Metallography Principles and Procedures

LECO

EMPOWERING RESULTS

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Metallographic Sample Preparation

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A

Metallography Principles and Procedures

LECO Corporation would like to thank Cornelius A. Johnson for his contributions to this project.

Metallographic Sample Preparation

Introduction

Metallography—the study of the microstructure of metals using various techniques—has been an invaluable tool for the advancement of science and industry for over one hundred years. Metallography is used to reveal the microstructure of metals, which is affected by alloy composition and processing conditions; including cold working, heat treatment and welding. A finished part's environment can also affect its microstructure and cause problems such as corrosion and decarburization.

Analysis of a material's metallographic microstructure aids in determining if the material has been processed correctly and is therefore a critical step for determining product reliability and/or for determining why a material failed.

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 meet the following criteria.

- *Be flat and free from scratches, stains, and other imperfections which tend to mar the surface.*
- *Contain all non-metallic inclusions intact.*
- *Show no chipping or galling of hard and brittle intermetallic compounds.*
- *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 (Definitions)

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 hot thermosetting powder or cold castable mounting material 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/Pre-Polishing

Removing the zone of deformation caused by Sectioning and Coarse Grinding. The depths of deformation during this stage need to be limited 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 as 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) 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. See Figure 1, which shows possible damage traceable to improper sectioning techniques.

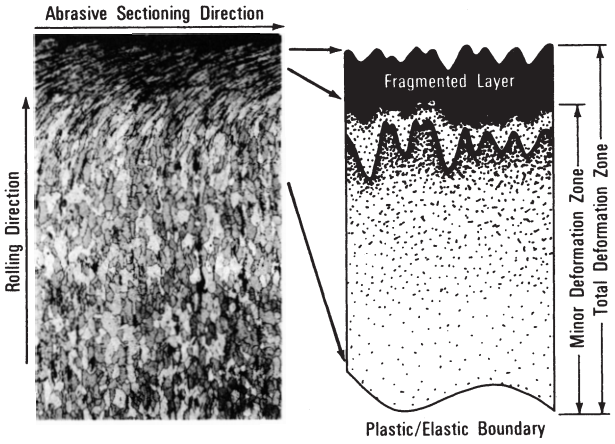


Figure 1

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 (Figure 2). Many intermediate strain boundaries extend in rays from the "V" contours of the scratches.

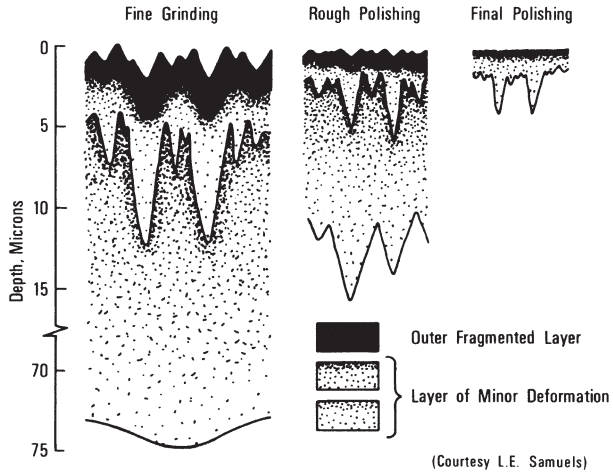


Figure 2

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 affects the magnitude of the strain boundary levels. Scratch depth and total zone deformation can be considered inversely proportional to an increase in material hardness (Figure 2A).

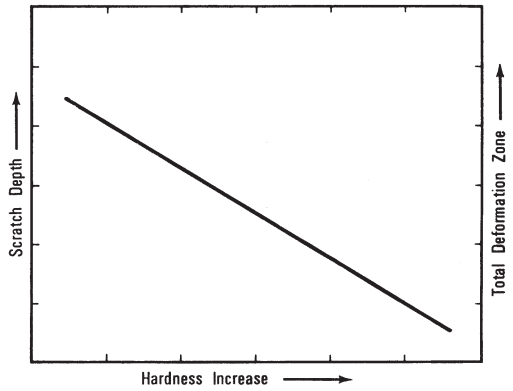


Figure 2A

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 (Figure 3).

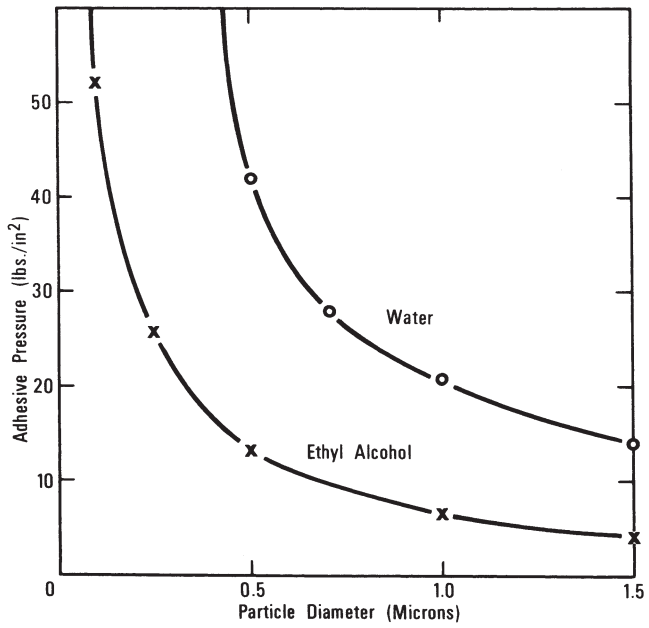


Figure 3 (Courtesy of C. Johnson & J. M. Parks)

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 (Figure 4).

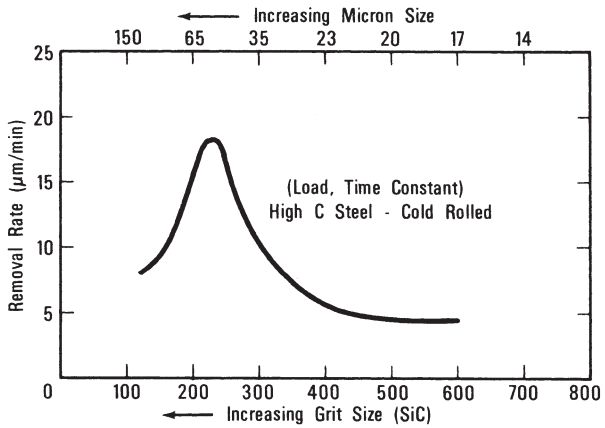


Figure 4

The coarse size range (50 to 180 grit; 70 to 350 microns) of abrasive materials commonly used 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 (Figures 5 and 6).

