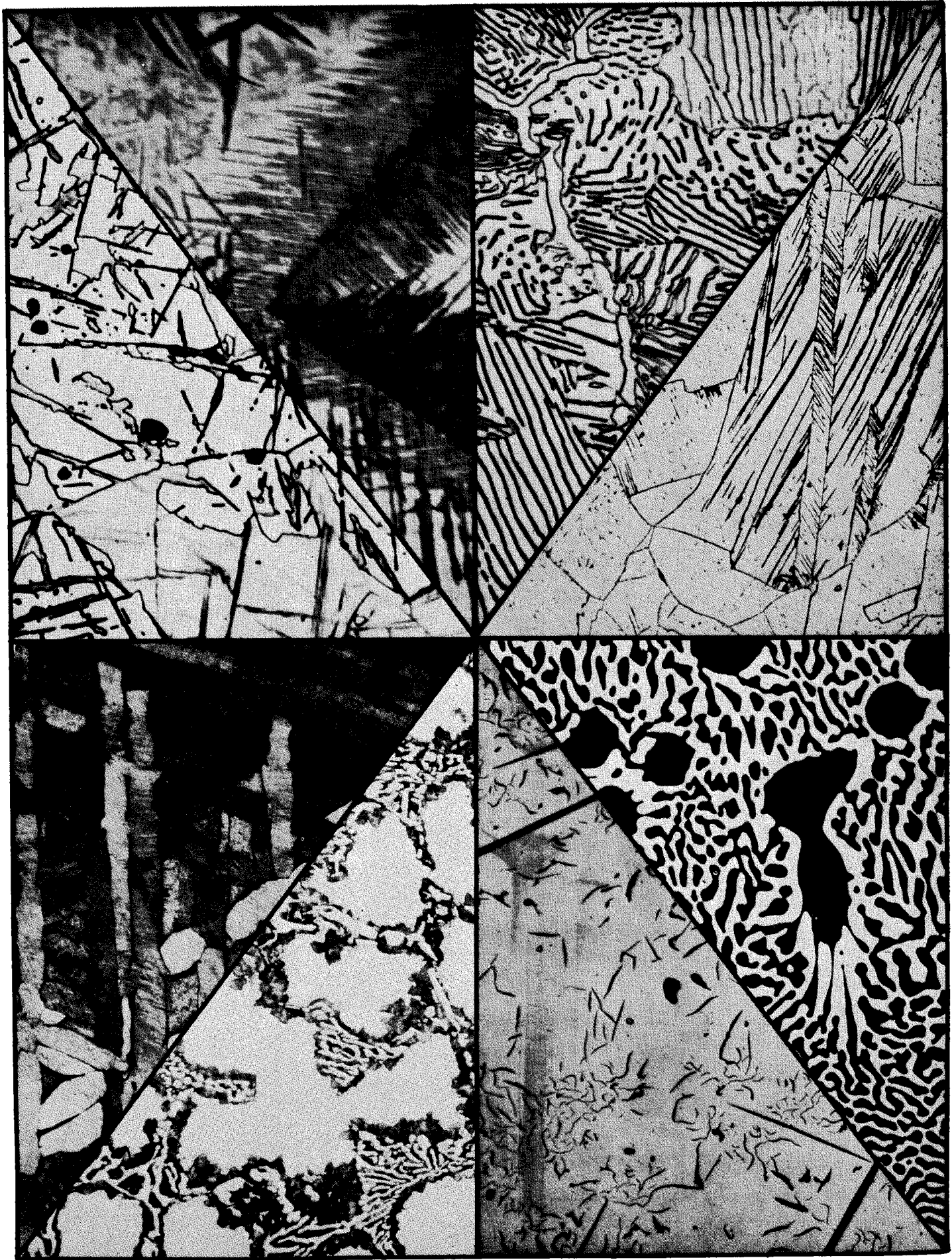
X 150  
Cu Wire



X 400  
Co-Ta-Carbide

<b>Manual or Semi-Automatic Polishing Procedure</b>	<b>Page</b>
I. Polishing Procedure for Ferrous Materials . . .	19
General . . . . .	19
Graphitic Cast Irons . . . . .	19
Galvanized Coatings . . . . .	19
Stainless Steels, Stainless Steel Casting . . . . .	19
Alloys, Heat-Resisting Alloys . . . . .	19
II. Polishing Procedures for Copper Based Materials . . . . .	19,20
III. Polishing Procedures for Aluminum and Magnesium Based Materials . . . . .	20
IV. Polishing Procedures for Titanium, Zirconium, Hafnium and Alloys . . . . .	20,21
V. Polishing Procedures for Cemented Carbides .	21
VI. Polishing Procedures for Lead Alloys, Tin Alloys, Zinc Based Die Castings . . . . .	21
VII. Polishing Procedure for Refractory Alloys and Metals (Nb, Mo, W, V, Ta) . . . . .	21,22
VIII. Polishing Procedure for Plated Sections . . . . .	22
IX. Polishing Procedure for Powder Metals and Alloys . . . . .	22
X. Polishing Procedure for Ceramics . . . . .	22





## 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:

- Al<sub>2</sub>O<sub>3</sub>-Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.
- 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<sub>2</sub>O<sub>3</sub>.

### 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:

- 240, 320, 400, 600 grit SiC or Al<sub>2</sub>O<sub>3</sub> paper or disc with water as lubricant. Clean thoroughly and dry.
- Etch rather heavily. Use 4% Picric Acid Solution for pearlitic matrix materials and 4% Nital Solution for ferritic matrix materials.
- 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

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

- 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.
- 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.
- Check the possibilities of Electropolishing, particularly with solid solution alloys and transformed structures.
- 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:

- Al<sub>2</sub>O<sub>3</sub>-Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.
- 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<sub>2</sub>O<sub>3</sub>.

### 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

- Check the possibilities of Electropolishing particularly with microstructure amenable to same.
- 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.
- 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:

- $Al_2O_3$ -Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.
- 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 ( $\mu m$ )	Nylon Cloth	Lapping Oil

#### FINAL POLISHING

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

#### REMARKS:

- 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.
- 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.

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

- $Al_2O_3$ -Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.
- 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 ( $\mu m$ )	Nylon Cloth	Lapping Oil

#### FINAL POLISHING

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

#### REMARKS:

- 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 ( $\mu\text{m}$ ) (280 mesh)	Resin-Bonded Diamond Disc	Water
Diamond 15 ( $\mu\text{m}$ )	Resin-Bonded Diamond Disc	Water

### REMARKS:

For gross surface removal, employ a 63-74  $\mu\text{m}$  (220 Mesh) Resin-Bonded Diamond Disc to decrease wear on 45 ( $\mu\text{m}$ ) (280 Mesh) Disc.

### ROUGH POLISHING

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond Paste 6 ( $\mu\text{m}$ )	Pan K (PVC Chemotextile)	Lapping Oil
Diamond Paste 3 ( $\mu\text{m}$ )	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 ( $\mu\text{m}$ )	Pan K (PVC Chemotextile)	Lapping Oil

### REMARKS:

If extremely fine scratches are visible in the binder material, a short cycle on Lecloth<sup>®</sup> 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)  $\text{Al}_2\text{O}_3$  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  $\text{Al}_2\text{O}_3$ .

### 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<sup>®</sup> 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 <sup>®</sup> (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 ( $\mu\text{m}$ )	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 ( $\mu\text{m}$ )	Lecloth <sup>®</sup> (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:

- Al<sub>2</sub>O<sub>3</sub>-Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action may be less severe.
- 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<sub>2</sub>O<sub>3</sub>.

#### 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

#### REMARKS:

- During preparation softer electrodeposits may tend to flow and the interfaces between the various layers will not be clearly delineated.
- Edge Protection — Suggestions for Post Plating and Fluid Bedding are described under Mounting Procedures.
- 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:

- Al<sub>2</sub>O<sub>3</sub>-Coated Products (waterproof). Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.
- 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<sub>2</sub>O<sub>3</sub>.

#### 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:

- 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.

- 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.

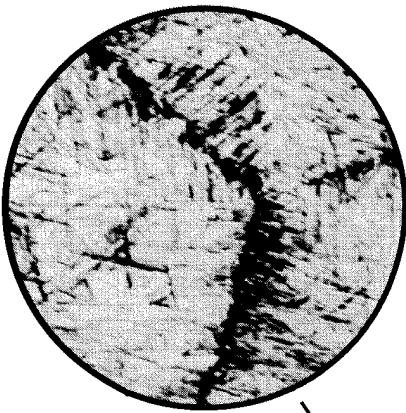
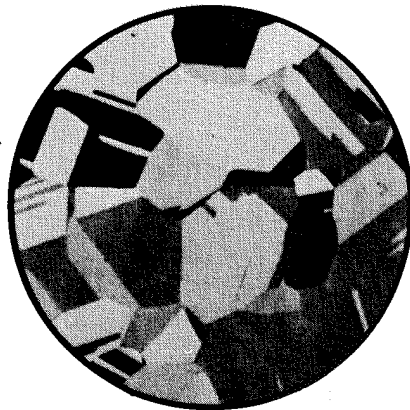
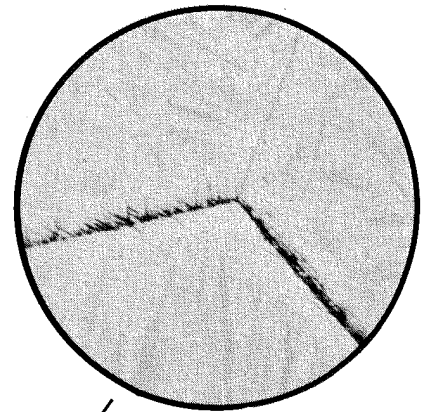
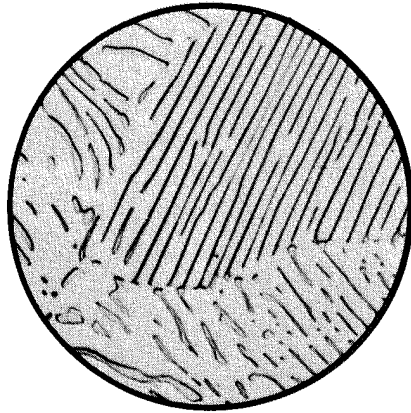
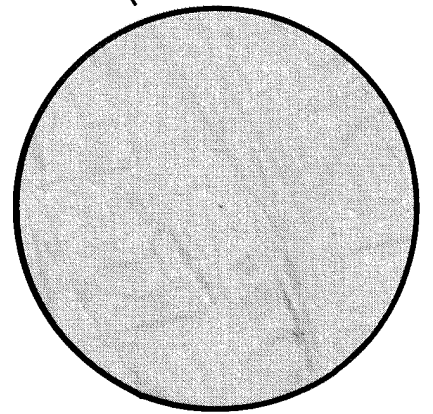
#### FINAL POLISHING

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.

<b>Electrolytic Polishing</b>	<b>Page</b>
Theory of Electropolishing .....	23, 24
Recommended Electrolytes for Various Materials ..	25
Electrolyte Compositions .....	26
Chemical Reagent Specifications .....	27
Electrolyte Hazards .....	27
Causes and Suggested Cures for Unsatisfactory Surfaces .....	28

D



# ELECTROLYTIC POLISHING

Electrolytic Polishing is a process wherein a metal sample is made the anode in an electrolytic cell. Material is removed by electrolytic action in such a manner that surface irregularities are removed and the finish becomes smooth and bright. Dependent upon solutions and electrolysis conditions the metal anode may be either etched, polished, completely or partially passivated.

The use of anodic polishing in sample preparation demands the selectivity of attack on the existent phases within a microstructure remains within certain limitations. This is necessary to prevent exaggerated artifacts in the overall structure which might preclude proper interpretation.

With electropolishing, a potential difference is existent between the matrix and secondary phases. Should the second phase be anodic or less noble, it will be preferentially attacked. If it is cathodic or more noble, the matrix will be attacked in the areas adjacent to the second phase.

The anode film (see Fig. 22) controls the rate at which the dissolution products can diffuse away from the anode. Since there is a difference in concentration gradient, (i.e. a thin film with a high concentration gradient existent at a depression), preferential dissolution occurs at the asperity and the surface becomes "smoothed."

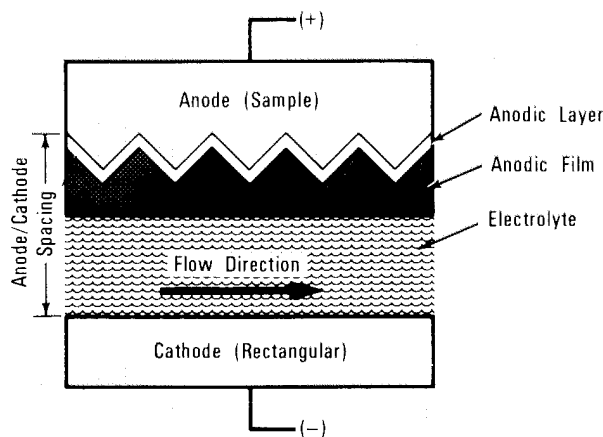


Fig. 22

There must be another factor present in the electrolytic process responsible for the "brightening." Strong evidence supports the formation of some type layer between the anodic film and anodic surface as the contributing factor, see Figure 22. Perhaps in explanation this anodic layer follows the contour of the anodic surface and is uniformly attacked by the electrolyte. The passage of ions across the anodic layer interface must occur at the same rate at all points to maintain this layer and cause "brightening."

The existence of such a surface layer could also explain the difference between etching and polishing conditions. Etching will occur if the electrolyte has free access to the anode surface since anodic dissolution occurs preferentially at the sites of high energy.

Anodic dissolution of a metal under polishing conditions involves both electrical and chemical phenomena. The existence of a viscous anodic film can actually be observed during polishing.

Observations have shown (see Figure 22):

- (1) The anodic film thickness remains approximately constant during the preparation cycle.
- (2) The same film is also relatively thick in comparison to the depth of the surface irregularities.
- (3) A planar surface exists between the anodic film and the electrolyte.

There are basically two shapes of Voltage-Current Density Curves inherent to electrolytes, Figure 23 is typical of Low Resistance Electrical Solutions whereas Figure 24 is representative of High Resistance Solutions.

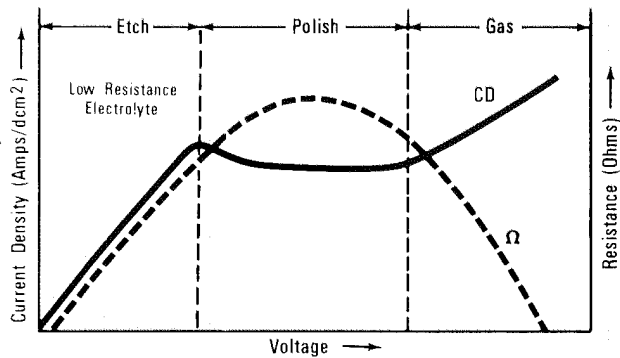


Fig. 23

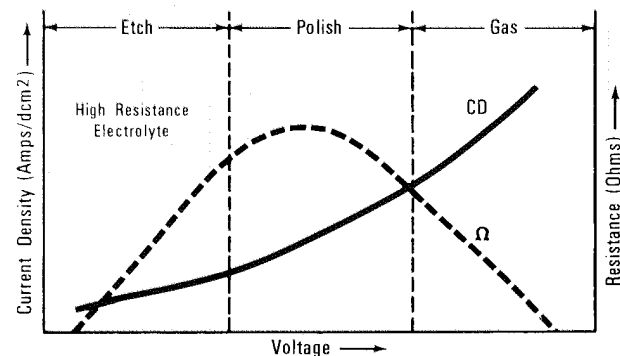


Fig. 24

There are three distinct areas in the above Voltage-Current Density Curves. At relatively low current densities electrolytic etching will occur as the anodic film is either nonexistent or has not reached stability. At increased current densities, polishing will occur. At higher current densities, hydrogen evolution occurs at the cathode. This rapid gas evolution may produce a polished surface, but "pitting" imperfections will be evident due to adherent gas bubbles.



Proper electropolishing conditions exist when the anodic film has maximum resistance.

For idealized electropolishing conditions, it is important to: (a) Closely control or minimize heating of the electrolyte and anode, (b) Operate under conditions to produce maximum resistance in the anodic film, (c) Operate at minimal Current Densities.

Should too much heat be generated, the polished surface may become etched by direct chemical attack and/or show evidence of pitting. These undesirable results may be attributed to the fact that the viscous anodic film is a poor conductor and the anode, having higher conductivity than the electrolyte, will absorb most of the heat generated.

The effects of temperature control at various levels of Current Density demands and Anodic Film Resistance for a typical high resistance electrolyte are shown in Figures 25 and 26.

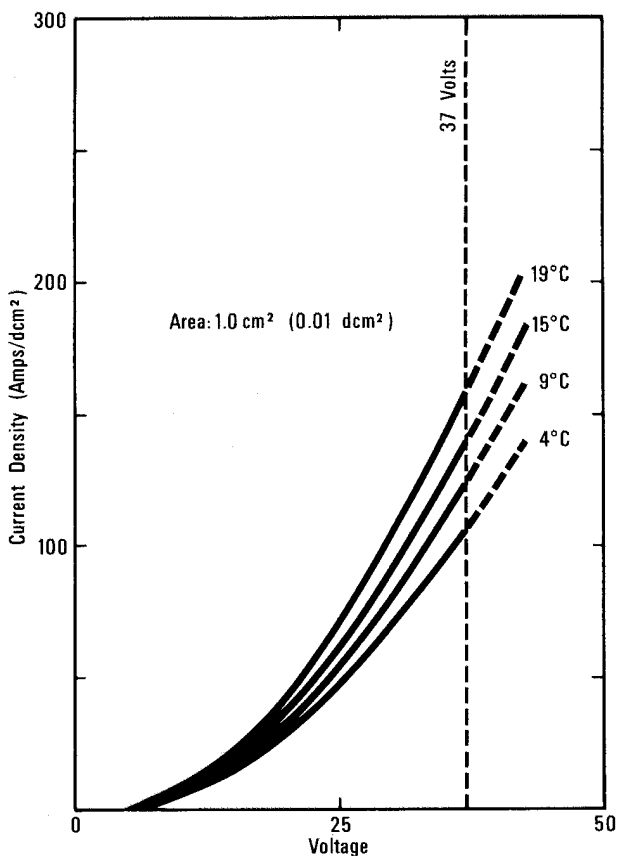


Fig. 25

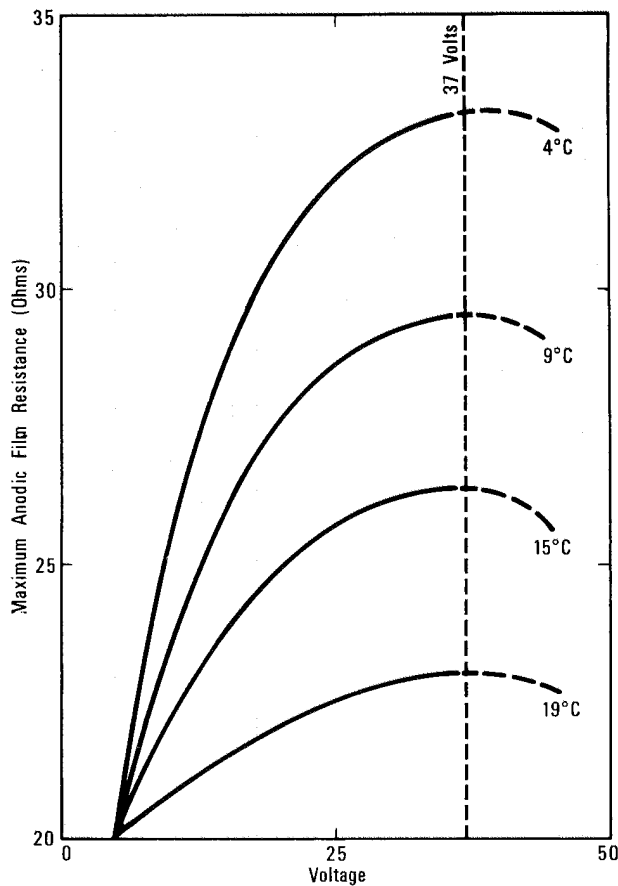


Fig. 26

A specific time is necessary to produce a polished surface with any current density. The time of polishing should be no longer than necessary; or the secondary effects, such as nobility factor, etc., may be intensified. Time is also dependent upon the preparation of the sample prior to electrolytic treatment.

During polishing the metallic content of the electrolyte continually increases due to the dissolution products from the anode. An increase in metallic content increases the conductivity of the solution, as well as the electrolyte life within certain limitations.

The degree of stirring or electrolyte flow is dependent upon cell design. Care must be taken so as not to modify the current paths. The disruption may cause locally etched or unpolished areas.

## Recommended Electrolytes for Various Materials:

### L1 Electrolyte

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Plain Carbon Steels  
High Speed Steels  
Alloy Steels  
Stainless Steels  
Aluminum  
Aluminum Alloys  
Nickel  
Tin

### L2 Electrolyte

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Molybdenum  
Vanadium

### L3 Electrolyte

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Lead  
(Some Pb/Sn Alloys)  
Aluminum-Copper-Magnesium

### L5 Electrolyte

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Cast Iron: Gray, Malleable, Nodular

### L7 Electrolyte

---

Zinc  
(Some Zn Alloys)  
Magnesium

### L8 Electrolyte

---

Copper  
Copper-Nickel  
Brasses (red)  
Brasses (yellow)  
Bronzes (Al)

### L9 Electrolyte

---

Copper  
Bronzes (Mn)  
Bronzes (P)  
Aluminum-Copper

### L10 Electrolyte

---

Materials with disturbing inclusions, such as Low C Steels,  
Irons, Cast Irons, Brasses and Bronzes.