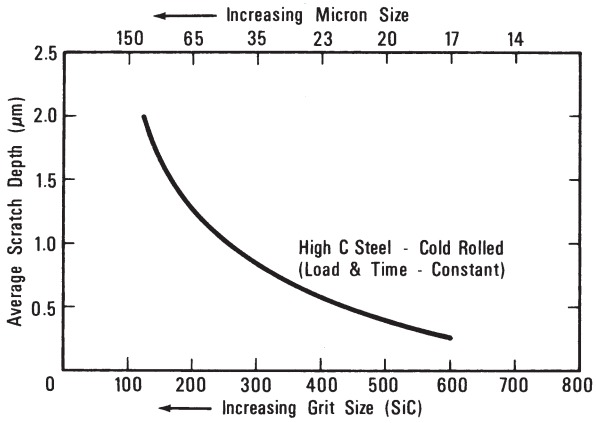


Figure 5

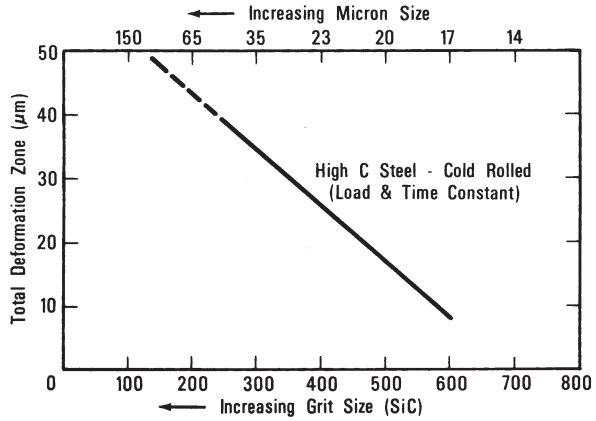


Figure 6

A

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 through IV.

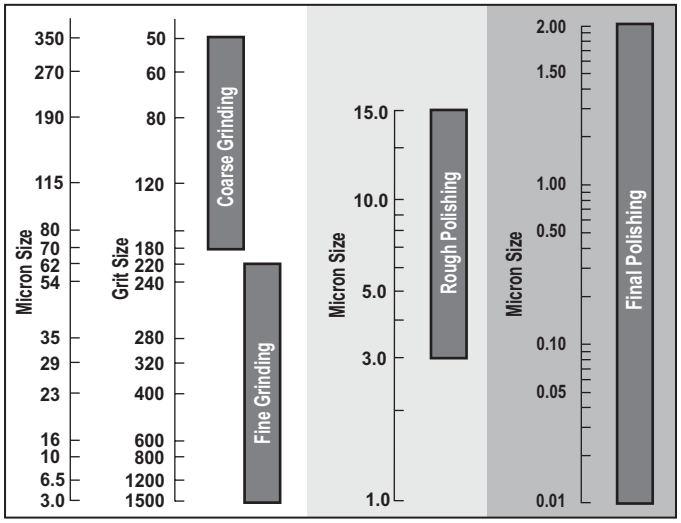


Figure 7

Table I—Grit Comparison Guide

CAMI Designation (USA)	FEPA* Designation (Europe)	Trizact Finishing Scale	Average Particle Size in Inches	Average Particle Size in Microns
—	P24	—	0.02913	740
24	—	—	0.02815	715
30	—	—	0.02512	638
—	P30	—	0.02449	622
36	—	—	0.02106	535
—	P36	—	0.02063	524
40	—	—	0.01685	428
—	P40	—	0.01622	412
50	—		0.01382	351
—	P50	—	0.01283	326
60	—	—	0.01055	268
—	P60	—	0.01024	260
—	P80	—	0.00776	197
80	—	—	0.00756	192
—	P100	A200	0.00614	156
100	—	—	0.00555	141
—	P120	A160	0.00500	127
120	—	—	0.00457	116
—	P150	—	0.00382	97
150	—	A130	0.00366	93
180	P180	A110	0.00307	78
220	—	A85	0.00260	66
—	P220	A90	0.00256	65
—	—	—	0.00236	60
—	P240	A75	0.00230	58.5
—	—	—	0.00217	55
240	—	A65	0.00211	53.5
—	P280	—	0.00207	52.5
—	—	—	0.00197	50
—	P320	A60	0.00182	46.2
—	—	—	0.00177	45
—	—	—	0.00173	44
—	P360	—	0.00159	40.5
—	—	—	0.00157	40
320	—	—	0.00142	36
—	P400	A45	0.00138	35
—	P500	—	0.00119	30.2
—	—	—	0.00118	30
360	—	—	0.00113	28.8
—	P600	A35	0.00101	25.75
—	—	—	0.00098	25
400	—	A30	0.00093	23.6
—	P800	A25	0.00086	21.8
—	—	—	0.00079	20
500	—	—	0.00078	19.7
—	P1000	—	0.00072	18.3
600	—	—	0.00063	16
—	P1200	A16	0.00060	15.3
—	P1500	—	0.00050	12.6
800	—	—	0.00048	12.2
—	P2000	—	0.00041	10.3
1000	—	—	0.00036	9.2
—	P2500	—	0.00033	8.4
1200	—	—	0.00026	6.5
1500		—	0.00012	3.0

*Federation of European Producers of Abrasives

Table II—Emery

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

Table III—Diamond Pastes

Micron Size* (μm)	Size Range (μm)	Mesh Equivalent (Approx.)
1/10**	0-1/0	—
1/4**	0-1/2	100,000
1/2	0-1	60,000
1	0-2	14,000
3	2-4	8,000
6	4-8	3,000
9	8-12	1,800
15	12-22	1,200
30	22-36	600
45	36-54	325
60	54-80	230-325
90***	—	170-230

*NIST

**Ultra fine grades, not covered by NIST

***Not covered by NIST

Table IV A—Polishing Powders

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

Table IV B—Polishing Suspensions

Type	Particle Size (μm)	Application
Levigated Alumina	5.0	Rough Polishing
Gamma Alumina	0.05	Final Polishing
Alpha Alumina	0.3	Rough and/or Final Polishing
Chrome Oxide	1.0	Rough Polishing
Chrome Oxide	0.05	Final Polishing
Chrome Oxide*	0.05	Final Polishing
Cerium Oxide**	0.05	Final Polishing
Colloidal Silica	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.

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

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

b) ***Bond***
Rubber
Resinoid
Resin/Rubber

c) ***Hardness of Bond***
Soft
Medium
Hard

d) ***Density (Structure)***
Open
Dense

Abrasive Grain

Aluminum Oxide is generally recommended for sectioning steels and high tensile strength materials. Silicon Carbide abrasives are used for some grades of iron, nonferrous 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

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 solids, sample standards, and tubing of medium wall thickness.

Concave edges indicate proper wheel for sectioning light wall tubing of thin wall sections.

A

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

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

Pointed edges indicate wheel bond is too hard.

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 or CBN rimmed wheels are used. The speed range is 0 to 1000 RPM and the load range is 0 to 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. Diamond structured fixed abrasives and spot pattern discs are also for coarse grinding applications.

The abrasive action is very aggressive with this grit range. With higher speed coarse grinding (i.e. increased surface feet per minute), the resulting 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 that 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. Pre-polishing diamond structured discs with 3 to 15 μ diamond suspensions are also utilized for this stage preparation. Generally, fixed type abrasives are used (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 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, which can correspondingly affect deformation depth.

Stage 5—Rough Polishing

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

Abrasives

Diamond abrasives fall into the above category. Other contributing properties are high hardness, inertness, and low coefficient of 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 (Figures 8 and 9).

Suspension Medium

The suspension medium for diamond powders is very important as it provides particle suspension and 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 the surface.

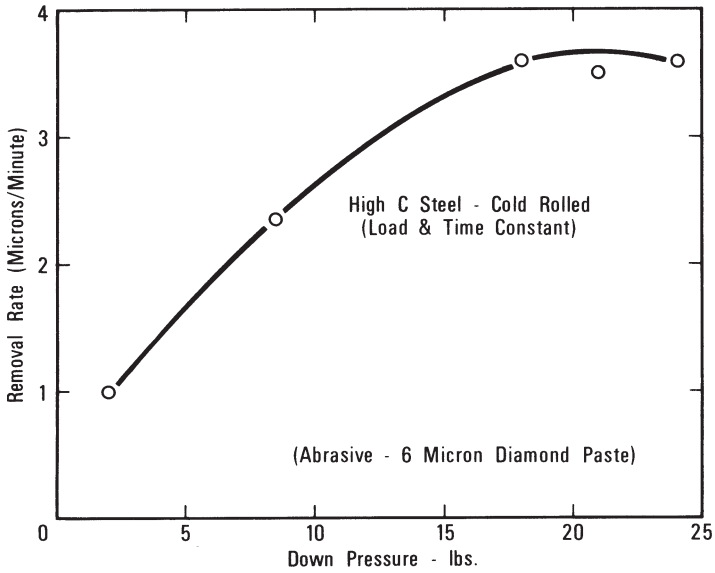
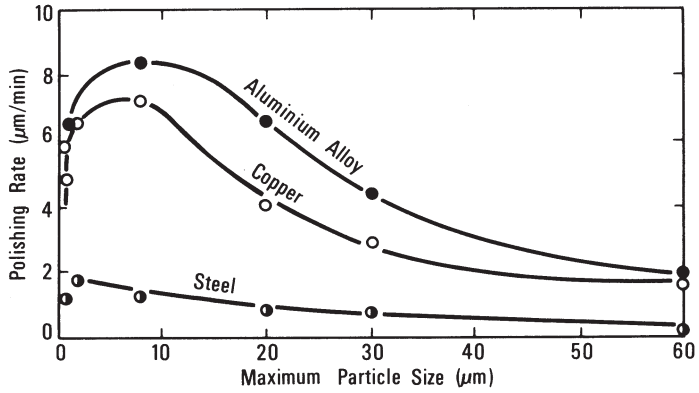


Figure 8



(Courtesy L.E. Samuels)

Figure 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 to10 microns. The 6 micron (range 4 to 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 (Figure 9).

As previously stated, removal rates will increase linearly with pressure to a critical point (Figure 8).

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, silk, chemotextile materials, etc., should be used. Cloths of this nature will hold relief and undercutting at interfaces to a minimum, as "pile whip" is non-existent. Selection should also be such that the cloth itself does not produce any abrasive artifacts. The hardness of the material being prepared is the guide point.

Stage 6—Final Polishing

As previously stated, the final polishing stage serves to remove any deformation zone resulting from rough polishing. Here, a uniformly polished and scratch-free surface must be produced.

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

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

A 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 the 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.

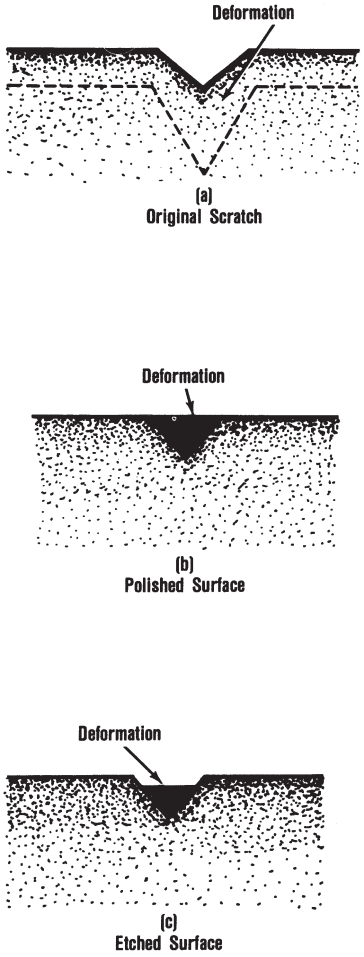


Figure 10

Abrasives

A wide variety of abrasive materials are used for final polishing. The most common are Aluminum Oxide, Chromium Oxide, Magnesium Oxide, Cerium Oxide, Colloidal Silica, and Diamond.

Aluminum Oxide is the most extensively used material. Two types are available—levigated and high-purity synthetic powders. Preference is for the synthetic materials in either powder or suspension form within the 1 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 material, the alpha form may be profitably used as an intermediate step.

Other commonly used media are liquid suspensions of chromium oxide, and chromium 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 (Silica) have been used with remarkable success in the electronic wafer industry. Colloidal silica is an excellent final polishing media for ferrous, nonferrous, and electronic components.

Diamond abrasives have several sizing levels below the 4 to 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 grading 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 of 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 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. For example, water must be avoided when polishing galvanized steel samples.

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 that 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 luster.

When using diamond abrasives, improved removal rates are encouraged by low viscosity oil extenders or ethanol-based extenders (Ultralap). 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 (Figure 11). When manual dexterity has been achieved, motion in both directions may be employed.

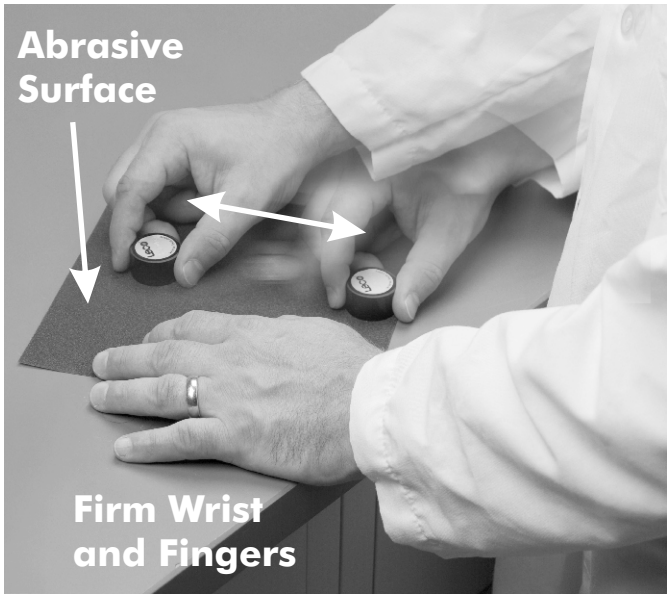


Figure 11

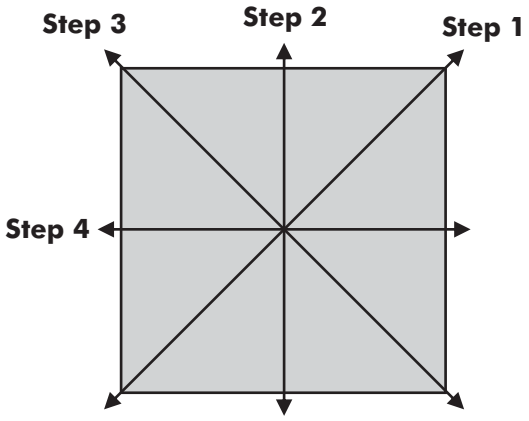
The operator should be positioned to allow free passage of the elbow past the side of the body for linear coordination between the shoulder and elbow joints.

Finger and wrist joints should remain rigid, and the 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 (Figure 12A).