### L11 Electrolyte

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Tungsten  
Cemented Tungsten Carbides

### L12 Electrolyte

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Titanium  
Zirconium  
Hafnium  
Molybdenum  
Vanadium  
Plain C Steels  
High Temperature Alloys

### L13 Electrolyte

---

Cast Irons: Gray, Malleable, Nodular

ELECTROLYTE	COMPOSITIONS	MIXING PROCEDURE
L1	Perchloric Acid ..... 62cc Ethanol ..... 700cc Butyl Cellusolve ... 100cc Distilled Water .... 137cc	Cautiously add perchloric acid to previously prepared mixtures of other components.
L2	Perchloric Acid ..... 47cc Methanol ..... 600cc Butyl Cellusolve ... 360cc Distilled Water ..... 13cc	Same as for Electrolyte No. L1
L3	Perchloric Acid ..... 90cc Ethanol ..... 600cc Acetic Acid ..... 180cc Sodium Hydroxide .. 32g Sodium Acetate ..... 45g	Mix acetic acid and ethanol. Dissolve sodium hydroxide in this mixture. Add sodium acetate to this mixture. Sodium acetate will dissolve as the perchloric acid is added to mixture.
L4	Perchloric Acid ..... 78cc Ethanol ..... 700cc Butyl Cellusolve ... 100cc Distilled Water .... 120cc Tartaric Acid ..... 50g	Mix distilled water and ethanol. Dissolve tartaric acid in this mixture. Add butyl cellusolve and then the perchloric acid.
L5	Perchloric Acid ..... 15cc Ethanol ..... 800cc Propanol ..... 100cc Quinolinol ..... 10g Sodium Thiocyanate 60g Citric Acid ..... 75g	Dissolve citric acid, quinolinol and sodium thiocyanate in the ethanol and propanol alcohol mixture, heating may be required. Add perchloric acid after solution has cooled.
L6	Hydrochloric Acid ... 10cc Methanol ..... 1000cc	Add the hydrochloric acid to the methanol.
L7	Ethanol ..... 800cc Butyl Cellusolve .... 80cc Distilled Water ..... 20cc Sodium Thiocyanate 160g	Dissolve sodium thiocyanate in mixture of ethanol and distilled water. When completely in solution, add butyl cellusolve.
L8	Phosphoric Acid ... 250cc Distilled Water .... 500cc Ethanol ..... 250cc Propanol ..... 50cc Urea ..... 5g	Mix in order listed.
L9	Nitric Acid ..... 30cc Methanol ..... 900cc Cupric Nitrate ..... 300g	Dissolve cupric nitrate in methanol by slowly heating. Allow to cool and carefully add nitric acid immediately before use. <b>DO NOT STORE — DISPOSE OF ELECTROLYTE AFTER USE.</b>
L10	Acetic Acid ..... 120cc Methanol (pure) ... 900cc Propanol ..... 140cc Butyl Cellusolve ... 200cc Cobalt Nitrate ..... 400g Ferric Nitrate ..... 40g Tartaric Acid ..... 140g Urea ..... 4g	Mix methanol and propanol. Dissolve the four (4) dry chemicals in this mixture. <i>NOTE: Tartaric acid will dissolve slowly. Add acetic acid and finally the butyl cellusolve to this mixture.</i>
L11	Sodium Hydroxide .. 20g Distilled Water ... 1000cc	Dissolve sodium hydroxide in water.
L12	Perchloric Acid ..... 60cc Methanol ..... 590cc Butyl Cellusolve ... 350cc	Cautiously add perchloric acid to previously prepared mixture of other components.
L13	Perchloric Acid ..... 50cc Ethanol ..... 750cc Distilled Water .... 140cc	Cautiously add perchloric acid to previously prepared mixture of other components.



#### CHEMICAL REAGENT SPECIFICATIONS:

Acetic Acid (glacial) . . . . .	99% CH <sub>3</sub> COOH
Butyl Cellusolve . . . . .	Ethylene Glycol Monobutyl Ether, CH <sub>3</sub> (CH <sub>2</sub> ) <sub>2</sub> CH <sub>2</sub> OCH <sub>2</sub> CH <sub>2</sub> OH
Citric Acid . . . . .	[(COOH)CH <sub>2</sub> ] <sub>2</sub> COHCOOH•H <sub>2</sub> O
Cobalt Nitrate . . . . .	Co(NO <sub>3</sub> ) <sub>2</sub> •6H <sub>2</sub> O
Cupric Nitrate . . . . .	Cu(NO <sub>3</sub> ) <sub>2</sub> •3H <sub>2</sub> O
Ethanol . . . . .	(Ethanol - 96% vol.), CH <sub>3</sub> CH <sub>2</sub> OH
Ferric Nitrate . . . . .	Fe(NO <sub>3</sub> ) <sub>3</sub> •9H <sub>2</sub> O
Hydrochloric Acid . . . . .	37% HCl
Manganese Nitrate . . . . .	Mn(NO <sub>3</sub> ) <sub>2</sub> •6H <sub>2</sub> O
Methanol (absolute) . . . . .	CH <sub>3</sub> OH
Nitric Acid . . . . .	70% HNO <sub>3</sub>
Perchloric Acid . . . . .	70% HClO <sub>4</sub>
Phosphoric Acid . . . . .	85% H <sub>3</sub> PO <sub>4</sub> (ortho phosphoric)
Propanol . . . . .	n•propanol
Quinolinol . . . . .	C <sub>9</sub> H <sub>7</sub> NO
Sodium Acetate . . . . .	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> Na•3H <sub>2</sub> O
Sodium Hydroxide . . . . .	NaOH (pellets)
Urea . . . . .	CO(NH <sub>2</sub> ) <sub>2</sub> (carbomide)

*NOTE: All chemicals should be analytical grade.*

#### Electrolyte Hazards

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We warn against solutions containing high percentages of perchloric acid in acetic acid or acetic anhydride and those with obnoxious and toxic fumes.

There are other solutions which are strong oxidizing agents, particularly chromic acid or nitric acid. When these are mixed with organic chemicals, such as acetic anhydride or methyl alcohol, any acceleration of the exothermic oxidation could cause explosion.

The following reference sources are cited for available literature in handling such materials.

#### Perchloric Acid

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- (1) ASTM Standard E50-73

“Recommended Practices for Apparatus, Reagents and Safety Precautions for Chemical Analysis of Metals”, American Society for Testing Materials”,  
1916 Race Street.  
Philadelphia, Pa. 19103

- (2) “Bulletin SD11”

Manufacturing Chemists Association  
1825 Connecticut Avenue, N.W.  
Washington, D.C. 20009

#### Chromium Trioxide

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“Bulletin SD44”  
Manufacturing Chemists Association  
1825 Connecticut Avenue, N.W.  
Washington, D.C. 20009

#### Nitric Acid

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“Bulletin SD 5”  
Manufacturing Chemists Association  
1825 Connecticut Avenue, N.W.  
Washington, D.C. 20009

## CAUSES AND SUGGESTED CURES FOR UNSATISFACTORY SURFACES

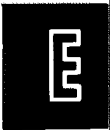
The use of an electrolyte is the most important source of deterioration. Impurities and too great a metallic ion content act as catalysts in deteriorating a solution. This may cause artifacts and lead to misinterpretation of a microstructure.

Surface Condition	Probable Cause	Suggested Correction
Edge of specimen pitted or etched.	Film too viscous or too thin.	Increase flowrate. Use less viscous electrolyte. Decrease voltage.
Center of specimen etched.	No film formed at center of specimen.	Increase voltage. Reduce flowrate.
Deposits on specimen.	Insoluble anode reaction product.	Use fresh electrolyte. Use different electrolyte. Increase voltage.
Etching or staining of specimen.	Chemical attack after current is "OFF".	(1) Remove specimen immediately after current is off. (2) Use a less corrosive electrolyte.
Relief effects.	(1) Polishing film inadequate. (2) Nobility relationship of phases present.	(1) Increase voltage. (2) Improve pre-preparation of sample and reduce polishing time.
Waviness on polished surface.	(1) Insufficient time. (2) Flowrate too high or low. (3) Unsuitable preparation.	(1) Increase voltage. (2) Increase or decrease flowrate (3) Improve preparation. (4) Decrease time.
Unpolished spots.	Gas bubbles.	(1) Increase flowrate. (2) Too high electrolyte temperature. (3) Decrease voltage.
Roughness or matte surface.	Insufficient or no polishing film.	(1) Increase voltage. (2) Use more viscous electrolyte.
Pitting.	Too long polishing time. Too high voltage.	(1) Improve pre-preparation. (2) Decrease voltage. (3) Decrease time. (4) Determine if the pits are inclusions or phases attacked due to nobility factor.

<b>Slurry (Etch-Attack) Polishing</b>	Page
Theory and Techniques .....	29
Table VI - Slurry (Etch-Attack) Solutions .....	29,30,31

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<b>Chemical Polishing</b>	Page
Theory and Technique .....	32
Table VII - Chemical Polishing Solutions .....	32,33



# SLURRY (ETCH-ATTACK) POLISHING

Slurry Polishing is a process wherein an etching reagent is added to the polishing compound to increase removal rate. The techniques are generally restricted to the Rough & Final Polishing Stages.

Relief polishing and etch pit tendencies are greatly reduced. Some additions are not reactive with the sample material, but continually function to remove any passivating film.

Concentration of the additive etchant must be adjusted so the mechanical removal rate slightly exceeds the chemical removal rate.

Solutions which may be helpful are listed in Table VI.

TABLE VI- SLURRY (ETCH-ATTACK) SOLUTIONS

Metal	Reagent	Abrasive	Remarks
Beryllium	Oxalic Acid . . . 10g Water . . . . . 100g Abrasive . . . . . 5-8g	Al <sub>2</sub> O <sub>3</sub>	Rough polishing: 0.3 (μm)-grade alumina on a silk cloth. Final polishing: 0.05 (μm)-grade alumina on a napped cloth.
Chromium	Acetic acid . . . 15g Water . . . . . 150ml 1% oxalic acid in 1:1 ethyl alcohol, water solution.	Al <sub>2</sub> O <sub>3</sub>	Final polishing: Chemical resistant cloth necessary.
Columbium (Niobium) & Columbium Alloys	Ammonium Hydroxide . . . 200ml Copper Sulfate 40g Water . . . . . 300ml  Sodium Hydroxide . . . . . 2g Potassium Ferricyanide . . . . . 35g Water . . . . . 150ml	Al <sub>2</sub> O <sub>3</sub>	Rough polishing: 0.3 (μm)-grade on a nylon cloth necessary. Final polishing: 0.05 (μm)-grade on a napped cloth. *PVC or polyethylene polishing laps recommended.  Final polishing: 0.05 (μm)-grade on a napped cloth. *PVC or polyethylene laps recommended.
Copper	Up to 3% ammonium persulphate solutions.  Ammonia, ammonium persulphate, cupric ammonium persulphate.  2-10% chromic acid solution	MgO  Al <sub>2</sub> O <sub>3</sub> Al <sub>2</sub> O <sub>3</sub>	Final polishing: by skidding technique.  Added to polishing pad.  Abrasive suspended in reagent and used on a napped cloth.
Gold	5-10% chromic acid solution  Pot. Iodide . 12.5g Water . . . . . 100ml	Al <sub>2</sub> O <sub>3</sub> Al <sub>2</sub> O <sub>3</sub>	Abrasive suspended in reagent and used on a napped cloth.  Drops of reagent added to normal polishing pad.
Magnesium	2% Pot. Dichromate solution 20ml Sat. solution boric acid . . . . . 150ml Nitric acid (concentrated) 15 drops  Ammonium hydroxide	Al <sub>2</sub> O <sub>3</sub> CeO	Suitable for pure magnesium, slight variations being desirable for alloys.  Final polishing on napped cloth. Magnesium-lithium alloys when corrosion is encountered.



TABLE VI — SLURRY (ETCH - ATTACK) SOLUTIONS, continued

Metal	Reagent	Abrasive	Remarks
Silver	Chromic acid, solution . . . 100ml Hydrochloric acid, (10% soln.) . . . 45ml Water . . . . . 800ml	nil	Nylon cloth necessary. Specimen should only lightly contact the cloth. Suitable for removing large thicknesses, the removal rate being about 25 $\mu\text{m}$ /min. After polishing, surface must be swabbed lightly in the polishing solution for 30 seconds while being rinsed under water; otherwise a thin adherent film of chromate will form.
Tantalum & Tantalum Alloys	See solution Columbium (Niobium) & Columbium Alloys above.		
Thorium	10% oxalic acid solution	$\text{Al}_2\text{O}_3$	Rough polishing: Abrasive suspended in reagent.
	Nitric acid . . . 10ml Hydrofluoric acid . . . . . 1ml Water . . . . . 98ml	$\text{Al}_2\text{O}_3$	Final polishing: Reagent added to normal polishing pad.
Titanium	20% chromic acid solution . . . . . 30ml Water . . . . . 200ml Abrasive . . . . . 15g	$\text{Al}_2\text{O}_3$	Rough polishing on a high-speed wheel. Surface produced is passive, but passivity can be removed by a further treatment on a normal pad.
	Hydrofluoric acid . . . . . 1.5ml Nitric acid . . 3.5ml Water . . . . . 95ml	$\text{Al}_2\text{O}_3$	Final polishing: Add few drops to a normal pad at the end of the treatment.
	5% oxalic acid solution	$\text{Al}_2\text{O}_3$	Final polishing: Suspend abrasive in reagent.
	Hydrofluoric acid . . . . . 1.5ml Nitric acid . . 1.5ml Water . . . . . 100ml Abrasive . . . . . 10g	$\text{Al}_2\text{O}_3$	Final polishing.
	1% Hydrofluoric acid solution	$\text{Al}_2\text{O}_3$	Final polishing: Suspend abrasive in reagent.
	See solution (2) Columbium (Niobium) & Columbium Alloys above.		
Tungsten	Potassium Ferricyanide . . . 3.5g Sod. hydroxide . 1g Water . . . . . 100ml Abrasive . . . . . 10g	$\text{Al}_2\text{O}_3$	Final polishing: Develops some grain structure.
	Copper sulphate 1g Amm. Hydroxide . . . . . 5-10ml Water . . . 1,000ml	$\text{Al}_2\text{O}_3$	Final polishing: Suspend abrasive in reagent. Give alternate treatments on a normal polishing pad.

TABLE VI — SLURRY (ETCH-ATTACK) SOLUTIONS, continued

Metal	Reagent	Abrasive	Remarks
Uranium	Chromic acid . 10g Nitric acid ... 10ml Water..... 1,000ml	Al <sub>2</sub> O <sub>3</sub>	Rough polishing: Suspend abrasive in reagent. Polythene or PVC apparatus and chemical resistant cloth necessary.
	Hydrofluoric acid ..... 30ml Nitric acid ... 30ml Water..... 60ml Alumina ..... 5-8g	Al <sub>2</sub> O <sub>3</sub>	Final polishing: Swab surface with concentrated nitric acid immediately after polishing.
	5% suspension of abrasive in hydrogen peroxide ..... ..... (30 wt.%)	Al <sub>2</sub> O <sub>3</sub>	Final polishing: Polyethylene or PVC apparatus and chemical resistant cloth necessary.
Zirconium	Hydrofluoric acid ... 4-30 drops Water..... 10ml Abrasive..... 5-6g	Al <sub>2</sub> O <sub>3</sub>	Final polishing: Polyethylene or PVC apparatus and chemical resistant cloth necessary.

(Courtesy Samuels, Coons, Gerardi, et al)

# CHEMICAL POLISHING

These techniques are usually employed after the Final Polishing Stage to aid in the removal of any remnant surface deformation.

The specimen surface is polished by immersion in a suitable solution without applying an external potential.

An explanation of the theory involves the selective dissolution of a surface by setting up local voltaic cells between those regions where the anodic film is

easily removed and the depressions where the film is more stable.

Suggested solutions are given in Table VII.

Unless otherwise specified, the acids in the solutions are concentrated reagent grades.

Orthophosphoric Acid	Sp. gr. 1.70
Sulphuric Acid	Sp. gr. 1.84
Nitric Acid	Sp. gr. 1.40
Hydrochloric Acid	Sp. gr. 1.75
Glacial Acetic Acid	Sp. gr. 1.065

TABLE VII — CHEMICAL POLISHING SOLUTIONS<sup>1</sup>

Metal	Solution	Time	Temp.(°C)	Remarks
Aluminium and Alloys	25 ml sulphuric acid 70 ml orthophosphoric acid 5 ml nitric acid	30 sec. 2 min.	85	Very useful for studying alloys containing intermetallic compounds e.g. Al-Cu, Al-Fe and Al-Si alloys.
Beryllium	5 wt per cent sulphuric acid 75 wt per cent orthophosphoric acid 7 wt per cent chromic acid Remainder water	Several min.	49-50	Rate of metal removal is approx. 1 micron/min. Passive film formed may be removed by immersion in 10 percent sulphuric acid.
Cadmium	75 ml fuming nitric acid 25 ml water	5-10 sec.	20	Cycles of dipping for a few seconds followed immediately by washing in a rapid stream of water are used until a bright surface is obtained. A suitable etching procedure uses 70 ml glacial acetic acid, 30 ml fuming nitric acid for 30 sec. to 1 min. Can also be used for zinc.
Copper	33 ml nitric acid 33 ml orthophosphoric acid 33 ml glacial acetic acid	1-2 min.	60-70	Finish is better when copper oxide is absent. A modified solution namely, 20 ml nitric acid, 55 ml orthophosphoric acid, 25 ml glacial acetic acid, appears to give a better polish than the original.
Copper Alloys	30 ml nitric acid 10 ml hydrochloric acid 10 ml orthophosphoric acid 50 ml glacial acetic acid	1-2 min.	70-80	Specimen should be agitated.
Copper-zinc Alloys	80 ml fuming nitric acid 20 ml water	5 sec.	40	Use periods of 5 sec. immersion followed immediately by washing in a rapid stream of water. Slight variations in composition are needed for $\alpha$ - $\beta$ and $\beta$ - $\gamma$ brasses to prevent differential attack. With $\beta$ - $\gamma$ alloys, a dull film forms and this can be removed by immersion in a saturated solution of chromic acid in fuming nitric acid for a few seconds followed by washing.
Germanium	15 ml hydrofluoric acid 25 ml nitric acid 15 ml glacial acetic acid 3-4 drops bromine	5-10 sec.	20	
Iron	3 ml nitric acid 7 ml hydrofluoric acid (comm.) 30 ml water	2-3 min.	60-70	Dense brown viscous layer forms on surface; layer is soluble in solution. Low carbon steels can also be polished, but the cementite is attacked preferentially.

<sup>1</sup>Tegart, W. J., *The Electrolytic & Chemical Polishing of Metals*, Headington Hill Hall, Oxford, England: Pergamon Press Ltd. pages 99, 100, 101, 102 (Reprinted with permission)

TABLE VII — CHEMICAL POLISHING SOLUTIONS continued

Metal	Solution	Time	Temp.(°C)	Remarks
Iron and Steels	80 ml distilled water 28 ml oxalic acid (100 g/l) 4 ml hydrogen peroxide (30 per cent)	15 min.	35	The solution must be prepared freshly before use. Careful washing is necessary before treatment. A microstructure is obtained similar to that produced by mechanical polishing, followed by etching with Nital.
Lead	20 ml hydrogen peroxide (30 per cent) 80 ml glacial acetic acid	Periods of 5-10 sec.	20	McAFEE recommends using alternate immersions in this solution of 10 g molybdc acid and 140 ml ammonium hydroxide in 240 ml water to which 60 ml nitric acid is finally added.
Magnesium	75 ml fuming nitric acid 25 ml water	Periods of 3 sec.		The reaction reaches almost explosive violence after about a minute but if allowed to continue it ceases after several minutes leaving a polished surface ready for examination. Specimen should be washed immediately after removal from solution.
Nickel	30 ml nitric acid 10 ml sulphuric acid 10 ml orthophosphoric acid 50 ml glacial acetic acid	1/2-1 min.	85-95	This solution gives a very good polish.
Silicon	20 ml nitric acid 5 ml hydrofluoric acid (comm.)	5-10 sec.	20	
Tantalum	50 ml sulphuric acid 20 ml nitric acid 20 ml hydrofluoric acid (comm.)	5-10 sec.	20	Solution is useful for preparing surfaces prior to anodizing.
Zinc	200 g chromic acid 15 g sodium sulphate 50 ml nitric acid 950 ml water	Several min. to 1/2 hr.	20	Dense layer formed during treatment is soluble in water. Rate of dissolution is approximately 7 microns/min.
Zirconium	45 ml nitric acid 8-10 ml hydrofluoroic acid (comm.) 45 ml water or hydrogen peroxide (30 per cent)	5-10 sec.	20	Specimen is swabbed with solution. After a few seconds, a brownish - yellow vapour is evolved on the surface. Swabbing is continued for 5-10 sec. and then specimen is rinsed in running water. Similar solution can be used for Titanium and Hafnium.

E



<b>Microetching</b>	<b>Page</b>
Definition .....	35
Standard Methods for Microetching Metals and Alloys (ASTM Designation: E 407-70)	
1. Scope .....	35
2. Summary .....	35
3. Safety Precautions .....	35, 36
4. Miscellaneous Information .....	36
Table 1 - Etchants for Metals and Alloys .....	36, 37, 38, 39, 40
Table 2 - Numerical List of Etchants .....	40, 41, 42, 43
	44, 45, 46, 47
	48, 49, 50, 51
Table 3 — Etchant Names .....	51

#### **Metal Progress Data Book**

Table 4 - Etching Reagents for Iron and Steel .....	52
I General Reagents for Iron and Steel .....	52
II General Reagents for Alloy Steels .....	52, 53, 54
III Miscellaneous Reagents .....	54, 55
IV Reagents for Non-metallic Inclusions and Intermetallic Compounds.....	55
V Reagents for Macroscopic Examination .....	56, 57
VI Electrolytes for Polishing and Etching .....	57

# 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<sup>2</sup>

### 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 under-

lined 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 gear (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.