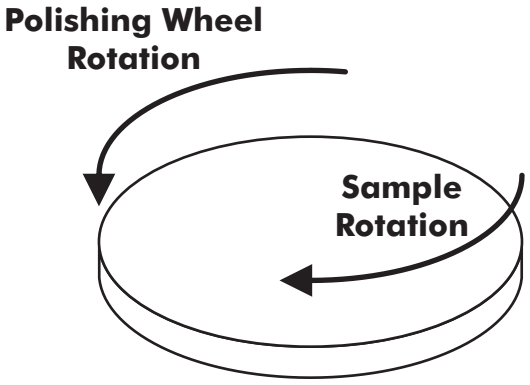
Rough and Final Polishing

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 directional traces; "fishtailing" of certain family type inclusions; and/or "pull-out" of phases poorly consolidated within a microstructure. Such manipulation also provides equal material removal over the entire surface (Figure 12B).



(a)

Figure 12A



(b)

Figure 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 the use of a glass beaker, and positioning the 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 (Figure 13).

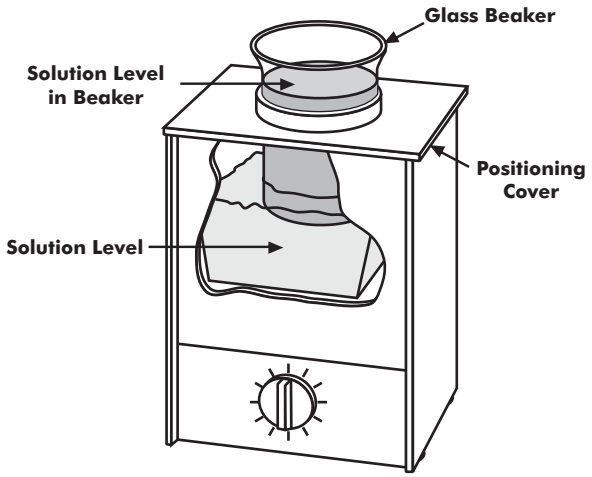


Figure 13

Cold Water and Cotton Balls

An alternative to ultrasonic cleaning (if not available) is cleaning using cold water and a cotton ball. Place the sample or sample holder underneath cold running water, saturate a 100% cotton ball with cold water, and clean by rubbing the sample or samples. Rinse thoroughly with water, give a final rinse with ethyl alcohol, and dry. Use of 100% cotton balls will not scratch the sample; *synthetic cotton will*.

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.

A

Mounting Procedures

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B

Mounting Procedures

Specimen Mounting (Purposes)

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 amount of time. The materials used are classified as thermosetting and 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.

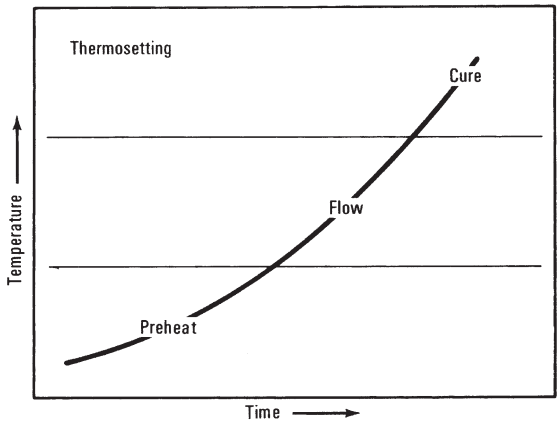


Figure 15

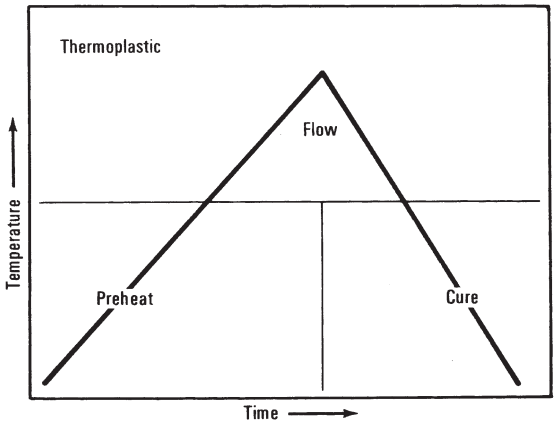


Figure 16

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

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.

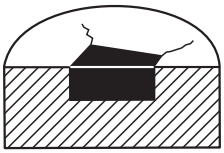
The material readily flows into small areas. This factor, plus the allowable pressure variances, make the material very desirable for small, fragile pieces. These possibilities very often offset the longer times involved in molding transparent mounts.

Table V—Molding Temperatures and Pressures

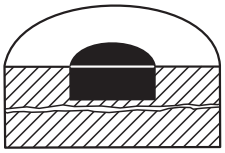
Material	Form	Classification	Molding Temp (°F)	Molding Pressure (psi)
Bakelite®	Powder	Thermosetting	280-320°	3800-4400
Bakelite	Preform	Thermosetting	280-320°	3800-4400
Diallyl-Phthalate[1]	Powder	Thermosetting	300-360°	3800-4400
Epoxy	Powder	Thermosetting	280-320°	3800-4400
Lucite (transparent)	Powder	Thermoplastic	280-320°	3800-4400

[1] *Including Copper Diallyl Phthalate for conductive applications*

Thermosetting Molding Defects

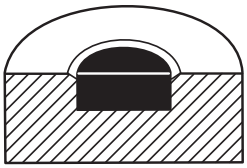


Split (Radial): Section too large for mold area. Sharp corners on specimen. Use larger mold size. Reduce specimen size and eliminate sharp corners if possible.

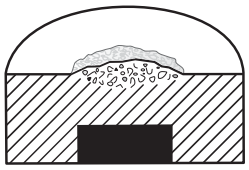


Split (Circumferential): Material has absorbed moisture. Gases released by chemical reaction. Use preheated powders or premolds. Momentarily release pressure during flow stage.

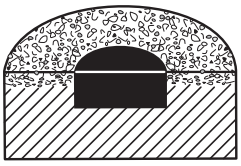
B



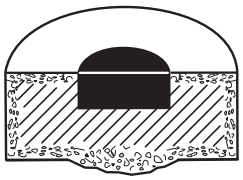
Shrinkage (Edge): Poor adhesion to sample surface with excessive shrinkage at interface. Use lower molding temperature.



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.



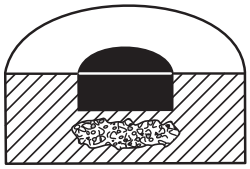
Case Hardening & Blister: Excessive mold temperature. Decrease mold temperature. Momentarily release pressure during flow stage.

General

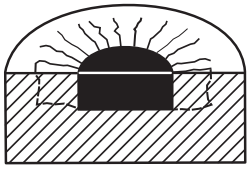
If mold temperature is too high, the following difficulties may also be encountered.

- Mounts sticking to mold surface regardless of finish or application of release agent.
- Dull surfaces on mounts.
- Case hardening of outer mount surfaces.
- Excessive flash.
- 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 Epoxides, Polyesters, and 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 light pink hue. The acrylics are opaque. Other acrylics, when placed and cured in a pressure vessel at 2 bars (28.8 psi) of pressure, produce a semi-transparent mount when transparency is needed for locating a particular area.

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

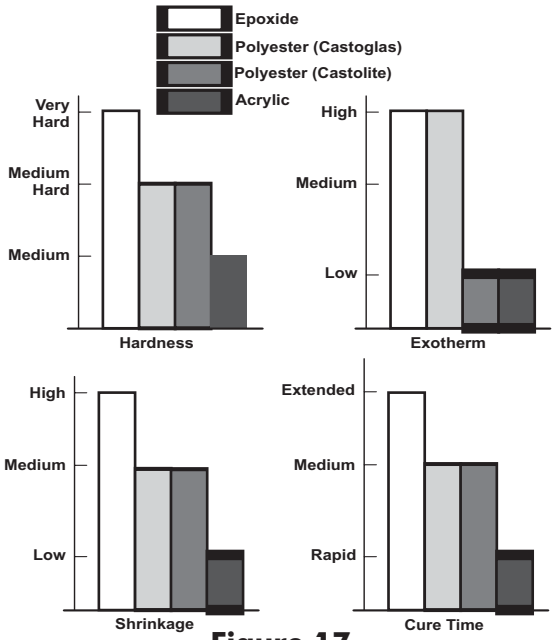
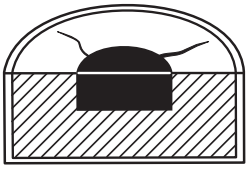
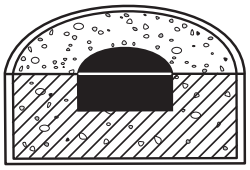


Figure 17

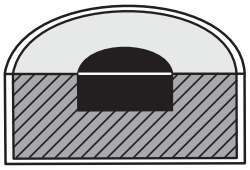
Epoxide Molding Defects



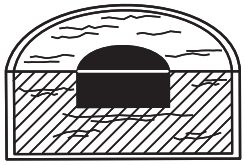
Cracking: Resin-to-hardener ratio incorrect. Exotherm too extreme. Correct resin-to-hardener ratio. Use forced cool air to control rate of exotherm.



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

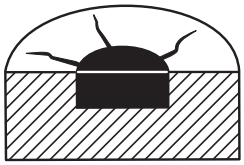


Discoloring: Hardener has oxidized. Resin-to-hardener ratio incorrect. Keep containers tightly sealed. Correct resin-to-hardener ratio.

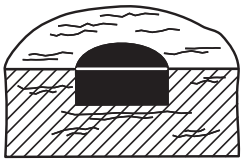


Soft Mounts: Resin-to-hardener ratio incorrect. Incomplete blending of resin and hardener mixture. Correct resin-to-hardener ratio. Completely blend mixture.

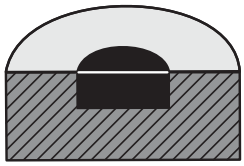
Polyester Molding Defects



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

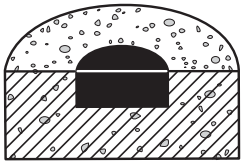


Discoloration: Resin has oxidized. Resin-to-hardener ratio incorrect. Keep container tightly sealed. Correct resin-to-hardener ratio.



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 re-sectioning.

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

B

Suggested equipment for using Bakelite® ring forms of standardized mount diameters is shown in Figure 18. 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 the same. Do not coat inside diameter of the 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 is 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 the epoxy and the sample. Intermittent release and reactivation of vacuum will indicate when all the 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.

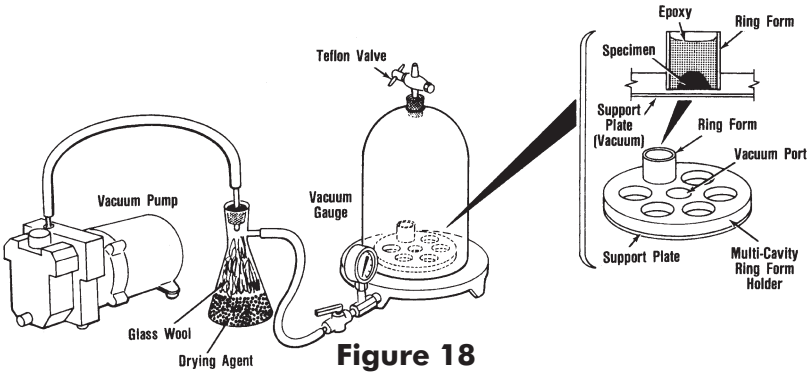


Figure 18

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 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, and 1.50 inches in diameter, and are 0.5 to 0.75 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 that may be programmed with an automatic timer is shown in Figure 19.

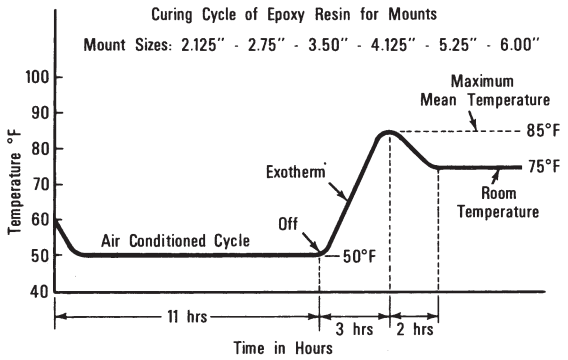


Figure 19

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 time 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.

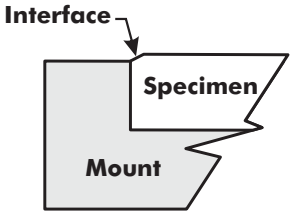
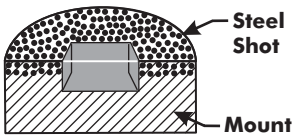
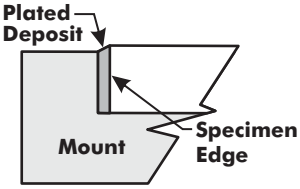


Figure 20

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

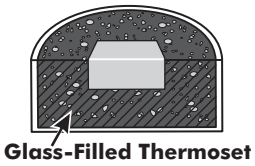


Steel Shot: This technique serves the same purpose as the following suggestion.

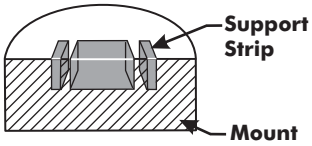


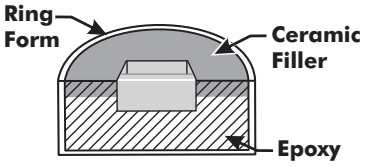
Electro or Electroless Deposition: The section is plated with a coating sufficiently thick to compensate for edge rounding during preparation. The more common materials include Ni and Cu. Nickel will sometimes peel away from a surface due to its stressed condition. Copper is generally used for post-plating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

B

Grams of mounting media required to produce a finished mount 5/8" (16 mm) tall

	Mount Diameter (Inches)			
Hot	**1.0**	**1.25**	**1.5**	**2.0**
Bakelite	11.0	17.1	24.6	43.8
Epoxy	15.9	24.8	35.7	63.5
Diallyl Phthalate	14.9	23.2	33.5	59.5
Copper Diallyl Phthalate	34.3	53.6	77.2	137.2
Lucite	9.2	14.4	20.7	36.7

Cold	**1.0**	**1.25**	**1.5**	**2.0**
Long/Quick Cure Epoxy	9.4	14.6	21.1	37.4
Lecoset 100	9.6	15.0	21.6	38.4
Lecoset 7007	9.4	14.7	21.1	37.5
Lecoset 7000	5.9	9.3	13.4	23.8

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C

Specific Grinding and Polishing Procedures

Polishing Procedures for Ferrous Materials

Fine Grinding

Abrasive & Size	Lap or Wheel Covering	Lubricant
SiC 180 grit	Paper or Disc (waterproof)	Water
SiC 320 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.