<sup>2</sup>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
<i>Aluminum base:</i>		
Pure Al	1a, 2, 3 <u>4</u> , <u>5</u> 1b	general structure grain structure under polarized light grain boundaries and slip lines
1000 series	1a, 3, 2 <u>4</u> , <u>5</u> 6, 7	general structure grain structure under polarized light phase identifications
2000 series	3, 2, 1a 8a, 6, 7	general structure phase identifications
3000 series	3, 1a <u>4</u> , <u>5</u> 8a, 6, 7	general structure grain structure under polarized light phase identifications
4000 series	3, 1a	general structure
5000 series	3, 1a, 2, 6, 8a	general structure
6000 series	<u>4</u> , <u>5</u> 3, 1a, 2, 6, 8a <u>4</u> , <u>5</u>	grain structure under polarized light general structure grain structure under polarized light
7000 series	1a, 2, 7, 6, 8a 3, 1a, 2 <u>4</u> , <u>5</u> 3b, 6	phase identifications general structure grain structure under polarized light 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
<i>Beryllium base:</i>		
Pure Be	9, 10	general structure via polarized light
Be alloys	11	general structure
<i>Chromium base:</i>	12, 13c	general structure
<i>Cobalt base:</i>		
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
<i>Columbium base (see niobium base)</i>		
<i>Copper base:</i>		
Pure Cu	26, 27, 28, 29, 30, 31d, 32, 33	general structure
	34b, 35, 36, 37, 38, 39, 40, 41,	
	42, 8b	
	43, 28	chemical polish and etch
Cu-Al (Aluminum bronze)	44, 31d, 34b, 35, 36, 37, 38, 39,	general structure
	40, 45, 28	
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,	general structure
	32, 33, 34b, 35, 36, 37, 38, 39	
	53, 43, 28, 49	chemical polish and etch
	42, 49	darkens beta in alpha-beta brass
	54	etching of cold worked brass
<i>Dysprosium base:</i>	55, 56	general structure
<i>Erbium base:</i>	55, 56	general structure
<i>Gadolinium base:</i>	55, 56, 57	general structure
<i>Germanium base:</i>	58, 59, 60	general structure
<i>Gold base:</i>		
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
<i>Hafnium base:</i>	66, 67, 68, 69, 70	general structure
	71	grain structure under polarized light
	72	chemical polish and etch
<i>Holmium base:</i>	55, 56	general structure
<i>Iridium base:</i>	73c	general structure
<i>Iron base:</i>		
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, <u>93</u> , 94, 95, 91 96, 97, 98 <u>31c</u> 86	general structure  sigma phase carbides chemical polish — etch
Fe + 12-20 Cr + 4-10 Ni + < 7% other elements (controlled transformation, precipita- tion hardening, stainless maraging alloys)	80, <u>31c</u> , 89, 99, 100, 91 <u>31c</u> 86	general structure carbides chemical polish — etch
Fe + 15-30 Cr + 6-40 Ni + < 5% other elements (300 Series)	<u>13b</u> , 89, 87, 88, <u>83a</u> , 80, 94, 95, 91, 101 <u>13a</u> , <u>102</u> 48, <u>96</u> , <u>97</u> , 98	general structure  carbides and sensitization stains sigma phase
and Fe + 16-25 Cr + 3-6 Ni + 5-10 Mn (200 Series)	103, 104, 98 103, 104 86	delineates sigma phase and ferrite welds of dissimilar metals chemical polish — etch
High temperature	89, 25, 105, 106, <u>97</u> <u>107</u> , 108 86	general structure $\gamma$ precipitate chemical polish — etch
Nonstainless maraging steels	109, 89, 99, 100 83b 86	general structure grain boundaries chemical polish — etch
Tool steels	74a, 80, 14 110 86	general structure grain boundaries in tempered tool steel chemical polish — etch
Superalloys	<u>111</u> <u>111</u>	general structure $\gamma$ depletion
<i>Lead base:</i>		
Pure Pb	57, 112 113	general structure for alternate polishing and etching
Pb + < 2 Sb	114, 115, 57, 74b 113	general structure for alternate polishing and etching
Pb + > 2 Sb	114, 57, 74b 113	general structure for alternate polishing and etching
Pb + Ca	112 113	general structure for alternate polishing and etching
Pb alloys	116, 117b	general structure
Babbitt	74b	general structure
<i>Magnesium base:</i>		
Pure Mg	118, 119, 74a, 120, 121, 122 123	general structure 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, <u>123</u> , 122 120, 125, 126, 127 124, 126, 127	general structure phase identification grain structure
Mg-Al, Mg-Al-Zn (Al + Zn > 5%)	118, 119, 74a, 125, 124, 121, 122 120, 125, 126, 127	general structure phase identification
Mg-Zn-Zr and Mg-Zn-Th-Zr	118, 119, 74a, 1d, 128, 124, 126 127, 121, 122 120, 121	general structure  phase identification
Mg-Th-Zr and Mg-Rare Earth-Zr	118, 119, 74a, 1d, 124, 127, 121, 122 120, 121	general structure  phase identification
<i>Molybdenum base:</i>		
As cast	98c, 129, <u>130</u> , <u>131</u> 132a	general structure chemical polish prior to etching
<i>Nickel base:</i>		
Pure Ni and high Ni alloys	133, <u>134</u> , 47, 135, <u>136</u> , 25, <u>108</u> <u>31c</u> 137	general structure  grain boundary sulfidation
Ni-Ag	38, 138, 50, 139	general structure
Ni-Al	50, 140, 141, <u>142</u> , 89, 143	general structure
Ni-Cr	<u>144</u> , 50, <u>83</u> , <u>134</u> , <u>145</u> , 98, 146, 147, <u>13a</u>	general structure
Ni-Cu	38, 138, <u>50</u> , <u>133</u> , 140, 25, <u>134</u> , 47, 48b, 94, <u>108</u> , 34	general structure
Ni-Fe	<u>50</u> , 140, 141, <u>83</u> , <u>134</u> , 148, 40, 107, 149 74e, 25, 150	general structure  orientation pitting

TABLE I — continued

Metal	Etchants	Uses
Ni-Mn	74e	general structure
Ni-Mo	143	general structure
Ni-Ti	143, 151, <u>50</u> , 133	general structure
Ni-Zn	152	general structure
Superalloys	94, 105, 138, 153, 12	general structure
	25	grain size
	<u>107</u> , <u>111</u> , <u>13a</u>	reveals microstructural inhomogeneity
	133	grain boundary sulfidation
	154	fine precipitation structure
	<u>19b</u> , 155, <u>156</u>	differential matrix and nonmetallic staining
	22a	for passive alloys (for example Inconel 625)
	157	specific for Hastelloy W
	<u>107</u>	submicroscopic structure in aged superalloys particularly for electron microscopy. Stains the matrix when $\gamma'$ precipitates are present
	154	$\gamma'$ banding
	18	pre-etch activation for passive samples
<i>Niobium (Columbium) base:</i>	129, 66, 158, 159, 160, 161, 162, 163	general structure
	164, 129, 160	grain boundaries
<i>Osmium base:</i>	<u>165a</u>	general structure
	<u>165a</u>	etch — polishing for viewing grains with polarized light
<i>Palladium base:</i>		
Pure Pd	61, 166, 62, <u>165a</u>	general structure
Pd alloys	166, 64a, 62, <u>165a</u>	general structure
> 90% noble metals	61	general structure
< 90% noble metals	65	general structure
<i>Platinum base:</i>		
Pure Pt	64a, <u>73a</u>	general structure
	<u>167</u>	electrolytic polish and etch
Pt alloys	64b, <u>73a</u>	general structure
	<u>167</u>	electrolytic polish and etch
> 90% noble metals	61	general structure
< 90% noble metals	65	general structure
Pt-10% Rh	<u>168</u>	general structure
<i>Plutonium base:</i>	<u>169</u>	general structure
<i>Rhenium base:</i>	<u>13b</u> , 98c, 132b, 170a	general structure
<i>Rhodium base:</i>	<u>171</u>	general structure
<i>Ruthenium base:</i>	<u>73b</u>	general structure
	<u>73b</u>	etch — polishing for viewing grains with polarized light
<i>Silver base:</i>		
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
<i>Tantalum base:</i>		
Pure Ta	177	general structure
Ta alloys	159, 66, 178, 163, 161, <u>179</u>	general structure
	164	grain boundaries and inclusions
	158	grain boundaries — retains carbide precipitate
<i>Thorium base:</i>		
Pure Th	185	general structure
Th alloys	185	general structure
<i>Tin base:</i>		
Pure Sn	74d, 180, 51	general structure
	181	grain boundaries
Sn-Cd	74d	general structure
Sn-Fe	74d, <u>117a</u>	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
<i>Titanium base:</i>		
Pure Ti	186, 187, <u>67</u> , <u>68</u> , <u>69</u> 188 72	general structure removes stain 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, <u>67</u> , <u>68</u> , <u>69</u> , 3a 11, 1c 72, 192, 178 170a	general structure reveals alpha case chemical polish and etch outlines and darkens hydride in some alloys removes stain
<i>Tungsten base:</i>		
Pure W	98c, <u>131</u>	general structure
As cast	132a	chemical polish prior to etching
W-Th	209	general structure
<i>Uranium base:</i>		
Pure U	<u>67</u> , <u>69</u> , <u>195</u> , <u>196</u>	general structure
U + Zr	<u>68</u>	general structure
U beryllides	170a	general structure
U alloys	<u>67</u> , <u>69</u> , <u>195</u> , <u>196</u> 207	general structure carbides
<i>Vandaium base:</i>		
Pure V	170b, <u>165b</u> <u>197</u> , <u>198</u>	general structure grain boundaries
V alloys	<u>199</u> , <u>198</u>	general structure
<i>Zinc base:</i>		
Pure Zn	200a	general structure
Zn-Co	177	general structure
Zn-Cu	201 <u>203</u>	general structure distinguishes gamma and epsilon
Zn-Fe	74a	structure of galvanized sheet
Die castings	202	general structure
<i>Zirconium base:</i>		
	<u>66</u> , <u>67</u> , <u>204</u> , <u>68</u> , <u>69</u> , <u>205</u> <u>206</u> 71 72	general structure electrolytic polish and etch grain structure under polarized light 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 <sub>3</sub> 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 — <i>Mix fresh.</i>
4	25 ml H <sub>3</sub> PO <sub>4</sub> 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.

TABLE 2 — continued

Etchant	Composition	Procedure
5	5 g HBF <sub>4</sub> 200 ml water	Electrolytic: Use Al, Pb. or stainless steel cathode. Anodize 1 to 3 minutes, 20 to 45 V d-c. At 30 V, etch for 1 minute.
6	25 ml HNO <sub>3</sub> 75 ml water	Immerse 40 seconds at 70 C (160 F). Rinse in cold water.
7	10-20- ml H <sub>2</sub> SO <sub>4</sub> 80 ml water	Immerse 30 seconds at 70 C (160 F). Rinse in cold water.
8	10 ml H <sub>3</sub> PO <sub>4</sub> 90 ml water	(a) Immerse 1 to 3 minutes at 50 C (120 F). (b) Electrolytic at 1 to 8 V for 5 to 10 seconds.
9	3-4 g sulfamic acid 5 drops HF 100 ml water	Use just prior to the last polishing operation. It is not intended as a final etchant. The sample is examined as polished under polarized light.
10	10 ml HF 90 ml methanol (95%)	Immerse 10 to 30 seconds.
11	2 ml HF 100 ml water	Immerse or swab few seconds to a minute.
12	20 ml HNO <sub>3</sub> 60 ml HCl	<b>Use Hood. Do not Store.</b> Immerse or swab 5 to 60 seconds.
13	10 g oxalic acid 100 ml water	Electrolytic at 6 V: (a) 10 to 15 seconds. (b) 1 minute. (c) 2 to 3 seconds. Use stainless steel cathode and platinum or Nichrome connection to specimen. Immerse few seconds to a minute.
14	10 ml HNO <sub>3</sub> 90 ml methanol (95%)	
15	15 ml HNO <sub>3</sub> 15 ml acetic acid 60 ml HCl 15 ml water	Age before use. Immerse 5 to 30 seconds. May be used electrolytically.
16	5-10 ml HCl 100 ml water	Electrolytic at 3 V for 2 to 10 seconds.
17	5 ml HCl 10 g FeCl <sub>3</sub> 100 ml water	Electrolytic at 6 V for few seconds.
18	2-10 g CrO <sub>3</sub> 100 ml water	Electrolytic at 3 V for 2 to 10 seconds.
19	A 2 g CrO <sub>3</sub> 100 ml water B 4 g NaOH 10 g KMnO <sub>4</sub> 85 ml water	Two step process: Electrolytic at 3 V for 2 seconds in Solution A, then rinse in water. While still wet, immerse in Solution B for 5 to 10 seconds (use fresh).
20	5 ml H <sub>2</sub> O <sub>2</sub> (30%) 100 ml HCl	<b>Use Hood. Mix fresh.</b> Immerse polished face up for few seconds.
21	1 g CrO <sub>3</sub> 140 ml HCl	<b>Use Hood.</b> To mix, add the HCl to CrO <sub>3</sub> . Electrolytic at 3 V for 2 to 10 seconds.
22	100 ml HCl 0.5 ml H <sub>2</sub> O <sub>2</sub> (30%)	<b>Use Hood. Do not Store.</b> (a) Immerse or swab 1/2 to 3 minutes Add H <sub>2</sub> O <sub>2</sub> dropwise to maintain action. (b) Electrolytic, 4 V, 3 to 5 seconds.
23	5 ml HCl 95 ml ethanol (95%) or methanol (95%)	Electrolytic at 6 V for 10 to 20 seconds.
24	5 ml HNO <sub>3</sub> 200 ml HCl	<b>Use Hood.</b> Immerse few seconds.
25	65 g FeCl <sub>3</sub> 10 g CuSO <sub>4</sub> 50 ml HCl 50 ml water	Immerse or swab 5 to 60 sec. Made more active by adding few drops of H <sub>2</sub> SO <sub>4</sub> just before use.
26	5 g FeCl <sub>3</sub> 10 ml HCl 50 ml glycerin 30 ml water	Swab 16 to 60 sec. Activity may be decreased by substituting glycerin for water.
27	1 g KOH 20 ml H <sub>2</sub> O <sub>2</sub> (3%) 50 ml NH <sub>4</sub> OH 30 ml water	Dissolve KOH in water, then slowly add NH <sub>4</sub> OH to solution. Add 3% H <sub>2</sub> O <sub>2</sub> last. Use fresh — immerse few seconds to a minute.
28	1 g FeNO <sub>3</sub> 100 ml water	Swab or immerse few seconds to a minute.



TABLE 2 — continued

Etchant	Composition	Procedure
29	1 g $K_2Cr_2O_7$ 4 ml $H_2SO_4$ 50 ml water	Add 2 drops of HCl just before using. Swab few seconds to a minute.
30	25 ml $NH_4OH$ 25 ml water 50 ml $H_2O_2$ (3%)	Mix $NH_4OH$ and water before adding $H_2O_2$ . Must be used fresh. Swab 5 to 45 seconds.
31	10 g ammonium persulfate 100 ml water	(a) Swab or immerse to 5 seconds. (b) Immerse to 2 minutes to darken matrix to reveal carbides and phosphides. (c) Electrolytic at 6 V for few seconds to a minute. (d) Immerse 3 to 60 seconds. Can be heated to increase activity.
32	60 g $CrO_3$ 100 ml water	Saturated solution. Immerse or swab 5 to 30 seconds.
33	10 g $CrO_3$ 2-4 drops HCl 100 ml water	Add HCl just before use. Immerse 3 to 30 seconds Phases can be colored by Nos. 35, 36, 37.
34	5 g $FeCl_3$ 50 ml HCl 100 ml water	(a) Immerse or swab few seconds to few minutes. Small additions of $HNO_3$ activate solution and minimize pitting.  (b) Immerse or swab few seconds at a time. Repeat as necessary.
35	20 g $FeCl_3$ 5 ml HCl 1 g $CrO_3$ 100 ml water	Immerse or swab few seconds at a time until desired results are obtained.
36	25 g $FeCl_3$ 25 ml HCl 100 ml water	Immerse or swab few seconds at a time until desired results are obtained.
37	1 g $FeCl_3$ 10 ml HCl 100 ml water	Immerse or swab few seconds at a time until desired results are obtained.
38	8 g $FeCl_3$ 25 ml HCl 100 ml water	Swab 5 to 30 seconds.
39	5 g $FeCl_3$ 10 ml HCl 1 g $CuCl_2$ 0.1 g $SnCl_2$ 100 ml water	Immerse or swab few seconds at a time until desired results are obtained.
40	5 g $FeCl_3$ 15 ml HCl 60 ml ethanol (95%) or methanol (95%)	Immerse or swab few seconds to few minutes.
41	2 g $K_2Cr_2O_7$ 8 ml $H_2SO_4$ 4 drops HCl 100 ml water	Add the HCl just before using. Immerse 3 to 60 seconds.
42	10 g ammonium cupric chloride 100 ml water $NH_4OH$	Add $NH_4OH$ to solution until neutral or slightly alkaline. Immerse 5 to 60 seconds.
43	20 ml $NH_4OH$ 1 g ammonium persulfate 60 ml water	Immerse 5 to 30 seconds.
44	50 ml $NH_4OH$ 20-50 ml $H_2O_2$ (3%) 0-50 ml water	Use fresh. Peroxide content varies directly with copper content of alloy to be etched. Immerse or swab to 1 minute. Film on etched aluminum bronze removed by No. 82.
45	1 g $CrO_3$ 100 ml water	Electrolytic at 6 V for 3 to 6 seconds. Use aluminum cathode.
46	15 ml $NH_4OH$ 15 ml $H_2O_2$ (3%) 15 ml water 4 pellets NaOH	When mixing, add NaOH pellets last. For best results use before pellets have dissolved.
47	5 g NaCN or KCN 5 g $(NH_4)_2S_2O_8$ 100 ml water	Use Hood. — Can give off extremely poisonous hydrogen cyanide. <b>Precaution</b> — Also poisonous by ingestion as well as contact. To discard, neutralize (or turn basic) with ammonia and flush down acid drain with copious amount of water.

TABLE 2 — continued

Etchant	Composition	Procedure
48	10 g NaCN 100 ml water	<b>Use Hood.</b> — Can give off extremely poisonous hydrogen cyanide. <b>Precaution</b> — Also poisonous by ingestion as well as contact. To discard, neutralize (or turn basic) with ammonia and flush down acid drain with copious amount of water. Electrolytic at 6 V: (a) 5 seconds for sigma. (b) 30 seconds for ferrite and general structure. (c) to 5 minutes for carbides Electrolytic at 8-10 V (0.1 A) for 5 to 15 seconds.
49	3 g FeSO <sub>4</sub> 0.4 g NaOH 10 ml H <sub>2</sub> SO <sub>4</sub> 190 ml water	
50	5 ml acetic acid 10 ml HNO <sub>3</sub> 85 ml water	<b>Use Hood. Do not Store.</b> Electrolytic at 1.5 V for 20 to 60 seconds. Use platinum wires.
51	2 g FeCl <sub>3</sub> 5 ml HCl 30 ml water 60 ml ethanol or methanol	Immerse few minutes.
52	1 g sodium dichromate 1 g NaCl 4 ml H <sub>2</sub> SO <sub>4</sub> 250 ml water	Swab few seconds.
53	1-5 ml NH <sub>4</sub> OH 100 ml water	Immerse 5 to 60 seconds.
54	1 g ammonium acetate 3 g sodium thiosulfate 7 ml NH <sub>4</sub> OH 1300 ml water	Electrolytic at 0.3 A/cm <sup>2</sup> for 5 to 30 seconds.
55	1 ml H <sub>2</sub> SO <sub>4</sub> 15 ml NHO <sub>3</sub> 10 ml acetic acid 5 ml H <sub>3</sub> PO <sub>4</sub> 20 ml lactic acid	Swab gently 10 to 15 seconds. Rinse with methanol and blow dry. Helps to chemically polish. If final etch is too mild, follow with No. 98.
56	30 ml NHO <sub>3</sub> 10 ml H <sub>3</sub> PO <sub>4</sub> 20 ml acetic acid 10 ml lactic acid	Swab gently 5 to 15 sec. Rinse with methanol or ethanol and blow dry.
57	75 ml acetic acid	Immerse 6 to 15 seconds.
58	25 ml H <sub>2</sub> O <sub>2</sub> (30%) 25 ml HF 25 ml HNO <sub>3</sub> 5 ml water	Swab 3 to 20 seconds.
59	2 AgNO <sub>3</sub> 40 ml water 40 ml HF 20 ml HNO <sub>3</sub>	Mix AgNO <sub>3</sub> and water, then add HF and HNO <sub>3</sub> . Swab 1/2 to 2 minutes.
60	25 ml HNO <sub>3</sub> 15 ml acetic acid 15 ml HF 5-7 drops bromine	<b>Use Hood.</b> Let stand 1/2 hour before using. Swab 3 to 20 seconds.
61	60 ml HCl 40 ml HNO <sub>3</sub>	<b>Use Hood.</b> Immerse few seconds to a minute.
62	1-5 g CrO <sub>3</sub> 100 ml HCl	Vary composition of reagent and aging of reagent after mixing to suit alloy. Swab or immerse few seconds to a minute.
63	0.1 g CrO <sub>3</sub> 10 ml HNO <sub>3</sub> 100 ml HCl	Swab few seconds to a minute.
64	5 ml HNO <sub>3</sub> 25 ml HCl 30 ml water	(a) Immerse 1 to 5 minutes. (b) Use hot. Will form chloride film on gold alloys if much silver is present. Ammonia will remove film.
65	A 10 g ammonium persulfate 100 ml water B 10 g KCN 100 ml water	<b>Use Hood.</b> Mix 1 + 1 mixture of Solutions A and B just before use. (A mixture of 5 drops of each will cover the surface of a 1 inch dia. mount.) Immerse 1/2 to 2 minutes.
66	30 ml HF 15 ml HNO <sub>3</sub> 30 ml HCl	Swab 3 to 10 sec. or immerse to 2 minutes.

TABLE 2 — continued

Etchant	Composition	Procedure
67	10 ml perchloric acid 10 ml 2-butoxyethanol 70 ml ethanol (95%) 10 ml water	<b>Precaution.</b> Keep cool when mixing. Electrolytic at 30 to 65 V for 10 to 60 seconds.
68	3 ml perchloric acid 35 ml 2-butoxyethanol 60 ml methanol (absolute)	Electrolytic at 60-150 V for 5 to 30 seconds.
69	5 ml perchloric acid 80 ml acetic acid	Electrolytic at 20-60 V for 1 to 5 minutes.
70	5 ml HF 2 ml AgNO <sub>3</sub> (5%) 200 ml water	Swab for 5 to 60 seconds.
71	5 ml HF 95 ml water	Add 5 to 10 drops of this solution to the cloth on the final polishing wheel which has been charged with the polishing solution. The specimen is polished on this wheel until the surface turns black. Distilled water is then slowly added to the wheel and polishing continued until the surface is bright. At this time the specimen should be ready for examination via polarized light. Note — Use inert substance between cloth and wheel to prevent attack of the wheel.
72	10 ml HF 45 ml HNO <sub>3</sub> 45 ml water	Swab for 5 to 20 seconds.
73	20 ml HCl 25 g NaCl 65 ml water	Electrolytic etch — use carbon cathode and platinum wire connection to specimen. (a) 6 V ac for 1 minute. (b) 5 V to 20 V ac for 1 to 2 minutes. (c) 20 V ac for 1 to 2 minutes. For etch-polishing, use shorter times. After etching, water rinse, alcohol rinse, and dry.
74	1-5 ml HNO <sub>3</sub> 100 ml ethanol (95%) or methanol (95%)	Etching rate is increased, selectivity decreased with increased percentage of HNO <sub>3</sub> . (a) Immerse few seconds to a minute. (b) Immerse 5 to 40 seconds in 5% HNO <sub>3</sub> solution. To remove stain, immerse 25 seconds in 10% HCl-methanol solution. (c) For Inconels and Nimonics, use 5 ml HNO <sub>3</sub> solution-electrolytic at 5 to 10 V for 5 to 20 seconds. (d) Swab or immerse several minutes. (e) Swab 5 to 60 sec. HNO <sub>3</sub> may be increased to 30 ml in <i>methanol only</i> depending on alloy. (Ethanol is unstable with over 5% HNO <sub>3</sub> ). <b>Do not Store.</b>
75	5 g picric acid 8 g CuCl <sub>2</sub> 20 ml HCl 6 ml HNO <sub>3</sub> 20 ml ethanol (95%) or methanol (95%)	Immerse 1 to 2 sec. at a time and immediately rinse with methanol. Repeat as often as necessary. (Long immersion times will result in copper deposition on surface.)
76	10 g picric acid 100 ml ethanol (95%) or methanol (95%)	Composition given will saturate the solution with picric acid. Immerse few seconds to a minute or more.
77	10 g picric acid 5 drops HCl 100 ml ethanol (95%) or methanol (95%)	Composition given will saturate the solution with picric acid. Immerse few seconds to a minute or more.
78	10 g potassium metabisulfite 100 ml water	Immerse 1 to 15 seconds. Better results are sometimes obtained by first etching lightly with No. 76 or 74.
79	40 ml HCl 5 g CuCl <sub>2</sub> 30 ml water 25 ml ethanol (95%) or methanol (95%)	Swab few seconds to a minute.
80	5 ml HCl 1 g picric acid 100 ml ethanol (95%) or methanol (95%)	Immerse or swab few seconds to 15 minutes. Reaction may be accelerated by adding a few drops of 3% H <sub>2</sub> O <sub>2</sub> . Optional (for prior austenite grain boundaries) — temper specimen at 600-900 F prior to preparation.
81	2 g picric acid 1 g sodium tridecylbenzene sulfonate 100 ml water	Composition given will saturate the solution with picric acid. (a) Immerse few seconds to a minute. (b) Immerse to 15 minutes with occasional swabbing for heavy grain boundary attack.

TABLE 2 — continued

Etchant	Composition	Procedure
82	5 g FeCl <sub>3</sub> 5 drops HCl 100 ml water	Immerse 5 to 10 seconds.
83	10 g CrO <sub>3</sub> 100 ml water	(a) Electrolytic at 6 V for 5 to 60 seconds. Attacks carbides. (b) Electrolytic at 6 V for 3 to 5 seconds.
84	10 ml H <sub>2</sub> SO <sub>4</sub> 10 ml HNO <sub>3</sub> 80 ml water	<b>Precaution</b> — Add H <sub>2</sub> SO <sub>4</sub> slowly to water and cool, then add HNO <sub>3</sub> . Immerse 30 seconds. Swab in running water. Repeat three times and repolish lightly.
85	2 g picric acid 25 g NaOH 100 ml water	Immerse in boiling solution for 5 minutes. <b>Precaution</b> — Do not boil dry — anhydrous picric acid is unstable and highly explosive. Alternative: Electrolytic at 6 V for 40 seconds (room temperature). Use stainless steel cathode.
86	3 g oxalic acid 4 ml H <sub>2</sub> O <sub>2</sub> (30%) 100 ml water	Solution should be freshly prepared. Immerse 15 to 25 min. when samples or parts cannot be given usual metallographic polish. Multiple etching may be required.
87	10 ml HNO <sub>3</sub> 20-50 ml HCl 30 ml glycerin	<b>Use Hood.</b> — Can give off nitrogen dioxide gas. <b>Precaution</b> — Mix HCl and glycerin thoroughly before adding HNO <sub>3</sub> . <b>Do not Store.</b> Discard before solution attains a dark orange color. Immerse or swab few seconds to few minutes. Higher percentage of HCl minimizes pitting. A hot water rinse just prior to etching may be used to activate the reaction. Sometimes a few passes on the final polishing wheel is also necessary to remove a passive surface.
88	10 ml HNO <sub>3</sub> 20 ml HCl 30 ml water	<b>Use Hood.</b> — Can give off nitrogen dioxide gas. <b>Precaution</b> — Discard before solution attains a dark orange color. Immerse few seconds to a minute. Much stronger reaction than No. 87.
89	10 ml HNO <sub>3</sub> 10 ml acetic acid 15 ml HCl 2-5 drops glycerin	<b>Use Hood. Do not Store.</b> Immerse or swab few seconds to few minutes.
90	10 ml HNO <sub>3</sub> 20 ml HF 20-40 ml glycerin	Immerse 2 to 10 seconds.
91	5 ml HNO <sub>3</sub> 5 ml HCl 1 g picric acid 200 ml ethanol (95%) or methanol (95%)	This etchant is equivalent to a 1 + 1 mixture of No. 80 and No. 74 (5% HNO <sub>3</sub> ). Swab for 30 seconds or longer.
92	10 ml HCl 100 ml ethanol (95%) or methanol (95%)	Immerse 5 to 30 minutes or electrolytic at 6 V for 3 to 5 seconds.
93	concentrated HNO <sub>3</sub>	<b>Use Hood.</b> Electrolytic at 0.2 A/cm <sup>2</sup> for few seconds.
94	2 g CuCl <sub>2</sub> 40 ml HCl 40-80 ml ethanol (95%) or methanol (95%)	Submerged swabbing for few seconds to several minutes. Attacks ferrite more readily than austenite.
95	2 g CuCl <sub>2</sub> 40 ml HCl 40-80 ml ethanol (95%) or methanol (95%) 40 ml water	Immerse or swab few seconds to few minutes.
96	85 g NaOH 50 ml water	Electrolytic at 6 V for 5 to 10 seconds.
97	45 g KOH 60 ml water	Composition of solution is approximately 10 normal. Electrolytic at 2.5 V for few seconds. Stains sigma and chi yellow to red brown, ferrite gray to blue gray, carbides barely touched, austenite not touched.
98	10 g K <sub>3</sub> Fe(CN) <sub>6</sub> 10 g KOH or NaOH 100 ml water	<b>Use Hood.</b> — Can give off extremely poisonous hydrogen cyanide. <b>Precaution</b> — Also poisonous by ingestion as well as contact. To discard, neutralize (or turn basic) with ammonia and flush down acid drain with copious amount of water. Use fresh. (a) Immerse or swab 15 to 60 seconds. Stains carbides and sigma. (To differentiate, No. 31 electrolytic at 4 V will attack sigma but not carbides. If pitting occurs, reduce voltage.) (b) Immerse in fresh, hot solution 2 to 20 minutes. Stains carbides dark, ferrite yellow, sigma blue. Austenite turns brown on overetching. (c) Swab 5 to 60 seconds. (Immersion will produce a stain etch). Follow with water rinse, alcohol rinse, dry.

TABLE 2 — continued

Etchant	Composition	Procedure
99	25 ml HCl 3 g ammonium bifluoride 125 ml water few grains potassium metabisulfite.	<i>Mix fresh.</i> (For stock solution, mix first three items. Add potassium metabisulfite just before use.) Immerse few seconds to a few minutes.
100	10 g FeCl <sub>3</sub> 90 ml water	Immerse few seconds.
101	2 g CrO <sub>3</sub> 20 ml HCl 80 ml water	Immerse 5 to 60 seconds. (CrO <sub>3</sub> may be increased up to 20 g for difficult alloys. Staining and pitting increase as CrO <sub>3</sub> is increased.)
102	concentrated NH <sub>4</sub> OH	Electrolytic at 6 V for 30 to 60 seconds. Attacks carbides only.
103	20 ml HNO <sub>3</sub> 4 ml HCl 20 ml methanol (99%)	Immerse 10 to 60 seconds.
104	5 ml HNO <sub>3</sub> 45 ml HCl 50 ml water	Immerse 10 minutes or longer.
105	5 ml H <sub>2</sub> SO <sub>4</sub> 3 ml HNO <sub>3</sub> 90 ml HCl	<b>Use Hood. Precaution</b> — Add H <sub>2</sub> SO <sub>4</sub> slowly to HCl with stirring, cool, then add HNO <sub>3</sub> . Discard when dark orange color. Swab 10 to 30 seconds.
106	7 ml HNO <sub>3</sub> 25 ml HCl 10 ml methanol (99%)	Use fresh to avoid pitting. Immerse or swab 10 to 60 seconds.
107	10 ml H <sub>3</sub> PO <sub>4</sub> 50 ml H <sub>2</sub> SO <sub>4</sub> 40 ml HNO <sub>3</sub>	<b>Use Hood. Precaution</b> — Mix H <sub>3</sub> PO <sub>4</sub> and HNO <sub>3</sub> thoroughly, then add H <sub>2</sub> SO <sub>4</sub> slowly with stirring. Use fresh, but allow to cool. Electrolytic at 6 V for few seconds. Brown discoloration will form at edges of specimen. To slow reaction, add water (to 100 ml) very carefully with stirring. Attacks bakelite mounts.
108	3-10 ml H <sub>2</sub> SO <sub>4</sub> 100 ml water	Electrolytic at 6 V for 5 to 10 sec. Tends to pit with longer times.
109	50 ml HCl 25 ml HNO <sub>3</sub> 1 g CuCl <sub>2</sub> 150 ml water	Make fresh but allow to stand 30 min. to avoid plating out copper. Immerse few seconds to a few minutes.
110	10 ml HCl 5 ml HNO <sub>3</sub> 85 ml ethanol (95%) or methanol (95%)	Immerse to several minutes until deeply etched. Follow with light repolish.
111	5 ml H <sub>2</sub> SO <sub>4</sub> 8 g CrO <sub>3</sub> 85 ml H <sub>3</sub> PO <sub>4</sub>	Electrolytic at 10 V (0.2 A/cm <sup>2</sup> ) for 5 to 30 sec. Reveals Ti & Cb-rich areas at a faster rate than grain boundaries.
112	60 ml acetic acid 30 ml H <sub>2</sub> O <sub>2</sub> (30%)	Immerse 8 to 15 seconds.
113	15 ml acetic acid 15 ml HNO <sub>3</sub> 60 ml glycerin	Use fresh solution at 80 C (176 F).
114	15 ml acetic acid 20 ml HNO <sub>3</sub> 80 ml water	Use fresh solution at 40 to 42 C (104 to 108 F). Immerse 4 to 30 minutes depending on depth of worked metal layer. Clean with cotton in running water.
115	100 ml acetic acid 10 ml H <sub>2</sub> O <sub>2</sub> (30%)	Immerse 10 to 30 minutes depending on depth of worked metal layer. Clean in HNO <sub>3</sub> if necessary.
116	5-10 g AgNO <sub>3</sub> 90 ml water	Swab.
117	10 ml HCl	(a) Immerse for 1/2 to 5 minutes. Follow with electrolytic etch at low current density in same solution. If sample has considerable surface flow, immerse in concentrated HCl for a few seconds, then follow above procedure. (b) Immerse for 1 to 2 minutes. Swab 3 to 5 seconds for F and T6, 10 seconds for T4 and 0 temper.
118	1 ml HNO <sub>3</sub> 75 ml diethylene glycol 25 ml water	Swab 1 to 3 seconds for F and T6, 10 seconds for T4 and 0 temper.
119	1 ml HNO <sub>3</sub> 20 ml acetic acid 60 ml diethylene glycol 20 ml water	Swab 1 to 3 seconds for F and T6, 10 seconds for T4 and 0 temper.
120	10 ml HF 90 ml water	Immerse with gentle agitation 3 to 30 seconds.

TABLE 2 — continued

Etchant	Composition	Procedure
121	0.7 ml H <sub>3</sub> PO <sub>4</sub> 4 g picric acid 100 ml methanol (95%) or ethanol (95%)	Composition critical. (a) Immerse with gentle agitation 10 to 30 seconds. (b) To increase staining immerse and withdraw with a meniscus layer. Tease etchant over surface until dark stain develops.
122	2 g oxalic acid 100 ml water	Swab.
123	60 ml H <sub>3</sub> PO <sub>4</sub> 100 ml ethanol (95%)	Electrolytic: Use stainless steel cathode. Space electrodes 2 cm apart. Start at 3 V dc. After 30 sec. maintain at 1½ V.
124	5 ml acetic acid 10 ml water 6 g picric acid 100 ml methanol (95%) or ethanol (95%)	Immerse with gentle agitation 10 to 60 seconds.
125	10 ml acetic acid 6 g picric acid 100 ml methanol (95%) or ethanol (95%)	Immerse with gentle agitation 15 to 30 seconds.
126	30 ml acetic acid 15 ml water 6 g picric acid 100 ml methanol (95%) or ethanol (95%)	Immerse with gentle agitation 1 to 30 seconds.
127	20 ml acetic acid 20 ml water 3 g picric acid 50 ml methanol (95%) or ethanol (95%)	Immerse with gentle agitation 5 to 30 seconds.
128	8 ml HF 5 ml HNO <sub>3</sub> 200 ml water	Immerse with gentle agitation 5 to 15 seconds.
129	10 ml HF 30 ml HNO <sub>3</sub> 60 ml lactic acid	Swab 10 to 20 seconds. Vary HF to increase or decrease activity.
130	25 ml HCl 10 ml H <sub>2</sub> SO <sub>4</sub> 75 ml methanol	<b>Caution</b> — Keep below 24 C (75F). Electrolytic at 30 V for 30 seconds.
131	5 ml H <sub>2</sub> SO <sub>4</sub> 1 ml HF 100 ml methanol (95%)	Electrolytic at 50 to 60 V for 10 to 20 seconds.
132	5 ml HF 10 ml HNO <sub>3</sub> 30 ml lactic acid	Use fresh. (a) Swab with heavy pressure for 5 to 10 sec. Water rinse, alcohol rinse, dry, then etch with No. 98c. (b) Swab for 5 to 30 seconds.
133	50 ml HNO <sub>3</sub> 50 ml acetic acid	<b>Use Hood. Do not Store. Mix fresh.</b> Immerse or swab 5 to 30 sec. Will chemically polish with longer times. Sulfidized grain boundaries etched before normal grain boundaries.
134	70 ml H <sub>3</sub> PO <sub>4</sub> 30 ml water	Electrolytic at 5 to 10 V for 5 to 60 sec. (Polishes at high currents.)
135	80 ml HNO <sub>3</sub> 3 ml HF	<b>Use Hood.</b> Warm specimen in boiling water prior to immersion for 10 to 120 seconds.
136	20 ml H <sub>3</sub> PO <sub>4</sub> 80 ml water	Electrolytic at 10-20 V for 10 to 15 seconds.
137	10 g NaNO <sub>3</sub> 100 ml water	Electrolytic 9.2 A/cm <sup>2</sup> , 1 minute.
138	5 g FeCl <sub>3</sub> 2 ml HCl 100 ml ethanol (95%) or methanol (95%)	Swab 10 to 60 seconds.
139	5 g KCN 100 ml water 0.5 ml H <sub>2</sub> O <sub>2</sub> (3%)	<b>Use Hood.</b> Can give off extremely poisonous hydrogen cyanide. <b>Precaution</b> — Also poisonous by ingestion as well as contact. To discard, neutralize (or turn basic) with ammonia and flush down acid drain with copious amount of water. Immerse 10 to 100 seconds.
140	50 ml acetic acid 50 ml HNO <sub>3</sub> 50 ml acetone	<b>Use Hood. Do not Store.</b> Decomposes with possible explosion on standing. Immerse 10 to 30 seconds.
141	3 f NH <sub>4</sub> Cl 3 g CrO <sub>3</sub> 10 ml HNO <sub>3</sub> 90 ml water	Swab 5 to 30 seconds. <b>Do not Store.</b>

TABLE 2 — continued

Etchant	Composition	Procedure
142	5 ml HF 10 ml glycerin 85 ml water	Electrolytic at 2-3 V for 2 to 10 seconds.
143	0.01-1 g CrO <sub>3</sub> 100 ml HCl	Allow solution to age a few minutes before using. Immerse or swab few seconds to few minutes.
144	A 10 g sodium thiosulfate 100 ml water B 10 ml HCl 90 ml water	Electrolytic in Solution A: sample is cathode, 10 V, 5 to 10 sec. Then electrolytic in Solution B: sample is anode, 10 V, 5 to 10 seconds.
145	2 ml H <sub>2</sub> SO <sub>4</sub> 100 ml water	Electrolytic at 3 to 10 V for 5 to 15 seconds. Use platinum wires. H <sub>2</sub> SO <sub>4</sub> may be increased to 20 ml for deeper attack.
146	10 ml HF 100 ml HNO <sub>3</sub>	Immerse 30 seconds to 3 minutes.
147	20 ml HNO <sub>3</sub> 80 ml HCl	Immerse 5 to 30 seconds.
148	5 ml HNO <sub>3</sub> 100 ml water	Immerse 10 to 30 seconds.
149	50 ml HCl 2 ml H <sub>2</sub> O <sub>2</sub> (30%) 50 ml water	Immerse 10 to 30 seconds. <b>Do not store.</b>
150	60 ml HCl 20 ml HNO <sub>3</sub> 40 ml glycerin	<b>Use Hood. Do not Store.</b> Swab few seconds to a minute. Discard when solution turns dark yellow.
151	10 ml HF 25 ml HNO <sub>3</sub> 150 ml water	Swab 5 to 30 seconds.
152	85 ml NH <sub>4</sub> OH 15 ml H <sub>2</sub> O <sub>2</sub> (30%)	Immerse 5 to 15 seconds. <b>Do not Store.</b> Decomposes.
153	10 ml HNO <sub>3</sub> 50 ml HCl 60 ml glycerin	<b>Use Hood. Do not Store.</b> Add HNO <sub>3</sub> last. Discard when dark yellow. Immerse 10 to 60 seconds. Preheating sample in boiling water hastens reaction.
154	50 ml HCl 50 ml ethanol (95%) or methanol (95%)	Immerse 10 to 100 seconds.
155	3 ml selenic acid 10 ml HCl 100 ml methanol (95%) or ethanol (95%)	Immerse 1 to 15 minutes. (Up to 30 ml of HCl may be used for more vigorous action.) Stable for 3 to 90 days, depending on HCl concentrations.
156	1 g thiourea 1 ml H <sub>3</sub> PO <sub>4</sub> 1000 ml water	Electrolytic, 0.005-0.01 A/cm <sup>2</sup> , 1 to 2 minutes.
157	25 g CrO <sub>3</sub> 150 ml HCl 50 ml water	Immerse 5 to 20 seconds.
158	10 ml HF 10 ml HNO <sub>3</sub> 20 ml glycerin	Swab 5 to 15 seconds.
159	5 ml HF 20 ml HNO <sub>3</sub> 50 ml acetic acid	Swab 10 to 30 seconds.
160	20 ml HF 15 ml H <sub>2</sub> SO <sub>4</sub> 5 ml HNO <sub>3</sub> 50 ml water	Immerse to 5 minutes.
161	25 ml HNO <sub>3</sub> 5 ml HF 50 ml water	Immerse 5 to 120 seconds.
162	A 50 ml lactic acid 30 ml HNO <sub>3</sub> 2 ml HF B 30 ml lactic acid 10 ml HNO <sub>3</sub> 10 ml HF	Swab 1 to 3 minutes in Solution A. (acts as etch polish). To etch, swab with Solution B for 5 seconds. Repeat if necessary. The HF may be varied to give more or less etching.
163	30 ml H <sub>2</sub> SO <sub>4</sub> 30 ml HF 3-5 drops H <sub>2</sub> O <sub>2</sub> (30%) 30 ml water	Immerse 5 to 60 seconds. Use this solution for alternate etch and polishing.