Rough Polishing

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

Final Polishing

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

Graphitic Cast Irons

Fine Grinding

- 240, 320, 400, 600 grit SiC or Al_2O_3 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 1200 grit paper dry. After a short period of abrasion, clean abrasive surface with a 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 and lapping oil as lubricant.

Final Polishing

- Sequencing through 0.3 micron Alpha Alumina and 0.05 Gamma Alumina, Lecloth®, 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.

Polishing Procedures for Copper-Based Materials

Fine Grinding

Abrasive & Size	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

C

Remarks

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

Rough Polishing

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

Final Polishing

Abrasive & Size	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 that 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.

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.

Rough Polishing

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

Final Polishing

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

- 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 the wheel covering differs from standard procedures. The entire surface should be pre-moistened 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.

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.

Rough Polishing

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

Final Polishing

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

- 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%) *Caution: Avoid contact with skin!*

Techniques

Swab vigorously with saturated cotton. Reaction is very active at the 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 your hands.*

Suggested Deviations

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

Polishing Procedures for Cemented Carbides

Fine Grinding

Abrasive & Size	Lap or Wheel Covering	Lubricant
Diamond (40 μm; 280 mesh)	Resin-Bonded Diamond Disc	Water
Diamond (15 μm)	Resin-Bonded Diamond Disc	Water

Remarks

- For gross surface removal, employ a 74 μm (220 mesh) resin-bonded diamond disc to decrease wear on 40 μm (280 mesh) disc.

Rough Polishing

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

- Very often the 3 micron diamond paste step may be omitted.

Final Polishing

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

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

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

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

Rough Polishing

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

- There may be some advantage to extend rough polishing into two steps by incorporating a 0.5 micron diamond with Lecloth® step. If lapping oil should attack any microconstituents, alcohol or ethylene-glycol may be substituted.

C

Final Polishing

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

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

Polishing Procedures for Refractory Alloys and Metals (Nb, Mo, W, V, Ta)

Fine Grinding

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

Rough Polishing

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

Remarks

- Extreme caution should be exercised in all preceding stages to avoid, or at least minimize, surface deformation.
- 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	Wheel Covering	Lubricant
Gamma Alumina (0.05 μm)	Lecloth® (flocked cotton sateen)	Distilled or Deionized Water

Suggested Deviations

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

Polishing Procedures for Plated Sections

Fine Grinding

Abrasive & Size	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.

Rough Polishing

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

Final Polishing

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

- During prepar[illegible] 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 in the Mounting Procedures section.
- Etching—The interfaces can be clearly delineated by etching. Specific details are given in Table 4 under Microetching.

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_2O_3-Coated Products (waterproof)—Same abrasive sequence and lubricant may be used. Abrasive action is less severe on some materials.

Rough Polishing

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

Final Polishing

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

General Inform

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 several stages with 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 in the Mounting Procedures section.

Polishing Procedures for Ceramics

Fine Grinding

Abrasive & Size	Wheel Covering	Lubricant
Diamond (40 μm; 280 Mesh)	Resin-Bonded Diamond Disc	Water
Diamond (15 μm)	Resin-Bonded Diamond Disc	Water

Rough Polishing

Abrasive & Size	Wheel Covering	Lubricant
Diamond Paste (9 μm)	Pan W	Lapping Oil
Diamond Paste (6 μm)	Pan W	Lapping Oil

Remarks

- For gross surface removal, employ a 63-74 micron (220 Mesh) resin bonded diamond disc to decrease wear on 40 micron (80 Mesh) disc.

Final Polishing

Abrasive & Size	Wheel Covering	Lubricant
Diamond Paste (3 μm)	Pan W	Lapping Oil
Alpha Alumina (0.3 μm)	Silk	Distilled Deionized Water

- 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.

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

C

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Microetching

Definition

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

Chemical 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.

- Acid and Basic Reagents
  - Immersion
  - Swabbing
- Electrolytic
  - Direct Current
  - 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 to 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 and Alloys

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 Tables 1 through 4 contain etchant information for irons and steels, aluminum, copper, and titanium. The “uses” column in each table provides specific alloy/etchant information

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 procedures for handling and mixing these chemicals.

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

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

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

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

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

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

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

3.2.7 Have available and use quick references as to toxicity and working precautions of various chemicals.

D

4. Miscellaneous Information

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

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

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

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

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

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

4.7 In electrolytic etching, DC current is implied unless indicated otherwise.

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

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

4.10 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.

4.11 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.

Metal Progress Data Book

Table I—Etching Reagents for Irons & Steels

General Reagents for Irons and Steels (Carbon, Low, and Medium-Alloy Steels)

Etching Reagent	Composition	Remarks	Uses
Nital	2 ml HNO_3, 98 ml ethyl 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 alcohol.	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, spheriodized, austempered.
Sodium metabisulfite	Solution A: 8 g $Na_2S_2O_5$, 100 ml distilled water.	similar to picral. Etching time: a few seconds to 1 minutes.	martensite.
	Solution B: 1 g $Na_2S_2O_5$, dilute to 100 ml with distilled water.	Immerse specimen in the solution for 2 minutes 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.
	General reagent for steel. Results	Darkens as-quenched	
Vilella's reagent	5 ml HCl, 1 g picric acid, 100 ml ethyl alcohol.	Best results obtained when martensite is tempered.	For revealing austenitic grain size in quenched, and quenched and tempered steels.

Table I—continued

Etching Reagent	Composition	Remarks	Uses
Klemm's reagent	50 ml saturated (in H_2O) $Na_2S_2O_3$ solution, 1 g $K_2S_2O_2$.	Etching time: 40 to 120 seconds. 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.
General Reagents for Alloy Steels (High Alloy, Stainless, and Tool Steels)			
Ferric chloride and hydrochloric acid	5 g $FeCl_3$, 50 ml HCl, 100 ml distilled water.	Immerse until structure is revealed.	Reveals structure of austenitic nickel and stainless steels.
Kalling's	5 g $CuCl_2$, 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).
Glyceregia	Solution A: 10 ml HNO_3, 20 ml HCl, 30 ml glycerin.	Mix HCl and glycerin thoroughly before adding HNO_3. 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.
	Solution B: 10 ml HNO_3, 20 ml HCl, 20 ml glycerin, 10 ml H_2O_2. .	**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.
Marble's reagent	4 g $CuSO_4$, 20 ml HCl, 20 ml distilled water.	Immerse to reveal structure.	Structure of stainless steels.
Vilella's reagent	5 ml HCl, 1 ml picric acid, 100 ml ethyl alcohol.	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.
Hydrochloric acid in alcohol	50 ml HCl, 50 ml ethyl alcohol.	More gradual etching can be obtained with less concentrated solutions (10 to 20%).	Suitable for etching steels containing chromium and nickel.
Nitric acid	5 to 10 ml HNO_3, 100 ethyl alcohol.	**Use Hood**. HNO_3 and ethyl alcohol are a dangerous mixture above 5% HNO_3.	General structure of high-speed tool steels.
Hydrochloric and nitric acids	10 ml HCl, 3 ml HNO_3, 100 ml methyl alcohol.	Etching time: 2 to 10 minutes.	To reveal the grain size of quenched, or quenched and tempered high-speed steel.
Electrolytes for Polishing and Etching			
Chromic acid	10 g CrO_3, 10 ml H_2O.	Specimen is used as anode; stainless steel or platinum as cathode, 3/4 to 1 inch 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_3, 50 ml H_2O.	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.