TABLE 2 — continued

Etchant	Composition	Procedure
164	50 ml HNO <sub>3</sub> 30 g ammonium bifluoride 20 ml water	Use Hood. Swab 3 to 10 seconds.
165	10 ml HCl 90 ml ethanol	(a) Electrolytic at 10 V for 30 seconds. Use carbon cathode and platinum wire connection to specimen. For etch polishing, use shorter time. (b) Electrolytic at 6 V for 10 seconds. Use stainless steel cathode and platinum or Nichrome wire contact to specimen.
166	A 20 g ammonium persulfate 90 ml water B 20 g KCN 90 ml water	Use Hood. Can give off extremely poisonous hydrogen cyanide. <b>Precaution</b> — Also poisonous by ingestion as well as contact. To discard, neutralize (or turn basic) with ammonia and flush down acid drain with copious amount of water. Mix 1 + 1 ratio of Solutions A and B just before use. (A mixture of 5 drops of each will cover the surface of a 1 in. dia. mount.) Immerse to several minutes.
167	5 g NaCN 100 ml water	Use Hood. Can give off extremely poisonous hydrogen cyanide. <b>Precaution</b> — Also poisonous by ingestion as well as contact. To discard, neutralize (or turn basic) with ammonia and flush down acid drain with copious amount of water. Electrolytic at 1.5 V ac for 1 to 2 minutes Use platinum cathode.
168	20 ml HCl 35 g NaCl 80 ml water	Composition given will saturate the solution with NaCl. Electrolytic at 1½ V ac for 1 minute.
169	5 ml HNO <sub>3</sub> 50 ml ethylene glycol 20 ml ethanol (95%) or methanol (95%)	Electrolytic at 0.05 A/cm <sup>2</sup> for 2 minutes. Use stainless steel cathode.
170	1 ml HF 30 ml HNO <sub>3</sub> 30 ml lactic acid	(a) Swab 5 to 30 seconds. Follow with water rinse, alcohol rinse, dry. (b) Swab for 10 second intervals. Increase HF to exaggerate grain boundaries.
171	concentrated HCl	Use Hood. Electrolytic at 5 V ac for 1 to 2 minutes. For etch-polishing, use shorter times. Follow with water rinse, alcohol rinse, and dry.
172	A 5 g ammonium persulfate 100 ml water B 5 g KCN 100 ml water	Use Hood. Can give off extremely poisonous hydrogen cyanide. <b>Precaution</b> — Also poisonous by ingestion as well as contact. To discard, neutralize (or turn basic) with ammonia and flush down acid drain with copious amount of water. Prepare 1 + 1 mixture of Solutions A and B just before use. (A mixture of 5 drops of each will cover the surface of a 1 in. dia. mount.) Immerse 1 to 2 minutes.
173	50 ml NH <sub>4</sub> OH 10-30 ml H <sub>2</sub> O <sub>2</sub> (50%)	Immerse few seconds to a minute.
174	A 25 ml HNO <sub>3</sub> 1 g K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> 1000 ml water B 40 g CrO <sub>3</sub> 3 g Na <sub>2</sub> SO <sub>4</sub> 200 ml water	Prepare 1 + 1 mixture of Solutions A and B. Apply with camel's hair brush. Nonadherent film of silver chromate should form. If film adheres, add more of Solution A, if none forms, add Solution B.
175	1 g CrO <sub>3</sub> 1 ml H <sub>2</sub> SO <sub>4</sub> 1000 ml water	Immerse to 1 minute.
176	2 g FeCl <sub>3</sub> 100 ml water	Immerse 5 to 30 seconds.
177	10 g NaOH 100 ml water	Swab or immerse 5 to 15 seconds.
178	20 ml HF 20 ml HNO <sub>3</sub> 60 ml lactic acid	Swab for 5 to 20 seconds.
179	A 10 ml HF 10 ml HNO <sub>3</sub> 30 ml lactic acid B 10 ml HF 90 ml H <sub>2</sub> SO <sub>4</sub>	Use Hood. Mix Solution B very slowly. Solution B is used as a chemical polish, though some etching will occur. Swab 2 or more minutes for desired surface. If surface is insufficiently etched use Solution B electrolytically 1/2 to 1 V/in. <sup>2</sup> of specimen. Use carbon cathode and platinum wire connection to specimen. Discard Solution B after 1 hour.



TABLE 2 — continued

Etchant	Composition	Procedure
180	10 ml HNO <sub>3</sub> 30 ml acetic acid 50 ml glycerin	Immerse for 1/2 to 10 minutes at 38 to 42 C (100 to 108 F).
181	2 ml HCl 100 ml ethanol (95%) or methanol (95%)	Swab for 1 to 3 minutes.
182	10 ml HNO <sub>3</sub> 10 ml acetic acid 80 ml glycerin	Immerse for 1/2 to 10 minutes at 38 to 42 C (100 to 108 F).
183	2 drops HF 1 drop HNO <sub>3</sub> 25 ml glycerin	Immerse for 1 minute.
184	10 g FeCl <sub>3</sub> 2 ml HCl 100 ml water	Immerse for 1/2 to 5 minutes.
185	10 ml HF 10 ml HNO <sub>3</sub>	Swab for few seconds.
186	10 ml HF 5 ml HNO <sub>3</sub> 85 ml water	Swab 3 to 20 seconds.
187	10 ml HF 30 ml HNO <sub>3</sub> 50 ml water	Swab 3 to 20 seconds.
188	1 ml HF 2 ml HNO <sub>3</sub> 15 ml H <sub>2</sub> O <sub>2</sub> (30%) 50 ml water	Swab until stain is removed.
189	10 ml HF 25 ml HNO <sub>3</sub> 45 ml glycerin 20 ml water	Swab 3 to 20 seconds.
190	8 g KOH 10 ml H <sub>2</sub> O <sub>2</sub> (30%) 60 ml water	Swab 3 to 20 seconds.
191	25 ml HF 18 g benzalkonium chloride 35 ml methanol (95%) 40 ml glycerin	Swab 3 to 20 seconds.
192	1-3 ml HF 2-6 ml HNO <sub>3</sub> 100 ml water	Swab 3 to 10 sec. or immerse 10 to 30 sec. (HF attacks and HNO <sub>3</sub> brightens the surface of titanium. Make concentration changes on this basis.)
193	2 drops HF 1 drop HNO <sub>3</sub> 3 ml HCl 25 ml glycerin	Swab 3 to 20 seconds.
194	20 ml HF 20 ml HNO <sub>3</sub> 60 ml glycerin	Immerse 5 to 30 seconds.
195	30 ml H <sub>3</sub> PO <sub>4</sub> 30 ml ethylene glycol 50 ml ethanol (95%)	Electrolytic at 18 to 20 V (0.03 A/cm <sup>2</sup> ) for 5 to 15 minutes.
196	18 g CrO <sub>3</sub> 75 ml acetic acid 20 ml water	Dissolve CrO <sub>3</sub> in hot water and cool before adding acetic acid. Keep solution below 2 C (35 F) during use. Electrolytic at 80 V for 5 to 30 minutes. <b>Do not Store.</b>
197	5 g oxalic acid 100 ml water	Electrolytic at 6 V for 5 to 20 seconds.
198	30 ml HF 30 ml HNO <sub>3</sub> 30 ml glycerin	Swab for 60 seconds.
199	2 ml HF 5 g AgNO <sub>3</sub> 100 ml water	Swab for 5 seconds.
200	A 40 g CrO <sub>3</sub> 3 g Na <sub>2</sub> SO <sub>4</sub> 200 ml water B 40 g CrO <sub>3</sub> 200 ml water	Immerse in Solution A with gentle agitation for several seconds. Rinse in Solution B.

TABLE 2 — continued

Etchant	Composition	Procedure
201	A 40 g CrO <sub>3</sub> 1.5 g Na <sub>2</sub> SO <sub>4</sub> 200 ml water B 40 g CrO <sub>3</sub> 200 ml water	Immerse in Solution A with gentle agitation for several seconds. Rinse in Solution B.
202	A 10 g CrO <sub>3</sub> 1 g Na <sub>2</sub> SO <sub>4</sub> 200 ml water B 40 g CrO <sub>3</sub> 200 ml water	Immerse in Solution A for 2 to 5 seconds. Rinse in Solution B.
203	20 g CrO <sub>3</sub> 100 ml water	Electrolytic at 0.2 A/cm <sup>2</sup> for 5 seconds.
204	10 ml perchloric acid 10 ml glycerin 70 ml ethanol (95%) 10 ml water	<b>Precaution</b> — Keep cool when mixing. Electrolytic at 15 to 50 V for 15 to 60 seconds.
205	5 ml HF 2 ml AgNO <sub>3</sub> (5%) 100 ml water	Swab vigorously for 10 to 60 seconds. Wet cotton frequently.
206	5 ml HF 10 ml HNO <sub>3</sub> 100 ml glycerin	<b>Precaution</b> — Discard after use. Solution decomposes on standing. Electrolytic at 9 to 12 V for 1 to 10 minutes.
207	30 ml HNO <sub>3</sub> 30 ml acetic acid 30 ml water	Swab for 5 to 30 seconds.
208	1 ml NH <sub>4</sub> OH 3 g ammonium persulfate 100 ml water	Immerse or swab few seconds to a minute.
209	15 ml HNO <sub>3</sub> 3 ml HF 80 ml water	Immerse 5 to 60 seconds.

TABLE 3 — ETCHANT NAMES

Common Name	No.	Common Name	No.
Aqua regia	12	Grosbeck's	19
Barker's	5	Hatch	2
Beraha's	99	Howarth's	84
Beraha's E	155	Kalling's 1	95
Carapella	138	Kalling's 2	94
Chrome regia	101	Keller's	3
Contrast	141	Kroll's	192,187
CP4	60	Marble's	25
E1-1R	107	Murakami's	98
Flat	133	Nital	74
Frank's	104	Palmerton	200
Fry's	79	Phoschromic	111
G	107	Pical	76
Glyceregia	87	Super Pical	77
Gorsuch	75	Vilella's	80
Grard's No. 1	35	92-5-3	105
Green contrast	94		

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TABLE 4 —  
ETCHING REAGENTS FOR IRON & STEEL<sup>3</sup>

Bethlehem Steel Corporation  
Compiled by Homer Research Laboratories

Etching Reagent	Composition	Remarks	Uses
<b>I. General Reagents for Irons and Steels (Carbon, Low and Medium-Alloy Steels)</b>			
Nital	2 ml HNO <sub>3</sub> , 98 ml ethyl or methyl alcoho. (95% or absolute; also amyl alcohol).	Not as good as picral for high-resolution work with heat-treated structures. Excellent for outlining ferrite grain boundaries. Etching time: a few seconds to 1 minute.	For carbon steels: gives maximum contrast between pearlite and a ferrite or cementite network; reveals ferrite boundaries; differentiates ferrite from martensite.
Picral	4 g picric acid, 100 ml ethyl or methyl alcohol (95% or absolute; use absolute alcohol only when acid contains 10% or more moisture), 4 or 5 drops zephiran chloride (17%) — wetting agent.	Not as good as nital for revealing ferrite grain boundaries. Gives superior resolution with fine pearlite, martensite, tempered martensite, and bainitic structures. Detects carbides. Etching time: a few seconds to 1 minute or more.	For all grades of carbon steels: annealed, normalized, quenched, quenched and tempered, spheroidized, austempered.
Sodium metabisulfite	(A) 8 g Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> , 100 ml distilled water.	General reagent for steel. Results similar to picral. Etching time: a few seconds to 1 minute.	Darkens as-quenched martensite.
	(B) 1 g Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> , dilute to 100 ml with distilled water.	Immerse specimen in the solution for 2 min. or until the polished surface turns a bluish-red (Do not mount specimen in a steel clamp.)	Tint etches lath-type or plate-type martensite in Fe-C alloys.
Vilella's reagent	5 ml HCl, 1 g picric acid, 100 ml ethyl or methyl alcohol (95% or absolute).	Best results obtained when martensite is tempered.	For revealing austenitic grain size in quenched and quenched and tempered steels.
Heat tinting	Heat only.	Heat by placing specimen face up on a hot plate that has been preheated to 400-700 F. Time and temperature both have decided effects. Bath of sand or molten metal may be used.	Pearlite first to pass thru a given color, followed by ferrite; cementite less affected, iron phosphide still less.
Heat etching	Heat only.	Specimen is heated 10 to 60 minutes at 1,500-2,200 F in carefully purified hydrogen, and must have no contact with scale or reducible oxides. After etching, specimen is cooled in mercury to avoid oxidation.	For revealing austenitic grain size of polished specimens.
Klemm's reagent	50 ml saturated (in H <sub>2</sub> O) Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> solution, 1 g K <sub>2</sub> S <sub>2</sub> O <sub>2</sub> .	Etching time: 40-120 sec. Ferrite appears black-brown, while carbides, nitrides, and phosphides remain white. Also, phosphorus distribution can be detected more sensitively than with usual phosphorus reagents based on copper salts.	Tint etches pearlite, hardened structures of unalloyed steel, and cast iron.
<b>II. General Reagents for Alloy Steels (High Alloy, Stainless, and Tool Steels)</b>			
Ferric chloride and hydrochloric acid	5 g FeCl <sub>3</sub> , 50 ml HCl, 100 ml distilled water.	Immerse until structure is revealed.	Reveals structure of austenitic nickel and stainless steels.
Mixed acids in glycerin	(A) 10 ml HNO <sub>3</sub> , 20 ml HCl, 30 ml glycerin.	Mix HCl and glycerin thoroughly before adding HNO <sub>3</sub> . Before etching, heat specimen in hot water. Best results are obtained with alternate polishing and etching.	Etches structures of Fe-Cr alloys, high-speed steels, austenitic steels, and manganese steels. For austenitic alloys.
	(B) 10 ml HNO <sub>3</sub> , 20 ml HCl, 20 ml glycerin, 10 ml H <sub>2</sub> O <sub>2</sub> .	Use hood. Do not store. Action can be modified by varying the proportion of HCl.	Reveals the structures of Cr-Ni and Cr-Mn steels, and of all Fe-Cr austenitic alloys.
Cupric chloride and hydrochloric acid	5 g CuCl <sub>2</sub> , 100 ml HCl, 100 ml ethyl alcohol, 100 ml distilled water.	Use cold.	For austenitic and ferritic steels, the ferrite being most easily attacked (carbides and austenite are not attacked).

<sup>3</sup> From Metal Progress Databook.

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TABLE 4 — continued

Etching Reagent	Composition	Remarks	Uses
Nitric and hydrofluoric acids	5 ml HNO <sub>3</sub> , 1 ml HF (48%), 44 ml distilled water.	Use cold for about 5 min. under hood. HF is harmful to skin.	For revealing general structure of austenitic stainless steel with avoidance of strain markings.
Heat tinting	Heat only in air for 10 to 60 sec. at about 1,000-1,200 F. (595 to 650 C).	Carbides remain white, and austenite darkens less rapidly than ferrite; specimens preferably etched first with a chemical reagent. Use hood.	For austenitic stainless steels containing ferrite and carbides.
Ferric chloride and nitric acid	Saturated solution of FeCl <sub>3</sub> in HCl, to which a little HNO <sub>3</sub> is added.	Use full strength under hood.	Structure of stainless steels.
Mixed acids in cupric chloride	30 ml HCl, 10 ml HNO <sub>3</sub> , saturate with cupric chloride, and let stand 20 to 30 minutes before using.	Apply by swabbing.	For stainless alloys and others high in nickel or cobalt.
Nitric and acetic acids	30 ml HNO <sub>3</sub> , 20 ml CH <sub>3</sub> COOH.	Apply by swabbing under hood. Do not store.	For stainless alloys and others high in nickel or cobalt.
Marble's reagent	4 g CuSO <sub>4</sub> , 20 ml HCl, 20 ml distilled water.	Immerse to reveal structure.	Structure of stainless steels.
Ferricyanide solution	50 g K <sub>3</sub> Fe(CN) <sub>6</sub> , 50 g KOH, 100 ml distilled water.	Must be fresh. Use boiling 2 to 5 min. under hood. Do not acidify; deadly HCN may be released.	To distinguish between ferrite and sigma phase in Fe-Cr, Fe-Cr-Ni, Fe-Cr-Mn, and related alloys. Colors sigma blue, ferrite yellow.
Vilella's reagent	5 ml HCl, 1 ml picric acid, 100 ml ethyl or methyl alcohol (95% or absolute).	Immerse to reveal structure.	Can etch numerous types of Fe-Cr, Fe-Cr-Ni, and Fe-Cr-Mn steels. Also attacks the grain boundaries in Cr-Ni austenitic steels.
Cupric sulfate and perchloric acid	10 g CuSO <sub>4</sub> , 45 ml perchloric acid (70%), 55 ml distilled water.	Boil 15 min. Do not use in presence of organic materials. Use hood; do not concentrate acid. Highly explosive.	Etches stainless steels, and shows chromium segregation by revealing areas poor in chromium.
Acetic, nitric, and hydrochloric acids	25 ml CH <sub>3</sub> COOH, 15 ml HNO <sub>3</sub> , 15 ml HCl, 5 ml distilled water.	Apply by swabbing under hood. Do not store.	Fe-Al alloys, general microstructure.
Hydrochloric and chromic acids	25 ml HCl, 50 ml CrO <sub>3</sub> (10% chromic acid aqueous solution).	Activity is controlled by the amount of chromic acid. Use hood.	Suitable for heat-treated type 300 stainless steels.
Hydrochloric acid in alcohol	50 ml HCl, 50 ml ethyl alcohol.	More gradual etching can be obtained with less concentrated solutions (10-20%).	Suitable for etching steels containing chromium and nickel.
Mixed acids in ethyl alcohol	2.5 g FeCl <sub>3</sub> , 5 g picric acid, 2 ml HCl, 90 ml ethyl alcohol.	Etching time: 15 sec for austenitic cast irons to 1 hour or more for high-chromium ferritic irons.	For high-chromium, high-carbon cast irons.
Nitric acid	5 to 10 ml HNO <sub>3</sub> , 100 methyl or ethyl alcohol (95% or better).	Use hood. HNO <sub>3</sub> and ethyl alcohol are a dangerous mixture above 5% HNO <sub>3</sub> .	General structure of high-speed tool steels.
Sodium metabisulfite (1)	15 g Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> , 100 ml distilled water.	Etching time: a few seconds to 1 minute.	General structure of high-speed tool steels.
Hydrochloric and nitric acids	10 ml HCl, 3 ml HNO <sub>3</sub> , 100 ml methyl alcohol.	Etching time: 2 to 10 minutes.	To reveal the grain size of quenched or quenched and tempered high-speed steel.
Grosbeck's reagent	4 g KMnO <sub>4</sub> , 4 g NaOH, 100 ml distilled water.	Use at boiling point for 1 to 10 minutes.	For high-speed and chromium or cobalt-rich alloys.
Sodium metabisulfite (2)	Step No. 1: 25 ml HNO <sub>3</sub> , 75 ml ethyl or methyl alcohol.	Pre-etch 10 sec., outlines grain boundaries and some structure. (Caution: HNO <sub>3</sub> and ethyl dangerous at this concentration).	Tint etchant for Fe-Ni alloys from 5-25% Ni. Colors martensitic packets of different orientations different colors, and reveals the substructure of lath-type martensite.

TABLE 4 — continued

Etching Reagent	Composition	Remarks	Uses
Beraha's reagent (1)	Step No. 2: 15 to 35 g Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> , dilute to 100 ml with distilled water.	Immerse for 2 min. or until polished surface turns bluish-red.	Concentration of Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> varies with nickel content.
	3 g K <sub>2</sub> S <sub>2</sub> O <sub>5</sub> , 10 g Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> , dilute to 100 ml with distilled water.	Pre-etch with 4% picral for 1 to 2 minutes. Immerse for 2 min. or until polished surface turns bluish-red.	Tint etchant for Fe-Mn alloys from 5-18% Mn. Also good for revealing chemical and physical heterogeneity in Fe-C alloys. Colors ferrite while cementite remains white in Fe-C alloys.
Klemm's reagent	50 ml cold saturated (in H <sub>2</sub> O) Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> solution, 5 g K <sub>2</sub> S <sub>2</sub> O <sub>5</sub> .	Distinguishes between gamma, epsilon, and alpha phases. Epsilon-martensite remains white, alpha-martensite is colored black, and gamma, gray. Contrast can be improved in chromium-rich steels by addition of glacial acetic acid.	Tint etches Mn, Mn-C, and Mn-Cr steels.
Beraha's reagent (2)	<b>Stock Solutions</b>		
	A. 1 vol. HCl (35%) + 5 vol. distilled water.	Tint etchant No. 1: 100 ml of stock solution A or D plus 100 to 200 mg potassium metabisulfite. Immerse at room temperature and keep specimen moving until desired coloration is attained. Note: Containers and forceps of suitable plastic materials should be used for a reagent which contains ammonium bifluoride. Exposure to skin is dangerous. Use hood.	Tint etchant for martensitic stainless steel. Colors the matrix only; carbides and nitrides are unaffected and in contrast with the colored matrix.
	B. 1 vol. HCl (35%) + 2 vol. distilled water.		
	C. 1 vol. HCl (35%) + 1 vol. distilled water.		
	D. 20 g. ammonium bifluoride dissolved in 1,000 ml of stock solution A.		
	E. 40 g ammonium bifluoride dissolved in 1,000 ml of stock solution B.		
	F. 50 g ammonium bifluoride dissolved in 1,000 ml of stock solution C.	Tint etchant No. 2: Same as above except potassium metabisulfite 300 to 600 mg.	For ferritic and austenitic stainless steel. Same as above.
	G. 10 to 15 g iron chloride dissolved in 1,000 ml of stock solution B or C.	Tint etchant No. 3: 100 ml of stock solution (B, C, E, F, G, or H) + 300 to 800 mg potassium metabisulfite. If coloration takes place without etching, lower amount of potassium metabisulfite.	For corrosion and heat-resisting alloys. Same as above.
H. 10 g copper chloride dissolved in 1,000 ml of stock solution B or C.			
<b>III. Miscellaneous Reagents (Segregation, Depth of Case, Primary Structure, and Strain Lines)</b>			
Stead's reagent	1 g CuCl <sub>2</sub> , 4 g MgCl <sub>2</sub> , 1 ml HCl, 100 ml alcohol (absolute).	Dissolve salts in least possible quantity of hot water. Etch for about 1 minute, repeating if necessary.	To show segregation of phosphorous or other elements in solid solution; copper tends to deposit first on areas lowest in phosphorous. The structure may be more clearly delineated by light hand polish to remove the copper deposit after etching.
Fry's reagent	5 g CuCl <sub>2</sub> , 40 ml HCl, 30 ml distilled water, 25 ml ethyl alcohol.	May be used cold. Etching time: about 10 seconds.	To reveal strain lines.
Oberhoffer's reagent	30 g FeCl <sub>3</sub> , 1 g CuCl <sub>2</sub> , 0.5 g SnCl <sub>2</sub> , 50 ml HCl, 500 ml ethyl alcohol, 500 ml distilled water.	Immerse to reveal structure.	For showing phosphorus segregation and dendritic structure.
Alkaline chromate	16 g CrO <sub>3</sub> , 145 ml distilled water, 80 g NaOH.	Add NaOH slowly, and use when not over one day old, boiling at 244-248 F. for 7-20 min. Use hood. This solution is highly caustic; should be prepared and stored in plastic.	Shows oxygen segregation by darkening martensite rapidly, ferrite more slowly, and zones of high oxygen content much more slowly.

TABLE 4 — continued

Etching Reagent	Composition	Remarks	Uses
Cupric sulfate and cupric chloride	1.25 g CuSO <sub>4</sub> , 2.50 g CuCl <sub>2</sub> , 10 g MgCl <sub>2</sub> , 2 ml HCl, 100 ml distilled water. Dilute above solution to 1,000 ml with 95% ethyl alcohol.	Proportions must be accurate. Etch by immersion to avoid confusing edge effects. Etching time: 30 seconds to 1 minute.	For showing total depth of case, structure, and various zones of nitrided Cr-V steels and Nitralloy.
Picric and nitric acids	10 parts picric acid (4%), 1 part HNO <sub>3</sub> (4%).	Best results are obtained when specimen is annealed in lead at 1,475° F (800° C) before etching.	For depth of case and structure of Nitralloy.
Nital	1 ml HNO <sub>3</sub> , 100 ml ethyl or methyl alcohol (95% or absolute).		For structure and depth of case of nitrided steels.
Marble's reagent	4 g CuSO <sub>4</sub> , 20 ml HCl, 20 ml distilled water.		Total depth of nitrided case.
Ammonium acetate	10 ml CH <sub>3</sub> COONH <sub>4</sub> , 100 ml distilled water.		Stains high sulfur areas in steel, and stains lead particles brown in leaded steels.
<b>IV. Reagents for Nonmetallic Inclusions and Intermetallic Compounds</b>			
Ferricyanide solution	1 to 4 g K <sub>3</sub> Fe(CN) <sub>6</sub> , 10 g KOH, 100 ml distilled water.	Must be freshly made; etch 15 min. in boiling solution; 7 g of NaOH may be substituted for 10 g of KOH. Use hood; do not acidify as HCN may be released.	Differentiates between carbides and nitrides. Cementite is blackened, pearlite turns brown, and massive nitrides remain unchanged.
Murakami's reagent	10 g K <sub>3</sub> Fe(CN) <sub>6</sub> , 10 g KOH, 100 ml distilled water.	May be used cold, but preferably hot; should be freshly made; 7 g of NaOH may be substituted for 10 g KOH. Etching time 5-10 min. Use hood (see warning above).	Alloy, high-speed, and tungsten steels. Colors various carbides differently; cementite not affected.
Strong ferricyanide solution	40 g K <sub>3</sub> Fe(CN) <sub>6</sub> , 100 g KOH, 100 ml distilled water.	Use fresh, boiling for 15 min. Use hood (see warning above).	Stainless and alloy steels; differentiates between carbides and nitrides.
Chromic acid and heat tinting	8 g CrO <sub>3</sub> , 100 ml distilled water.	Etch first in 4% picric acid then for 1 min. in chromic acid; heat tint by heating face-up on a hot plate at about 500° F for 1 minute.	Distinguishes between iron phosphide and cementite in phosphide eutectic of cast iron; iron phosphide is colored darker.
Sodium picrate (alkaline)	2 g picric acid, 25 g NaOH, 100 ml distilled water.	Use boiling, 5 to 10 min. Do not boil dry.	Colors cementite, but not carbides high in chromium. Attacks sulfides; delineates grain boundaries in hyper-eutectoid steels in slowly cooled condition.
Hydrogen peroxide and sodium hydroxide	10 ml H <sub>2</sub> O <sub>2</sub> (30%), 20 ml NaOH 10% solution in distilled water.	Must be fresh. Etching time: 10 to 20 min. Highly caustic solution.	Attacks and darkens iron tungstide in carbon-free Fe-W alloys. When carbon is present, this solution darkens the compound (FeW, WC) in proportion to the amount of carbide present; tungsten carbide is darkened.
Sodium hydroxide and potassium permanganate	4 g NaOH, 4 g KMnO <sub>4</sub> , 100 ml distilled water.	Use boiling. Etching time: 1 to 10 minutes.	Alloy and high-speed steels. Colors precipitated carbides in manganese or chromium steels. Differentiates between carbides and tungstides. Vanadium carbide unattacked.
Sodium hydroxide and bromine	20 g NaOH, 4 ml bromine, 80 ml distilled water.	Does not keep. Use fresh under hood.	Colors iron phosphide.
Sodium hydroxide and lead nitrate	1 part 50% NaOH, 2 parts 10% Pb(NO <sub>3</sub> ) <sub>2</sub> .	Use fresh, cold or boiling. Highly caustic solution.	Steels, colors cementite, attacks phosphides and silicates.

TABLE 4 — continued

Etching Reagent	Composition	Remarks	Uses
<b>V. Reagents for Macroscopic Examination</b>			
Hydrochloric acid	50 ml HCl, 50 ml H <sub>2</sub> O.	Use at 160° to 180°F. (71° to 82°C) for 1 to 60 min. depending on the size of sample, type of steel, and type of structure to be developed. Use hood.	Shows segregation, porosity, cracks depth of hardened zone in tool steel, and so on. May produce cracks in strained steel.
Mixed acids	38 ml HCl, 12 ml H <sub>2</sub> SO <sub>4</sub> , 50 ml H <sub>2</sub> O.	Use hot or boiling, 15 to 45 min. or cold for 2 to 4 hours. Use hood.	Steel, general macro; one of the best. Shows segregation, cracks, hardened zone, soft spots, weld structures.
Nitric acid in water	(A) 25 ml HNO <sub>3</sub> , 75 ml H <sub>2</sub> O.	Use cold on large surfaces such as split ingots which cannot conveniently be heated.	Same as HCl reagent.
	(B) 0.5 to 1 ml HNO <sub>3</sub> , 99.5 to 99 ml H <sub>2</sub> O.	Immerse 30 to 60 sec. after grinding specimen on 240-grit emery belt and thorough cleaning.	To show weld structures.
Nital	5 ml HNO <sub>3</sub> , 95 ml ethyl alcohol.	Etch 5 minutes followed by 1 second in 10% HCl in H <sub>2</sub> O.	Shows cleanliness, depth of hardening, carburized or decarburized surface, and so on.
Ammonium persulfate	10 ml (NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub> , 90 ml H <sub>2</sub> O.	Surface should be rubbed with absorbent cotton during etching.	Brings out grain structure, excessive grain growth, recrystallization at welds, flow lines in Nitralloy, and so on.
Ammonium persulfate with iodide, and so on.	(A) 2.5 g (NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub> , 100 ml H <sub>2</sub> O.	After grinding on No. 320 abrasive paper, swab 15 min. with solution A, then 10 min. with B, then 5 min. with C, and 5 min. with D, finally washing with water and drying with alcohol.	Shows dendritic macrostructure of cast iron.
	(B) Same as A, plus 1.5 g KI.		
	(C) Same as B, plus 1.5 g HgCl <sub>2</sub> .		
	(D) Same as C, plus 15 ml H <sub>2</sub> SO <sub>4</sub> .		
Stead's reagent	2.5 g CuCl <sub>2</sub> , 10 g MgCl <sub>2</sub> , 5 ml HCl, up to 250 ml ethyl alcohol.	Salts are dissolved in HCl with the addition of the least possible quantity of water.	Brings out phosphorus-rich areas and phosphorus banding. May be used for general segregation.
Fry's reagent	(A) 90 g CuCl <sub>2</sub> , 120 ml HCl, 100 ml H <sub>2</sub> O.	Most useful for low-carbon steels, particularly bessemer and other high-nitrogen grades. Before etching, sample should be heated to 300°-480° F. for 5 to 30 min., depending on condition of steel. During etching, surface should be rubbed with cloth soaked in etching solution. Wash in alcohol or rinse in HCl (1:1) after etching to prevent deposition of copper.	Shows up strain lines due to cold work.
	(B) 45 g CuCl <sub>2</sub> , 180 ml HCl, 100 ml H <sub>2</sub> O.	Specimen can be washed in water without depositing copper. Gives contrast.	Shows strain lines due to cold work.
Humfrey's reagent	120 g Cu(NH <sub>3</sub> ) <sub>4</sub> Cl <sub>2</sub> , 50 ml HCl, 1,000 ml H <sub>2</sub> O.	Slight abrasion of surface after etching is recommended.	Develops dendritic segregation.
Kalling's reagent	1.5 g CuCl <sub>2</sub> , 33 ml HCl, 33 ml H <sub>2</sub> O, 33 ml alcohol.	Etching time very short.	Develops dendritic pattern in steel, attacks ferritic and martensitic stainless steels. Ferrite darkened, martensite darker, austenite light.
Marble's reagent	10 g CuSO <sub>4</sub> , 50 ml HCl, 50 ml H <sub>2</sub> O.	May be used hot.	Ni-Cr-Fe alloys, manganese and Cr-Mn steels, nitrated case. Carbide precipitation in austenitic alloys.

TABLE 4 — continued

Etching Reagent	Composition	Remarks	Uses
Vilella's reagent	1 g picric acid, 5 ml HCl, 100 ml alcohol.	Use hot.	Fe-Cr-Ni and Fe-Cr-Mn steels. Reveals austenitic grain boundaries.
<b>VI. Electrolytes for Polishing and Etching</b>			
Chromic acid	10 g CrO <sub>3</sub> , 100 ml H <sub>2</sub> O.	Specimen is used as anode; stainless steel or platinum as cathode, 3/4 to 1 in. apart; 6 V usually used. Etching time: 30 to 90 seconds.	For various structures except the grain boundaries of ferrite. Attacks cementite very rapidly, austenite less rapidly, ferrite and iron phosphide very slowly if at all.
Nitric acid in water	50 ml HNO <sub>3</sub> , 50 ml H <sub>2</sub> O.	Room temperature; stainless steel cathode; 1.5 V for 2 minutes or more. Use hood.	For austenitic or ferritic stainless steels; reveals grain boundaries.
Hydrochloric acid in alcohol	10 ml HCl, 90 ml anhydrous ethyl alcohol.	10 to 30 seconds at 6V.	Reveals delta ferrite, and the general structure of chromium and Cr-Ni steels.
Sulfuric acid in water	5 ml H <sub>2</sub> SO <sub>4</sub> , 95 ml H <sub>2</sub> O.	Room temperature; stainless steel cathode; 6 V (0.1 to 0.5 amp), 5 to 15 seconds. Use hood.	For Fe-Cr-Ni alloys.
Mixed acids in alcohol	45 ml lactic acid, 10 ml HCl, 45 ml ethyl alcohol.	10 to 30 seconds at 6V.	For chromium steels (4 to 30% Cr), or for delta ferrite in austenitic stainless steels.
Oxalic acid in water	10 ml oxalic acid, 100 ml H <sub>2</sub> O.	5 to 20 sec. at 6 V, using a platinum or stainless steel cathode. Gap between electrodes, 3/4 to 1 in.	For austenitic stainless steels and high-nickel alloys. Distinguishes between sigma phase and carbides. Sigma phase is attacked first, then carbides; ferrite and austenite can be attacked slightly. To investigate the carbides, operate at 1.5 to 3 V for a longer time.
Sodium hydroxide in water	40 g NaOH, 100 ml H <sub>2</sub> O (add slowly).	60 seconds at 1 to 3V. Highly caustic solution.	Reveals the sigma phase. Colors successively sigma phase, ferrite, and lastly carbides after a longer etching time.
Potassium hydroxide in water	56 g KOH, 100 ml H <sub>2</sub> O (add slowly).	60 seconds at 1 to 3V. Highly caustic solution.	Same as above, but sigma phase and ferrite are revealed simultaneously.
Sodium cyanide in water	10 g NaCN, 100 ml H <sub>2</sub> O.	5 min. or more at 6 V (not less than 5V). Use hood; if acidified, HCN develops.	Colors carbides without altering austenite or grain boundaries.
Ammonium persulfate in water	10 g (NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub> , 100 ml H <sub>2</sub> O.	Use fresh, 6 V for more than 15 seconds.	Surface attack occurs on the ferrite grains in low-carbon steels. Reveals the fine structures of nickel austenitic steels, and of transformer sheet.
Ammonium nitrate	Saturated aqueous solution of NH <sub>4</sub> NO <sub>3</sub> .	Use a current density of 1 amp per sq cm.	Detects overheating and burning. In overheating, this etchant leaves the boundaries of the pre-existing austenite grains white, while it blackens them in burned steel.
Cadmium acetate in water	10 g cadmium acetate, 100 ml H <sub>2</sub> O.	2 to 5 V for 3 to 20 seconds.	Brings out carbide grain boundaries.
Mixed acids in water	5 g ammonium molybdate, 7.5 ml HNO <sub>3</sub> , 10 ml HCl, 100 ml H <sub>2</sub> O.	12 V for 2 to 3 minutes.	Good for type 300 stainless steels.
Mixed acids in water	90 ml H <sub>2</sub> PO <sub>3</sub> , 8 ml HNO <sub>3</sub> , 2 ml H <sub>2</sub> O.	Use cold.	For polishing and etching Fe-Al alloys (to 16% Al).
Chrome regia	25 ml HCl, 5 to 50 ml CrO <sub>3</sub> solution (10%) in H <sub>2</sub> O.	Dilute with 2 parts alcohol and 2 parts glycerin. Etch for 20 to 60 seconds at 6V.	Heat-treated type 300 stainless steels.





<b>Macroetching</b>	<b>Pages</b>
Definition .....	59
Standard Methods for Macroetching Metals and Alloys (ASTM Designation: E 340-68)	
1. Scope .....	59
2. General Directions and Uses of Macroetching	59
Applications of Macroetching .....	59
Sampling .....	59, 60
Preparation .....	60
Solutions .....	60
Caution .....	60
Procedure .....	60, 61
3. Specific Preparation Procedures	
Aluminum and Aluminum Alloys .....	61
Beryllium and Beryllium Alloys .....	61
Cobalt and Cobalt Alloys .....	61
Copper and Copper Alloys .....	61, 62
Iron and Steel .....	62
Stainless Steel and High	
Temperature Alloys .....	62, 63
Lead and Lead Alloys .....	63
Magnesium and Magnesium Alloys .....	63
Nickel and Nickel Alloys .....	63
Noble Metals .....	63
Refractory Metals .....	63, 64
Tin and Tin Alloys .....	64
Titanium, Zirconium, Hafnium and	
Their Alloys .....	64
Zinc and Zinc Alloys .....	64
Table 1 - Macroetchants for Aluminum and Aluminum Alloys .....	64, 65
Table 2 - Macroetchants for Beryllium and Beryllium Alloys .....	65
Table 3 - Macroetchants for Cobalt and Cobalt Alloys .....	65
Table 4 - Macroetchants for Copper and Copper Alloys .....	65
Table 5 - Macroetchants for Iron and Steel .....	66
Table 6 - Macroetchants for Stainless Steels and High Temperature Alloys .....	67
Table 7 - Macroetchants for Lead and Lead Alloys ..	67, 68
Table 8 - Macroetchants for Magnesium and Magnesium Alloys .....	68
Table 9 - Macroetchants for Nickel and Nickel Alloys .....	69
Table 10 - Macroetchants for Noble Metals .....	69
Table 11 - Macroetchants for Refractory Metals ....	69
Table 12 - Macroetchants for Tin and Tin Alloys ...	69
Table 13 - Macroetchants for Titanium, Zirconium, Hafnium and Their Alloys .....	70
Table 14 - Macroetchants for Zinc and Zinc Alloys .	70



# MACROETCHING

Macroetching is the procedure used to reveal the quality of a material by subjecting a gross sample to the corrosive action of an etchant.

Examinations are limited to visual observation or magnification not exceeding ten diameters.

## STANDARD METHOD FOR MACROETCHING METALS AND ALLOYS<sup>4</sup>

### 1. SCOPE

1.1 These procedures describe the methods of macroetching metals and alloys to reveal their macrostructure.

### 2. GENERAL DIRECTIONS AND USES OF MACROETCHING

#### 2.1 Applications of Macroetching

2.1.1 Macroetching is used to reveal the heterogeneity of metals and alloys. Metallographic specimens and chemical analyses will provide the necessary detailed information about specific localities but they cannot give data about variation from one place to another unless an inordinate number of specimens are taken.

2.1.2 Macroetching, on the other hand, will provide information on variations in (1) structure, such as grain size, flow lines, columnar structure, dendrites, etc.; (2) variations in chemical composition as evidenced by segregation, carbide and ferrite banding, coring, inclusions, and depth of carburization or decarburization. The information provided about variations in chemical composition is strictly qualitative but the location of extremes in segregation will be shown. Chemical analyses or other means of determining the chemical composition would have to be performed to determine the extent of variation. Macroetching will also show the presence of discontinuities and voids, such as seams, laps, porosity, flakes, bursts, extrusion rupture, cracks, etc.

2.1.3 Other applications of macroetching in the fabrication of metals are the study of weld structure, definition of weld penetration, dilution of filler metal by base metals, entrapment of flux, porosity, and cracks in weld and heat affected zones, etc. It is also used in the heat treating shop to determine location of hard or soft spots, tong marks, quenching cracks, case depth in shallow hardening steels, case depth in carburization of dies, effectiveness of stop-off coatings in carburization, etc. In the machine shop, it can be used for the determination of grinding cracks in tools and dies.

2.1.4 Macroetching is used extensively for quality control in the steel industry, to determine the "tone" of a heat in billets with respect to inclusions, segregation, and structure. Forge shops, in addition, use macroetching to reveal flow lines in setting up the best forging practice, die design, and metal flow. For an example of the use of macroetching in the steel forging industry, see ASTM Method A 317, Macroetch Testing and Inspection of Steel Forgings. Forging shops and foundries also use macroetching to determine the presence of internal faults and surface defects. The copper industry uses macroetching for control of surface porosity in wire bar. In the aluminum industry, macroetching is used to evaluate extrusions as well as the other products such as forgings, sheets, etc. Defects such as coring, cracks, and port hole die welds are identified.

#### 2.2 Sampling:

2.2.1 As in any method of examination, sampling is very important. When macroetching is used to solve a problem, the problem itself largely dictates the source of the sample as to the location on the work piece and the stage of manufacture; for example, when looking for pipe, the sample should represent the top of the ingot, or when looking for bursts or flakes, the sample should be taken as soon after hot working as possible.

2.2.1.2 When macroetching is used as an inspection procedure, sampling ought to be done in an early stage of manufacturing so that if the material proves faulty, no wasteful unnecessary work is done. However, the sample should not be taken so early that further working can introduce serious defects. In the steel industry, for example, the sample is usually taken after ingot breakdown and after most chances of bursts or flakes occurring have passed. Billets or blooms going into small sizes are sampled after initial breakdown. Material going into forging billets or die blocks is sampled near finish size. Sampling may be done systematically or on a random basis.

2.2.2 Samples may be cold cut from the source by any convenient fashion; saws and abrasive cutoff wheels are particularly effective. The use of torch cutting or hot cutting should be used only when necessary to cut a sample from a large piece. The sample then is sectioned well away from the hot-cut surface. An example of permissible use of torch cutting is the excising of a piece from a large plate and then cutting a sample for macroetching 4 to 5 in. away from the torch cut edge.

2.2.3 Some common methods of sampling, listed by source are as follows:

<sup>4</sup> American Society and Testing Materials Designation: E340-68 (Reprinted with permission)

2.2.3.1 Billets, Blooms, and Hot-Rolled Products — Disks are usually cut from these products near the end. Samples cut too close to the end, however, may have false structures because of fish-tailing. Disks from large blooms are sometimes cut into smaller pieces for ease in handling.