D

Table I—continued

Etching Reagent	Composition	Remarks	Uses
Electrolytes for Polishing and Etching			
Sulfuric acid in water	5 ml H_2SO_4, 95 ml H_2O.	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_2O.	5 to 20 seconds at 6V, using a platinum or stainless steel cathode. Gap between electrodes, 3/4 to 1 inch.	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_2O (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_2O (add slowly).	60 seconds at 1 to 3V. Highly caustic solution.	Same as above, but sigma phase and ferrite are reveal simultaneously.
Ammonium persulfate in water	10 g $(NH_4)_2S_2O_8$, 100 ml H_2O.	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.

Table II—Etching Reagents for Aluminum and Aluminum Alloys

Etching Reagent	Composition	Remarks	Uses
Keller's	2 ml HF, 3 ml HCl, 5 ml HNO_3, 190 ml H_2O.	Immerse 10 to 20 seconds. Rinse in stream of water.	Reveals general structure.
Hatch	1 g NaOH, 100 ml H_2O.	Swab 10 seconds. Immerse 15 minutes and rinse for 10 minutes to form film with cross hatching.	Reveals general structure. Cross hatching varies with grain orientation.
Barker's	2 to 4% Hydrofluorboric Acid in 200 ml H_2O.	Electrolytic; 1 Amp for 2 to 3 minutes. View under polarized light.	Reveals grain structure.
Macro/Weld Etch	1 part HCl 1 part HF 1 part H_2O.	Rinse; immerse in 15% NaOH solution.	For welds and macro exams.

Table III—Etching Reagents for Copper and Copper Alloys

Etching Reagent	Composition	Remarks	Uses
For Pure Copper	50 ml NH_4OH, 5 ml H_2O_2.	Immerse; dilute with water as needed.	General structure
For Copper/Zinc Alloys (Brass)	50 ml NH_4OH, 5 ml H_2O_2, 50 ml H_2O.	Immerse.	General structure
For Copper/Tin Alloys (Bronze)	1 g NaCl, 2 ml chromic acid (20%), 2 ml H_2SO_4, 95 ml H_2O.	Immerse.	General structure

Table III—continued

Etching Reagent	Composition	Remarks	Uses
For Copper/ Beryllium Alloys	5 g Ferric Chloride, 10 ml HCl, 95 ml ethyl alcohol.	Immerse.	General structure.

Table IV—Etching Reagents for Titanium and Titanium Alloys

Etching Reagent	Composition	Remarks	Uses
Kroll's	6 ml HNO_3, 2 ml HF, 92 ml H_2O.	Swab for 2 to 30 seconds.	General structure.
Alternative to Kroll's	1-3 ml HF, 2-6 HNO_3 100 ml H2O.	Immerse or swab 3 to 30 seconds.	Adjust mixture to balance etching with brightness.
Alpha Case Etch	2 ml HF 100 ml H_2O.	Immerse or swab 2 to 60 seconds.	Reveals alpha case in alloys.

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Macroetching

Definition

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

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 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 is then 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 inches away from the torch-cut edge.

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

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.

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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 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 portions 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 finished 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 are often 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, with either 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 I).

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 General 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 μm Al_2O_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 μm green Cr_2O_3 in tap water on synthetic suede works best.

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

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 III).

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 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 IV).

3.5 Iron and Steel

3.5.1 Macroetching has been 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 V.

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 requires 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. Th specimen should be heavily etched in 1:1 HCl or 20% H_2SO_4. 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 V).

3.6 Stainless Steels and High Temperature Alloys

3.6.1 These alloys are generally more susceptible to cold working of the surface than 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_3 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% HNO_3. Scrubbing will provide a higher contrast for detection of segregation and inclusions. The desmutting, either by the addition of HNO_3 to the etching bath, or by the secondary rinse in HNO_3, 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, will often give trouble in etching. Aqua regia, HCl-H_2O_2, 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 VI).

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 recrystallize readily at temperatures which can be easily achieved in careless preparation (especially pure lead).

3.7.2 For best result 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-inch files 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, brushing 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 VII).

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-inch radius V-shaped tool fed at a rate of 0.002 to 0.003-inch 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 VIII).

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_3 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_2O_2 solutions.

3.9.2 Macroetchants for Nickel and Nickel Alloys (Table IX).

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

3.10.1 These metals are, in general, soft and ductile. Because of their expense, specimens for macroetching will usually 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 X).

3.11 Refractory Metals—Cr, Mo, W, V, Cb, 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 are 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.

3.11.2 Macroetchants for the Refractory Metals (Table XI).

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 μm 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 XII).

3.12.3 Preparation of Ammonium Polysulfide—Pass H_2S gas into 200 ml of NH_4OH (sp. gr. 0.9) until saturated. The solution should be kept in an ice bath during this operation. Add an additional 200 ml of NH_4OH (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 HCl-HF solution requires desmutting. The specimen should be rinsed between etching and desmutting (Table XIII).

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 XIV).

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Table I—Macroetchants for Aluminum and Aluminum Alloys

Alloy	Composition	Procedure	Comments
All	NaOH . . . . . 10 g H_2O . . . . . 100 ml	Immerse sample 5 to 15 min. in solution heated to 60° to 70°C (140° to 160°F). Rinse in water, and remove smut in strong HNO_3 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_3 (conc) . . . . . 25 ml HF (48%) . . . . . 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% water to slow down etching. Does not require fine grinding.
High purity Al 1XXX 3XXX 4XXX 5XXX 6XXX	HCl (conc) . . . . . 45 ml HNO_3 (conc) . . . . . 15 ml HF (48%) . . . . . 15 ml H_2O . . . . . 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_3 (conc) . . . . . 5 ml HF (48%) . . . . . 5 ml H_2O . . . . . 75 ml	Immerse specimen at room temperature until desired contrast is developed. Rinse in warm water and dry.	1+2 Tucker's; same as above but slower acting.

Table I—continued

Alloy	Composition	Procedure	Comments
2XXX High Cu alloys	HCl (conc) . . . . . 15 ml HF (48%) . . . . . 10 ml H_2O . . . . . 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_3. Rinse in warm water and dry.	Flick's reagent; best results are obtained with a ground surface; 180 grit will suffice.

Table II—Macroetchants for Beryllium and Beryllium Alloys

Metal	Composition	Procedure	Comments
Be	HCl. . . . . 10 ml NH_4Cl . . . . . 4 g H_2O . . . . . 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	HCl. . . . . 10 ml NH_4Cl . . . . . 2 g Picric acid . . . . . 2 g H_2O . . . . . 90 ml	Either swab or immerse at room temperature for a few minutes, rinse in water, and dry.	An alternative when No. 1 does not work. Fine-grained metal may not give good results in either case.

Table III—Macroetchants for Cobalt and Cobalt Alloys

Alloys	Composition	Procedure	Comments
49Co-49Fe-V Some Co-Cr alloys	HCl. . . . . 50 ml H_2O . . . . . 50 ml	Immerse specimen in hot solution 60° to 82°C (140° to 180°F) for 30 to 60 minutes.	Rinse in hot water and dry; general structure, porosity.
25Cr-10Ni-8W 21Cr-20Ni-3W-3Mo-1Cb	HCl . . . . . 50 ml HNO_3. . . . . 10 ml $FeCl_3$ . . . . . 10 g H_2O . . . . . 100 ml	Swab until desired contrast is obtained, then rinse in warm water, and dry.	Grain size, general structure.
18Cr-10Ni-14W	$CuCl_2 \cdot 2NH_4CL \cdot 2H_2O$. . . 2 g $FeCl_3$ . . . . . 5 g HNO_3. . . . . 5 ml HCl . . . . . 50 ml H_2O . . . . . 80 ml	Swab until desired contrast is obtained, then rinse in warm water, and dry.	Grain size, general structure.