2.2.3.2 Forgings and Extrusions — Disks cut transverse to the long dimension will show flakes, bursts, etc. Forgings may also be cut parallel to the long dimension to show flow lines. In complicated forgings, some thought will have to be given to the proper method of cutting so as to show flow lines. Macroetching of an unprepared specimen will show surface defects such as shuts, flats, seams, etc. In extrusions, coring and coarse grain are more commonly found in the back end of the extrusion.

2.2.3.3 Sheets and Plates — A sufficiently large sample should be taken when looking for surface defects. An ideal length would be the circumference of the last roll, but this may be inconveniently long. Several samples totaling some given fraction of the circumference can be used; however, there is always a chance then that a defect arising from faulty rolls would not be detected. When seeking information on laminations, a transverse section is used. In many cases, however, to reduce the size of the specimen, only a section out of the center of the plate may be taken.

2.2.3.4 Weldments — A disk cut perpendicular to the direction of welding will show weld penetration, heat affected zone, structure, etc. Careful preparation is usually rewarded with highly detailed structure giving a large amount of information. Welds involving dissimilar metals will produce problems in etching. The best method is to etch the least corrosion-resistant portion first and the more resistant portion afterward. Occasionally an intermediary etchant may be required. The boundaries between etched and unetched portion will give an idea of weld penetration and dilution.

2.2.3.5 Castings — Cut the specimen to display the defect or feature being sought.

2.2.3.6 Machined and Ground Parts — When looking for grinding cracks, etc., the surface itself is used as a sample. Because the machined or ground part is often the finished part, it may be undesirable to immerse the part in acid. In this case, other methods such as dye penetrant methods may be more desirable.

## 2.3 Preparation:

2.3.1 Sample preparation need not be elaborate. Any method of presenting a smooth surface with a minimum amount of cold work

will be satisfactory. Disks may be faced on a lathe or a shaper. The usual procedure is to take a roughing cut, then a finished cut. This will generate a smooth surface and remove cold work from prior operations. Sharp tools are necessary to produce a good specimen. Grinding is usually conducted in the same manner, using free-cutting wheels and light finishing cuts. When fine detail is required, the specimen should be ground down through the series of metallographic papers. Where necessary, details are given in the tabulation of procedures.

2.3.2 After surface preparation, the sample is cleaned carefully with suitable solvents. Any grease, oil, or other residue will produce uneven attack. Once cleaned, care should be taken not to touch the sample surface or contaminate it in any way.

## 2.4 Solutions:

2.4.1 The solutions used for macroetching are given in the tables listed under each alloy. In most cases a good grade of reagent should be used but need not be chemically pure or of analytical quality. The so-called technical grades are usually satisfactory. The solution should be clean and clear, free of suspended particles, scum, etc.

2.4.2 Caution must be observed in mixing. Many of the etchants are strong acids. In all cases, the various chemicals should be added slowly to the water or solvent while stirring. In the cases where hydrofluoric acid is used, the solution should be mixed and used in polyethylene vessels.

Note — CAUTION: Hydrofluoric acid should not be allowed to contact the skin since it can cause painful, serious ulcers if not washed off immediately.

## 2.5 Procedure:

2.5.1 Many of the solutions are aggressive and may give off irritating and corrosive fumes. Etching should be done in a well ventilated room, preferably under a fume hood. The solution should be mixed and placed in a corrosion-resistant tray or dish and brought to the operating temperature. The specimen or specimens should be placed in a tray of stainless steel screen or on some non-reactive support.

Glass rods often are placed on the bottom of the acid container and the specimens laid directly on the rods. When etching is completed, remove the specimens from the dish, taking great care not to touch the etched surface. When desmutting is required, dip the specimen into a second solution. After rinsing the specimen with hot water, blow dry with clean, compressed air.

2.5.2 In the case of large specimens, such as ingot sections, swabbing may be the only practical method of macroetching. Saturate a large wad of cotton held in stainless steel or nickel tongs with the etchant and sweep over the surface of the specimen. An effort should be made to wet the entire surface as soon as possible. After the initial wetting, keep the swab saturated with solution and frequently sweep over the surface of the specimen to renew the solution. When the structure has been suitably developed, rinse the specimen, either with a swab saturated with water, or better still, by pouring water over the specimen. After rinsing with hot water, blow the specimen dry with compressed air. Details of the procedure not discussed here are covered in the sections for the various metals and their alloys.

2.5.3 The times given in individual tabulations are only intended as guides. In fact, the progress of etching should be closely watched and etching stopped when the preferred structural details have been revealed. Specimens should be etched to develop structures. Generally, a light etch is better than a heavy etch; over-etching can often lead to misinterpretation. The actual time to develop a structure properly may be quite different from the one suggested.

### 3. SPECIFIC PREPARATION PROCEDURES AND RECOMMENDED SOLUTIONS

#### 3.1 Aluminum:

3.1.1 The specimens can be cut using the common cutting tools, hack saws, band saw, shears, abrasive cutoff wheels, etc. All these methods will cause cold work at the surface and will generate heat. The temperature rise can be enough to cause changes in structure. For these reasons, sharp tools and generous lubrication are necessary for sectioning.

3.1.2 The cold-worked surface should be removed by machining the surface. Again, sharp tools and copious lubrication are required. If fine detail is required, the machined surface should be ground using silicon carbide paper lubricated with water or kerosene.

3.1.3 Several of the solutions used in macroetching react vigorously with the metal and can overheat the specimen. In these cases the specimen is periodically removed from the solution, cooled in running water, and reimmersed in the etchant. This procedure is repeated until the desired degree of etching is obtained.

3.1.4 Macroetchants for Aluminum and Aluminum Alloys (Table 1).

#### 3.2 Beryllium:

3.2.1 While beryllium in the massive form is not dangerous, beryllium and its compounds in the finely divided state are extremely poisonous.

Note — CAUTION: Before starting any work involving beryllium, a review of hazards and plans for handling should be made. A number of references on beryllium are available. Particular mention may be made of "Toxicity of Beryllium" ASD-TR-62-7-667, prepared by the Kettering Laboratory for the Air Force.

3.2.2.1 Generally speaking, beryllium and its alloys have given difficulty in obtaining good macroetched specimens. First, beryllium is a rather brittle metal and sectioning can be difficult. Cut-off wheels with the designation C46FR70 have been the most successful. Secondly, beryllium does not grind easily; hence, specimens should be as small as possible to minimize grinding time. Grinding has been most successful with the entire sequence of wet silicon carbide papers.

3.2.2.2 The etching of fine grained metal may not always be entirely successful, and further preparation will be required. Rough polishing with  $16 \mu\text{m}$   $\text{Al}_2\text{O}_3$  suspended in water is performed on a low-nap cloth. Light pressure and frequent change of cutting direction produce the best results. If further polishing is required,  $1 \mu\text{m}$  green  $\text{Cr}_2\text{O}_3$  in tap water on synthetic suede works best.

3.2.3 Macroetchants for Beryllium and Beryllium Alloys (Table 2).

#### 3.3 Cobalt and Cobalt Alloys:

3.3.1 Many of the cobalt-base high temperature alloys can be etched using the same procedures as those for iron and nickel-base high temperature alloys. Other cobalt alloys, such as the stellites used as machine tools, require special treatment.

3.3.1.2 The cobalt-base alloys, as a group, are not easily machined. The specimens should be sectioned with abrasive cutoff wheels and ground on wet silicon carbide papers. Because of the rapid work hardening characteristics of these alloys, fresh paper and copious cooling should be used.

3.3.2 Macroetchants for Cobalt and Cobalt Alloys (Table 3).

#### 3.4 Copper and Copper Alloys:

3.4.1 These metals are usually macroetched to bring out the general structure of wire bar and billets as well as variations in grain size in extrusions and forgings.

3.4.2.1 Specimens may be sectioned using common cutting tools. To minimize cold working the tools should be kept sharp.

3.4.2.2 Good results can be obtained by machining a smooth surface in two stages, the first being a heavy cut to remove the cold work from sectioning and the second a fine cut with a V-shaped tool to remove the remaining cold work. Grinding through the series of metallographic papers will give more detailed results. The degree of grinding depends upon the amount of detail required. The etching solutions listed in the following table are simple to prepare and their use requires no special technique.

Note — It should be pointed out that heavy etching often will remove the effect of cold work but at the expense of producing a rough surface. If the specimen is then given light regrinding to remove the rough etched surface, the second etch will provide good results.

3.4.3 Macroetchants for Copper and Copper Alloys (Table 4).

### 3.5 Iron and Steel:

3.5.1 Macroetching has been most highly developed and is used extensively in the iron and steel industries. An example of the use of macroetching for inspection of tool steels is given in the appendix. In hot mill products such as bars, billets, sheet, and plate, the disk cut with a parting tool is prepared by facing on a lathe or by grinding. In facing, the first cut is moderately heavy with a sharp tool. The second facing is a light cut with a V-shaped tool run at high speed. Specimens produced in this manner are adequate for general inspection. A better though slower method is to grind the specimen. For inspection purposes, finishing on a 120-grit wheel will be sufficient. If machine grinding is used, the specimen should be of a size that can be held conveniently in one hand. The limiting size in machine grinding is usually 12 inches square since larger specimens are difficult to handle in the etching bath.

3.5.1.1 When the maximum amount of detail is required, as in weldments, polishing the specimen with the series of metallographic papers gives the best results. When examining for surface defects, the surface itself should be etched directly without much preparation. The only preparation that is advisable is to brush off the loose scale and then to give the specimen a light grinding pass with very coarse abrasive to break through the adherent scale. When etching in 1+1 HCl for example, this scale will be removed, exposing the surface underneath. If care has been exercised in the grinding operations, the grinding scratches will not interfere with examination.

3.5.1.2 The most commonly used solutions for macroetching iron and steel are Solution Nos. 1 and 3 in Table 5.

3.5.2 Forgings — In addition to the examination for internal structure, surface defects, and structure, closed die forgings are often sectioned to show flow lines. Etching for flow lines require extremely careful preparation to provide a smooth surface with a minimum of cold work. Long pieces, such as crank shafts, are awkward to handle and are best prepared on a grinding machine using successively finer grinding wheels. Sectioning into shorter lengths may be advisable. The specimen should be heavily etched in 1+1 HCl or 20 percent H<sub>2</sub>SO<sub>4</sub>. Contrast can often be increased by wiping the surface lightly with very fine metallographic paper after etching. Examination for structure, defects, etc. is carried out in the same fashion as hotmill products.

3.5.3 Special Tests for Segregation — There are a number of etchants containing copper salts which will reveal segregation. Careful specimen preparation through the metallographic papers is required. Very careful cleaning after grinding is extremely important. When a specimen is immersed in this type of solution, copper plates out into the specimen by a replacement reaction. The rate of deposition depends on the composition of the steel. The copper plating will cover the segregated regions. Sometimes the specimen can be left in a little longer than recommended and then rubbed lightly with metallographic papers to increase contrast.

3.5.4 Macroetchants for Iron and Steel (Table 5).

### 3.6 Stainless Steels and High-Temperature Alloys:

3.6.1 These alloys are generally more susceptible to cold working of the surface than are the lower alloy grades of steel. The best method of preparation is to grind the specimens as described for iron and steel. A smut tends to form on the surface of the steel when immersed in 1+1 HCl. This can be prevented by adding a small quantity of HNO<sub>3</sub> to the etching bath. It can also be removed by scrubbing the specimen with a vegetable fiber brush under running warm water or by immersion in warm 20 percent HNO<sub>3</sub>. Scrubbing will provide a higher contrast for detection of segregation and inclusions. The desmutting, either by the addition of HNO<sub>3</sub> to the etching bath or by the secondary rinse in HNO<sub>3</sub>, will provide a brighter surface which is suitable for determination of grain size and structure. High-alloy stainless steels and austenitic high-temperature alloys, because of their extreme corrosion resistance, often will give trouble in etching. Aqua regia, HCl-H<sub>2</sub>O<sub>2</sub>, and

Marble's reagent are the recommended etchants. All three of these require very careful specimen preparation.

3.6.2 Macroetchants for Stainless Steels and High-Temperature Alloys (Table 6).

### 3.7 Lead and Lead Alloys:

3.7.1 Lead and its alloys are among the most difficult metals to prepare for macroetching. They are not only very soft and cold work easily but they (pure lead especially) recrystallize readily at temperatures which can be easily achieved in careless preparation.

3.7.2 For best results in the macroetching of lead, all surfaces, other than that to be examined, must be masked from the macroetch by the use of several coats of a plastic spray. The surface to be examined should be filed prior to etching. Three 14-in. files<sup>3</sup> are usually required and used in the following order: (1) aluminum, Type A, (2) hand smooth, and (3) hand-finishing smooth.

3.7.3 The file is usually held in a fixture and the specimen is drawn over the file proceeding from the point of the file to the tang. Remove the filings after each pass of the specimen with a few short strokes of a brass file brush in the direction of the last cut of the file. The molybdate etch listed below is used in the removal of worked metal and also to reveal the structure of lead of low-alloy content. The preparation of the solution is of paramount importance.

3.7.4 Macroetchants for Lead and Lead Alloys (Table 7).

### 3.8 Magnesium:

3.8.1 Features detected by macroetching are grain size, segregation of intermetallic compounds, coring, cracks, porosity, laps, germinations, surface burning, and tears.

3.8.2 Cast or wrought magnesium alloys are prepared in a similar manner to aluminum or copper and brass. However, a final facing with a 0.005-in. radius V-shaped tool fed at a rate of 0.002 to 0.003-in. is often sufficient. For some applications, the specimen may be finished on a 400-grit wet wheel. To resolve small detail, additional polishing with a water suspension of 600 Alundum or further with alpha alumina may be necessary.

3.8.3 The finely divided magnesium as chips or swarf is highly combustible and precautions against it catching fire should be taken.

3.8.4 Macroetchants for Magnesium and Magnesium Alloys (Table 8).

### 3.9 Nickel and Nickel Alloys:

3.9.1 Nickel alloys cold work easily and their preparation is not always easy. Grinding produces the best specimens. Low-nickel and cobalt alloys of basically pure metal can be etched with Marble's reagent or strong HNO<sub>3</sub> solutions. The high-temperature alloys are difficult to etch. First of all, they are subject to cold-working problems, and secondly, the alloys are extremely corrosion-resistant. Best results have been obtained with aqua regia, modified Marble's reagent, or HCl-H<sub>2</sub>O<sub>2</sub> solutions.

3.9.2 Macroetchants for Nickel and Nickel Alloys: (Table 9).

3.10 Noble Metals —  
Ag, Au, Ru, Rh, Pd, Os, Ir, and Pt:

3.10.1 These metals are, in general, soft and ductile. Because of their expense, specimens for macroetching usually will be small and can be handled the same as microspecimens. Care should be used to avoid cold work. Well lubricated metallographic papers are recommended. Some of the platinum group metals, notably osmium and rhodium, are more abrasion-resistant than their hardness would indicate and, therefore, will require long grinding time.

3.10.1.1 Except for silver, all of these metals are corrosion-resistant and require the use of strong etchants. Etching should be done under a fume hood, with the proper precautions for the use of HF.

3.10.2 Macroetchants for the Noble Metals (Table 10).

3.11 Refractory Metals —  
Cr, Mo, W, V, Cb, and Ta:

3.11.1 The above six metals, the refractory metals, are found in Group V-B (V, Cb, Ta) and Group VI-B (Cr, Mo, W) of the periodic table. (Some tables list them as V-A and VI-A). In general, these metals are soft and ductile in the pure state, but, in the form usually encountered, are hard and brittle. Consequently, these metals and their alloys must be carefully ground before macroetching. The abrasives must be of sufficient hardness, and the particles on the laps must be sharp. Wet silicon carbide papers have proved satisfactory provided they are used with sufficient pressure to effectively cut the sample and are not used after they dull or worn. Longer grinding times than would be expected from the hardness of these metals and alloys are needed. The following solutions used for the metals specified will reveal defects, general structure, grain size, and segregation.

<sup>3</sup> Nicholson designations. Be sure to preserve the distinction between hand smooth and hand-finishing smooth.

3.11.2 Macroetchants for the Refractory Metals (Table 11).

3.12 Tin and Tin Alloys:

3.12.1.1 Tin and its alloys, like lead, are difficult to prepare. Because these metals cold work easily and recrystallize at room temperature, false structures are easily produced. The best method of preparation for macroetching is the same as for microetching. The specimen should be cut carefully and then ground gently on lubricated silicon carbide paper. This may be followed by polishing with 6-( $\mu\text{m}$ ) diamond paste on a moderately napped wheel.

3.12.1.2 Precision cast tin-base bearing alloys may be etched directly without preparation.

3.12.2 Macroetchant for Tin and Tin Alloys (Table 12).

3.12.3 Preparation of Ammonium Polysulfide — Pass  $\text{H}_2\text{S}$  gas into 200 ml of  $\text{NH}_4\text{OH}$  (sp. gr. 0.9) until saturated. The solution should be kept in an ice bath during this operation. Add an additional 200 ml of  $\text{NH}_4\text{OH}$  (sp. gr. 0.9) and dilute with water to make 1 liter. Add 100 g of sulfur. Stir occasionally over a period of 1 hour, then filter and use.

3.13 Titanium, Zirconium, Hafnium and their Alloys:

3.13.1 Titanium, zirconium, and hafnium, the reactive metals, are macroetched for general structure, grain size, and segregation of impurities. They require extreme care in

preparation. Sharp tools and fresh grinding paper are required to prevent cold work from blurring the structure. The best results are obtained by grinding. These metals grind slowly and require sharp, fresh abrasives. The recommendations of grinding wheel manufacturers should be followed closely for rough grinding. Silicon carbide papers, usually run wet, will give a fine finish. Papers should be discarded frequently to prevent loading. Chemical polishing preparations, while relatively untried, may have decided advantages in handling these materials.

3.13.2 Solutions in the table are not difficult to prepare but involve the use of hydrofluoric acid. This acid can give extremely serious, very painful burns. The  $\text{HCl-HF}$  solution requires desmutting. The specimen should be rinsed between etching and desmutting (Table 13).

3.14 Zinc and Zinc Alloys:

3.14.1 Zinc and its alloys cold work rapidly and recrystallize at low temperatures, thus allowing false structures to form easily. Coarse-grained zinc and zinc alloys are more prone to cold work than some of the fine grained die-casting alloys. A good test for the presence of cold work, especially in coarse-grained samples is the appearance of twinning after etching.

3.14.2 Samples should be cut with a sharp saw and ground on well lubricated silicon carbide papers at slow speeds.

3.14.3 Macroetchant for Zinc and Zinc Alloys (Table 14).

TABLE 1 MACROETCHANTS FOR ALUMINUM AND ALUMINUM ALLOYS

Alloy	Composition	Procedure	Comments
All	NaOH ..... 10 g H <sub>2</sub> O ..... 100 ml	Immerse sample 5 - 15 min. in solution heated to 60° to 70°C (140° to 160°F). Rinse in water, and remove smut in strong HNO <sub>3</sub> solution. Rinse and repeat etching if necessary.	Good general - purpose etchant, can be used on almost all aluminum alloys. Does not require fine grinding.
3XXX 4XXX 5XXX 6XXX High Si castings	HCl(conc) ..... 75 ml HNO <sub>3</sub> (conc) ..... 25 ml HF (48 percent) ..... 5 ml	Mix fresh before using. Use at room temperature. May be used as immersion etch or swabbed over specimen surface. Rinse specimen in warm water and dry.	Used to develop grain structure. May be diluted with 25 per cent water to slow down etching. Does not require fine grinding.
High purity Al 1XXX 3XXX 4XXX 5XXX 6XXX	HCl (conc) ..... 45 ml HNO <sub>3</sub> (conc) ..... 15 ml HF (48 percent) ..... 15 ml H <sub>2</sub> O ..... 25 ml	Immerse specimen at room temperature until desired contrast is developed. Rinse in warm water and dry.	Tucker's etch. General purpose etch for revealing microstructure of both cast and wrought aluminum. Does not require fine grinding.
All except high Si castings	HCl (conc) ..... 15 ml HNO <sub>3</sub> (conc) ..... 5 ml HF (48 percent) ..... 5 ml H <sub>2</sub> O ..... 75 ml	Same as above.	1+2 Tucker's Same as above but slower acting.

Table 1 — continued

2XXX High Cu alloys	HCl (conc) ..... 15 ml HF (48 percent) ..... 10 ml H <sub>2</sub> O ..... 90 ml	May be used as an immersion etch or swabbed over the specimen surface. When desired contrast is obtained, rinse in water and remove deposits with concentrated HNO <sub>3</sub> . Rinse in warm water and dry.	Flick's reagent. Best results are obtained with a ground surface. 180 grit will suffice.
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TABLE 2 — MACROETCHANTS FOR BERYLLIUM AND BERYLLIUM ALLOYS

Metal	Composition	Procedure	Comments
Be	1. HCl ..... 10 ml NH <sub>4</sub> Cl ..... 4 g H <sub>2</sub> O ..... 90 ml	Either swab or immerse at room temperature for a few minutes, rinse in water and dry.	Works best on coarse grained Be.
Be	2. HCl ..... 10 ml NH <sub>4</sub> Cl ..... 2 g Picric acid ..... 2 g H <sub>2</sub> O ..... 90 ml	As above.	An alternative when No. 1 does not work. Fine-grained metal may not give good results in either case.

TABLE 3 — MACROETCHANTS FOR COBALT AND COBALT ALLOYS

Alloy	Composition	Procedure	Comments
49Co-49Fe-V Some Co-Cr alloys	HCl ..... 50 ml H <sub>2</sub> O ..... 50 ml	Immerse specimen in hot solution (140° to 180° F) for 30 to 60 min. Rinse in hot water and dry.	General structure, porosity.
25Cr-10Ni-8W 21Cr-20Ni 3W-3Mo-1Cb	HCl ..... 50 ml HNO <sub>3</sub> ..... 10 ml FeCl <sub>3</sub> ..... 10 g H <sub>2</sub> O ..... 100 ml	Swab until desired contrast is obtained then rinse in warm water and dry.	Grain size, general structure.
18Cr-10Ni-14W	CuCl <sub>2</sub> ·2NH <sub>4</sub> Cl·2H <sub>2</sub> O ... 2 g FeCl <sub>3</sub> ..... 5 g HNO <sub>3</sub> ..... 5 ml HCl ..... 50 ml H <sub>2</sub> O ..... 80 ml	As above.	As above.

TABLE 4 — MACROETCHANTS FOR COPPER AND COPPER ALLOYS

Alloys	Composition	Procedure	Comments
Cu and all brasses	1. HNO <sub>3</sub> ..... 10 ml H <sub>2</sub> O ..... 90 ml	Immerse specimen in solution at room temperature for a few minutes. Rinse in water and dry.	Emphasize grains and cracks.
Cu and all brasses	2. HNO <sub>3</sub> ..... 50 ml H <sub>2</sub> O ..... 50 ml	As above.	Brings out grain contrast, pits result unless agitated. Aluminum bronzes may form smut which can be removed by brief immersion in concentrated HNO <sub>3</sub> .
Cu and all brasses	3. HCl ..... 30 ml FeCl <sub>3</sub> ..... 10 g H <sub>2</sub> O or ethanol ... 120 ml	As above.	Good grain contrast.
Cu, high Cu alloys, phosphorus, tin bronzes	4. K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> , sat solution of NaCl H <sub>2</sub> SO <sub>3</sub> H <sub>2</sub> O	Immerse specimen in solution at room temperature for 15 to 30 min. then swab with fresh solution. Rinse in warm water and dry.	Emphasizes grain boundaries and oxide inclusions.
All	5. HNO <sub>3</sub> ..... 50 ml AgNO <sub>3</sub> ..... 5 g H <sub>2</sub> O ..... 50 ml	Immerse specimen in solution temperature. Rinse in warm water and dry.	Brilliant deep etch.
Brass	6. 20 percent ..... 20 ml acetic acid 5 percent ..... 10 ml chromic acid 10 percent ..... 5 ml FeCl <sub>3</sub> in H <sub>2</sub> O	As above.	Strain lines.
Silicon brass or bronze	7. CrO <sub>3</sub> ..... 40 g NH <sub>4</sub> Cl ..... 7.5 g HNO <sub>3</sub> (conc) ..... 50 ml H <sub>2</sub> SO <sub>4</sub> (conc) ..... 8 ml H <sub>2</sub> O ..... 100 ml	Immerse specimen in solution at room temperature, rinse in warm water and dry.	



TABLE 5 — MACROETCHANTS FOR IRON AND STEEL

Alloys	Composition	Procedure	Comments
Plain and alloy steels, high speed and tool steels, cutlery (12 to 14% Cr) and stainless steels	1. HCl (conc)..... 50 ml H <sub>2</sub> O ..... 50 ml	Immerse specimen in solution heated to 160° to 180°F for 15 to 30 min. Desmut by vigorous scrubbing with vegetable fiber brush under running water. Stainless steels may be desmutted by dipping in a warm 20 percent HNO <sub>3</sub> to give a bright finish.	General purpose.
High alloy steels	2. HCl (conc)..... 50 ml HNO <sub>3</sub> (conc) ..... 25 ml H <sub>2</sub> O ..... 25 ml	Immerse specimen for 10 to 15 min. in solution at room temperature. Rinse in warm water and dry.	Ratio HCl:HNO <sub>3</sub> runs 2:1 to 3:1.
Plain and alloy steels, cutlery steels	3. HCl (conc) ..... 38 ml H <sub>2</sub> SO <sub>4</sub> (conc) ..... 12 ml H <sub>2</sub> O ..... 50 ml	Immerse specimen for 15 to 45 min. in solution heated to 160° to 180°F. Rinse in warm water and dry.	Works well on 12 per cent Cr steel.
High alloy steels	4. HNO <sub>3</sub> (conc) ..... 10 ml HF (48 percent) ... 4 ml H <sub>2</sub> O ..... 87 ml to HNO <sub>3</sub> (conc) ..... 40 ml HF (48 percent) .. 10 ml H <sub>2</sub> O ..... 50 ml	Immerse specimen in solution heated to 160° to 180°F until desired etch is obtained and rinse in warm water and dry.	Ratio HNO <sub>3</sub> -HF varies.
Stainless steels, high-alloy steels	5. HCl (conc) ..... 50 ml H <sub>2</sub> O ..... 50 ml H <sub>2</sub> O <sub>2</sub> (30 percent) 20 ml	Mix HCl and water then heat to 160° to 170°F, immerse specimen and add H <sub>2</sub> O <sub>2</sub> in several parts. Do not mix. Make each subsequent addition after foaming from previous addition has stopped.	Produces bright finish.
Austenitic stainless steels	6. HCl (conc)..... 50 ml saturated solution CuSO <sub>4</sub> in H <sub>2</sub> O <sub>2</sub> ... 25 ml	Immerse specimen in solution which may be heated or not depending upon alloy. Time also depends on alloy. Rinse in warm water and dry.	Marble's reagent. Light etch, good for structure.
Plain and low-alloy steels	7. (NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub> ..... 10 g (ammonium persulfate) H <sub>2</sub> O ..... 100 ml	Swab solution at room temperature over specimen. Rinse and dry.	Grain size, weldments.
Plain and alloy steels	8. CuCl <sub>2</sub> ..... 2.5 g MgCl <sub>2</sub> ..... 10 g HCl (conc) ..... 5 ml Alcohol — up to . 250 ml	Immerse in solution at room temperature until a coppery sheen appears. Rinse thoroughly and dry.	Stead's reagent. Salts dissolved in HCl with minimum of hot water to bring out P-rich areas and P-banding.
Mild steel. Besemer and high N <sub>2</sub> steel	9. CuCl <sub>2</sub> ..... 90 g HCl (conc) ..... 120 ml H <sub>2</sub> O ..... 100 ml	The surface should be rubbed with cloth soaked in etching solution. Wash in alcohol or rinse in HCl (1 + 1) after etching to prevent deposition of copper.	Fry's reagent. Before etching, sample should be heated to 200 to 250 C (302° to 482°F) for 5 to 30 min. depending on condition of steel. To show strain lines due to cold work.
Plain and alloy steels	10. CuCl <sub>2</sub> ..... 45 g HCl (conc) ..... 180 ml H <sub>2</sub> O ..... 100 ml	As above.	Modified Fry's reagent. Same as for reagent No. 9 but modified by Wazau, may give more contrast, specimen can be washed in water without depositing copper.
Stainless and high Cr steels	11. HCl ..... 10 ml Alcohol ..... 100 ml Picric acid ..... 1 g	Immerse specimen in solution at room temperature until desired contrast is obtained. Rinse and dry.	Vilella's reagent.

TABLE 6 — MACROETCHANTS FOR STAINLESS STEELS AND HIGH-TEMPERATURE ALLOYS

Alloys	Composition	Procedure	Comments
Stainless steels and iron-base high-temperature alloys	1. HCl (conc) ..... 50 ml H <sub>2</sub> O ..... 50 ml	Immerse specimen in solution heated to 160° to 180°F for 30 min. Desmut by vigorous scrubbing with vegetable brush under running water. Stainless steels may be desmuted by dipping in warm 20 percent HNO <sub>3</sub> to give bright finish.	General purpose.
Iron, cobalt, and nickel-base high-temperature alloys	2. HCl (conc) ..... 50 ml HNO <sub>3</sub> (conc) ..... 25 ml H <sub>2</sub> O ..... 25 ml	Immerse specimen in solution at room temperature for 10 to 30 minutes. Rinse and dry.	Ratio HCl + HNO <sub>3</sub> runs 2 + 1 to 3 + 1.
Stainless steels and high-temperature alloys	3. HNO <sub>3</sub> ..... 10 ml HF (48 percent) .... 3 ml H <sub>2</sub> O ..... 87 ml to HNO <sub>3</sub> (conc) ..... 40 ml HF (48 percent) .... 10 ml H <sub>2</sub> O ..... 50 ml	Immerse specimen in solution heated to 160° to 180°F until desired contrast is obtained. Rinse and dry.	Ratio HNO <sub>3</sub> HF varies.
Austenitic stainless steels and nickel-base alloys	4. I (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> ..... 15 g H <sub>2</sub> O ..... 75 ml II FeCl <sub>2</sub> ..... 250 g HCl (conc) ... 100 ml III HNO <sub>3</sub> ..... 30 ml (conc)	Combine I and II then add III. Immerse specimen in solution at room temperature until desired contrast is obtained. Rinse and dry.	Lepito's etch. I, mix fresh, grain structure.
Austenitic stainless steels and high-temperature alloys	5. HCl (conc) ..... 50 ml H <sub>2</sub> O ..... 50 ml H <sub>2</sub> O <sub>2</sub> (30 percent) . 20 ml	Mix HCl and water then heat, immerse specimen and add H <sub>2</sub> O <sub>2</sub> in several parts. Do not mix. Make each subsequent addition after foaming from previous addition has stopped.	
Austenitic stainless steels and high-temperature alloys	6. HCl (conc) ..... 50 ml Saturated solution of CuSO <sub>4</sub> in H <sub>2</sub> O 25 ml	Immerse specimen in solution which may be heated up to 170°F until desired contrast is obtained. Rinse and dry.	Marble's reagent. Light etch, good for structures. Amount of CuSO <sub>4</sub> solution may be increased to 1 + 1 ratio for difficult alloys.

TABLE 7 — MACROETCHANTS FOR LEAD AND LEAD ALLOYS

Alloy	Composition	Procedure	Comments
Lead and lead alloys	1A. H <sub>2</sub> O ..... 250 ml NH <sub>4</sub> OH(sp gro.90) 140 ml HNO <sub>3</sub> (conc) .... 60 ml Molybdic acid .. 100 ml (85 percent)	Add A to B and let precipitate redissolve. If B is added to A an insoluble precipitate forms. Add C to mixture of A and B after precipitate has redissolved.	
	B. H <sub>2</sub> O ..... 960 ml HNO <sub>3</sub> (conc) .... 400 ml	Swab surface of the specimen with mixed solution until desired contrast is obtained.	
	C. Glacial acetic acid 100 ml	Rinse and dry.	
Antimonial lead	2A. Glacial acetic acid 30 ml HNO <sub>3</sub> (conc) .... 40 ml H <sub>2</sub> O ..... 160 ml	Prepare surface on silk velvet wheel with Al <sub>2</sub> O <sub>3</sub> abrasive at 150 rpm. Etch with solution A at 42°C then re-polish until bright. Re-etch with B at room temperature for 1 to 2 hours.	
	B. Glacial acetic acid 1 ml H <sub>2</sub> O ..... 400 ml		
	3A. HNO <sub>3</sub> (conc) .... 80 ml H <sub>2</sub> O ..... 220 ml B. (NH <sub>4</sub> ) <sub>2</sub> MoO <sub>4</sub> ..... 45 g H <sub>2</sub> O	Mix equal quantities of A and B immediately before use. Immerse specimen in solution at room temperature until desired contrast is obtained. Rinse and dry.	Grain structure.



Table 7 — continued

Alloys	Composition	Procedure	Comments
4A.	(NH <sub>4</sub> ) <sub>2</sub> MoO <sub>4</sub> ..... 10 g Citric acid ..... 25 g H <sub>2</sub> O ..... 100 ml	Immerse specimen in solution at room temperature until desired contrast is obtained, then rinse and dry.	Bright etch, grain structure, defects.
5A.	Acetic acid ..... 75 ml H <sub>2</sub> O <sub>2</sub> ..... 25 ml	Mix with strongest H <sub>2</sub> O <sub>2</sub> available to minimize water content. Immerse dry specimen in solution at room temperature until desired contrast is obtained, then rinse and dry.	Chemical polish-etch.

TABLE 8 — MACROETCHANTS FOR MAGNESIUM AND MAGNESIUM ALLOYS

Alloy	Composition	Procedure	Comments
ZK60A	1. Glacial acetic acid 5 ml H <sub>2</sub> O ..... 95 ml	Immerse specimen in solution at room temperature for 1/2 to 8 minutes until desired contrast is obtained. Desmut with 50 percent HF (48 percent) in water. Rinse in rapidly flowing water and dry.	Flow lines in forgings.
AZ61A } AZ80A }	2. Glacial acetic acid 10 ml H <sub>2</sub> O ..... 90 ml	As above but for 1/2 to 5 min.	Grain size, surface casting defects.
AZ31B } AZ61A } AZ80A }	3. Glacial acetic acid 20 ml NaNO <sub>3</sub> ..... 5 g H <sub>2</sub> O ..... 80 ml H <sub>2</sub> O ..... 80 ml	As above but for 1/4 to 5 min.	Flow pattern in forgings. Surface casting defects. Glycolic acid may be substituted for acetic acid.
AZ31B	4. HNO <sub>3</sub> (conc) ..... 10 ml H <sub>2</sub> O ..... 90 ml	As above but for 1/2 to 5 min.	Flow pattern in forgings. Internal defects in cast slabs and ingots.
	5. Na <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> ..... 180 g HNO <sub>3</sub> (conc) ..... 180 ml H <sub>2</sub> O to make ... 1000 ml	As above but rinse in hot water.	General etch for defects in sand and die castings.
	6. CrO <sub>3</sub> ..... 280 g HNO <sub>3</sub> (conc) ..... 25 ml HF (48 percent) .. 10 ml H <sub>2</sub> O to make 1000 ml	As above.	Germination on sand cast surfaces. Surface defects of die castings.
AZ61A } AZ80A }	7. 6 percent picric acid in alcohol H <sub>2</sub> O ..... 10 ml Glacial acetic acid 5 ml	As above for 1/2 to 3 min. or may be swabbed.	Grain size and flow patterns of both cast and wrought forms. Requires fine finish (600 grit).
AZ21 } AZ31 }	8. 6 percent picric acid in alcohol H <sub>2</sub> O ..... 10 ml Glacial acetic acid 10 ml	As No. 7.	Grain size. Specimen should be finished on 600-grit paper.
ZK60A	9. 6 percent picric acid in alcohol H <sub>2</sub> O ..... 20 ml Glacial acetic acid 20 ml	As No. 7.	Flow pattern and grain size of homogenous alloy. Increase water to increase strain contrast. Specimen should be finished on 600-grit paper.
	10. 4 percent picric acid in alcohol H <sub>2</sub> PO <sub>4</sub> ..... .7 ml	Immerse specimen in solution at room temperature repeatedly until desired stain is obtained, rinse and dry.	Segregation of intermetallic compounds and associated cracks. Specimen should be finished on 600-grit paper.

TABLE 9 — MACROETCHANTS FOR NICKEL AND NICKEL ALLOYS

Alloys	Composition	Procedure	Comments
Ni	1. CuSO <sub>4</sub> ..... 10 g HCl ..... 50 ml H <sub>2</sub> O ..... 50 ml	Immerse specimen in solution at room temperature until desired contrast is obtained. Rinse and dry.	Marble's reagent for grain structure.
Low-Ni alloys	2. HNO <sub>3</sub> ..... 20 ml H <sub>2</sub> O ..... 10 ml CuSO <sub>4</sub> ..... 10 g	Immerse specimen in solution at room temperature for 20 to 30 min., rinse and dry.	Grain structure.
High-Ni alloys	3. HNO <sub>3</sub> (conc) ..... 50 ml H <sub>2</sub> O ..... 50 ml	As above.	Porosity and flowlines.
Alloys containing Cr, Fe, and other elements	4. HNO <sub>3</sub> ..... 50 ml Acetic acid ..... 50 ml	Immerse specimen in hot solution. Rinse in hot water and dry.	Grain structure.
	5. Acetic acid ..... 50 ml HNO <sub>3</sub> ..... 50 ml	Swab.	Lepito's reagent II, etchant for nickel welds.
	6. Sat soln of ..... 50 ml CuSO <sub>4</sub> in H <sub>2</sub> O HCl ..... 50 ml	Swab etchant	Modified Marble's reagent.
	7. HCl ..... 100 ml H <sub>2</sub> O ..... 100 ml H <sub>2</sub> O <sub>2</sub> (30 percent) . . . 40 ml	See 3.5.4.	See 3.5.4

TABLE 10 — MACROETCHANTS FOR THE NOBLE METALS

Metal	Composition of Solution	Condition of Use	Comments and Use
Ag	HNO <sub>3</sub> ..... 10 ml Methyl alcohol ..... 90 ml	RT, few minutes.	Grain contrast.
Au Pt alloys } Pd alloys }	HCl ..... 66 ml HNO <sub>3</sub> ..... 34 ml	Hot, few minutes.	Grain contrast.
Ru and alloys } Os and alloys } Rh and alloys }	Lactic acid ..... 50 ml HNO <sub>3</sub> ..... 20 ml HF ..... 30 ml	RT, few minutes.	Grain contrast.
As above	HCl ..... 30 ml HNO <sub>3</sub> ..... 15 ml HF ..... 30 ml	RT, few minutes.	Grain contrast.
Pt and alloys	Sat solution of ..... 80 ml NaCl in H <sub>2</sub> O HCl ..... 20 ml	Electrolytic, 6V, few minutes.	Grain contrast.

TABLE 11 — MACROETCHANTS FOR THE REFRACTORY METALS

Metal	Composition of Solution	Temperature	Time	Comments
Mo, W, V, Cb, and Ta . . .	HCl (conc) ..... 30 HNO <sub>3</sub> (conc) ..... 15 HF (48 percent) ..... 30	RT	5 to 20 minutes.	
Mo, W, and V . . . . .	HF (48 percent) ..... 15 HNO <sub>3</sub> (conc) ..... 35 H <sub>2</sub> O ..... 75	RT	10 to 20 minutes.	
W, V, Cb, and Ta . . . . .	HF (48 percent) ..... 10 HNO <sub>3</sub> (conc) ..... 30 Lactic acid (85 percent) . . . 50	RT	1 to 40 minutes.	Too fast for Mo, 1 to 5 seconds.
Cr . . . . .	H <sub>2</sub> SO <sub>4</sub> ..... 10 H <sub>2</sub> O ..... 90	Boiling	2 to 5 minutes.	

TABLE 12 — MACROETCHANTS FOR TIN AND TIN ALLOYS

Composition of Solution	Procedure	Comments
Saturated ammonium polysulfide	Immerse specimen in full strength solution at room temperature for 20 to 30 min. Do not swab surface during etching.	Grain structure.

TABLE 13 — MACROETCHANTS FOR TITANIUM, ZIRCONIUM,  
HAFNIUM AND THEIR ALLOYS

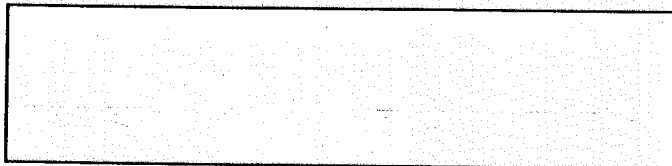
Alloy	Composition	Procedure	Comments
Ti alloys	1. HCl (conc) . . . . . 20 ml HF (48 percent) . . . 40 ml H <sub>2</sub> O . . . . . 50 ml	Immerse specimen in solution heated to 120° to 150°F for 20 to 30 min. Rinse. If smut forms immerse in 30 percent H <sub>2</sub> SO <sub>4</sub> for 3 minutes. Rinse and dry.	Desmut Ti, -13V-11 Cr-3Al alloys. Others do not normally need desmutting.
7Al-4Mo alloys	2. HNO <sub>3</sub> (conc) . . . . . 42 ml HF (48 percent) . . . . 8 ml H <sub>2</sub> O . . . . . 50 ml	As above.	
Zr, Hf, and low alloys	3. H <sub>2</sub> O <sub>2</sub> (30 percent) . 45 ml HNO <sub>3</sub> (conc) . . . . . 45 ml HF (48 percent) . . . 10 ml	Swab specimen with solution at room temperature. Rinse 10 sec. after yellow fumes form, then dry.	Should be used under a hood; chemical polishing solution.
Zr, Hf, high alloys	4. H O . . . . . 45 ml HNO <sub>3</sub> . . . . . 45 ml HF . . . . . 10 ml	As above.	As above.
1.5Sn-0.15Fe-0.10Cr, and Hf	5. H <sub>2</sub> O . . . . . 70 ml HNO <sub>3</sub> (conc) . . . . . 30 ml HF (48 percent) . . . . 5 ml	As above.	As above.
Iodide Ti	6. H <sub>2</sub> O <sub>2</sub> (30 percent) . 60 ml H <sub>2</sub> O . . . . . 30 ml HF (48 percent) . . . 10 ml	Swab with solution at room temperature until desired contrast is obtained. Rinse in cold water and dry.	As above.

TABLE 14 — MACROETCHANTS FOR ZINC AND ZINC ALLOYS

Alloy	Solution Composition	Procedure	Comments
Cu-free zinc alloys	HCl (conc) . . . . . 50 ml H <sub>2</sub> O . . . . . 50 ml	Immerse in solution for about 15 sec. Remove smut by wiping under running water. Repeat until desired contrast is obtained, then dry.	Grain structure.
Zn alloys containing Cu	CrO <sub>3</sub> . . . . . 20 g Na <sub>2</sub> SO <sub>4</sub> . . . . . 1.5 g or Na <sub>2</sub> SO <sub>4</sub> •10H <sub>2</sub> O . . . . . 3.4 g H <sub>2</sub> O . . . . . 100 ml	Immerse in solution until good contrast is obtained. Rinse in running water and dry. Repeat if necessary.	Grain structure.



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