Table IV—Macroetchants for Copper and Copper Alloys

Alloys	Composition	Procedure	Comments
Cu and all brasses	HNO_3 . . . . . 10 ml H_2O . . . . . 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	HNO_3 . . . . . 50 ml H_2O . . . . . 50 ml	Immerse specimen in solution at room temperature for a few minutes. Rinse in water and dry.	Brings out grain contrast, pits result unless agitated. Aluminum bronzes may form smut which can be removed by brief immersion in concentrated HNO_3.
Cu and all brasses	HCl . . . . . 30 ml $FeCl_3$ . . . . . 10 g H_2O or ethanol . . . . . 120 ml	Immerse specimen in solution at room temperature for a few minutes. Rinse in water and dry.	Good grain contrast.
Cu, high Cu alloys, phosphorus, tin bronzes	$K_2Cr_2O_7$ sat . . . . . 2 g solution of NaCl H_2SO_3 H_2O	Immerse specimen in solution at room temperature for 15 to 30 minutes then swab with fresh solution. Rinse in warm water and dry.	Emphasizes grain boundaries and oxide inclusions.
All	HNO_3. . . . . 50 ml $AgNO_3$. . . . . 5 g H_2O . . . . . 50 ml	Immerse specimen in solution temperature. Rinse in warm water and dry.	Brilliant deep etch.
Brass	20% acetic acid . . . . . 20 ml 5% chromic acid . . . . . 10 ml 10% $FeCl_3$ in H_2O. . . . . 5 ml	Immerse specimen in solution temperature. Rinse in warm water and dry.	Strain lines.

Table IV—continued

Alloys	Composition	Procedure	Comments
Silicon brass or bronze	CrO_3 . . . . . 40 g NH_4Cl . . . . . 7.5 g HNO_3 (conc) . . . . . 50 ml H_2SO_4 (conc) . . . . . 8 ml H_2O . . . . . 100 ml	Immerse specimen in solution at room temperature, rinse in warm water, and dry.	

Table V—Macroetchants for Irons and Steels

Alloys	Composition	Procedure	Comments
Plain and alloy steels, high speed and tool steels, cutlery (12 to 14% Cr), and stainless steels	HCl (conc) . . . . . 50 ml H_2O . . . . . 50 ml	Immerse specimen in solution heated to 60° to 82°C (160° to 180°F) for 15 to 30 minutes. Desmut by vigorous scrubbing with vegetable fiber brush under running water. Stainless steels may be desmutted by dipping in a warm 20% HNO_3 to give a bright finish.	General purpose.
High alloy steels	HCl (conc) . . . . . 50 ml HNO_3 (conc) . . . . . 25 ml H_2O . . . . . 25 ml	Immerse specimen for 10 to 15 minutes in solution at room temperature. Rinse in warm water and dry.	Ratio HCl:HNO_3 runs 2:1 to 3:1.
Plain and alloy steels, cutlery steels	HCl (conc) . . . . . 38 ml H_2SO_4 (conc) . . . . . 12 ml H_2O . . . . . 50 ml	Immerse specimen for 15 to 45 minutes in solution heated to 60° to 82°C (160 to 180°F) Rinse in warm water and dry.	Works well on 12% Cr steel.
High alloy steels	HNO_3 (conc) . . . . . 10 ml HF (48%) . . . . . 4 ml H_2O . . . . . 87 ml to HNO_3 (conc) . . . . . 40 ml HF (48%) . . . . . 10 ml H_2O . . . . . 50 ml	Immerse specimen in solution heated to 60° to 82°C (160° to 180°F) until desired etch is obtained, and rinse in warm water and dry.	Ratio HNO_3:HF varies.
Stainless steels, high-alloy steels	HCl (conc) . . . . . 50 ml H_2O . . . . . 50 ml H_2O_2 (30%) . . . . . 20 ml Mix HCl and water then heat to	60° to 77°C (160° to 170°F), immerse specimen, and add H_2O_2 in several parts. Do not mix. Make each subsequent addition after foaming from previous addition has stopped. Produces bright finish.	
Austenitic stainless steels	HCl (conc) . . . . . 50 ml saturated solution $CuSO_4$ in H_2O_2 . . . . . 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	$(NH_4)_2S_2O_8$ . . . . . 10 g (ammonium persulfate) H_2O . . . . . 100 ml	Swab solution at room temperature over specimen. Rinse and dry.	Grain size, weldments.
Plain and alloy steels	$CuCl_2$ . . . . . 2.5 g $MgCl_2$ . . . . . 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; Bessemer and high N_2 steel	$CuCl_2$ . . . . . 90 g HCl (conc) . . . . . 120 ml H_2O . . . . . 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 100 to 250°C (302° to 482°F) for 5 to 30 minutes depending on condition of steel. To show strain lines due to cold work.

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Table V—continued

Alloys	Composition	Procedure	Comments
Plain and alloy steels	$CuCl_2$ . . . . . 45 g HCl (conc) . . . . . 180 ml H_2O . . . . . 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.	Modified Fry's reagent; same as for reagent above, but modified by Wazau; may give more contrast; specimen can be washed in water without depositing copper.
Stainless and high Cr steels	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 VI—Macroetchants for Stainless Steels and High-Temperature Alloys

Alloys	Composition	Procedure	Comments
Stainless steels and iron-base high-temperature alloys	HCl (conc) . . . . . 50 ml H_2O . . . . . 50 ml	Immerse specimen in solution heated to 60° to 82°C (160° to 180°F) for 30 minutes. Desmut by vigorous scrubbing with vegetable brush under running water. Stainless steels may be desmutted by dipping in warm 20% HNO_3 to give bright finish.	General purpose.
Iron, cobalt, and nickel-base high-temperature alloys	HCl (conc) . . . . . 50 ml HNO_3 (conc) . . . . . 25 ml H_2O . . . . . 25 ml	Immerse specimen in solution at room temperature for 10 to 30 minutes. Rinse and dry.	Ratio HCl : HNO_3 runs 2:1 to 3:1.
Stainless steels and high-temperature alloys	HNO_3. . . . . 10 ml HF (48%) . . . . . 3 ml H_2O . . . . . 87 ml to HNO_3 (conc) . . . . . 40 ml HF (48%) . . . . . 10 ml H_2O . . . . . 50 ml	Immerse specimen in solution heated to 60° to 82°C (160° to 180°F) until desired contrast is obtained. Rinse and dry.	Ratio HNO_3 : HF varies.
Austenitic stainless steels and nickel-base alloys	A. $(NH_4)_2SO_4$. . . . . 15 g H_2O . . . . . 75 ml B. $FeCl_2$ . . . . . 250 g HCl (conc) . . . . . 100 ml C. HNO_3 (conc) . . . . . 30 ml	Combine A and B, then add C. 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	HCl (conc) . . . . . 50 ml H_2O . . . . . 50 ml H_2O_2 (30%) . . . . . 20 ml	Mix HCl and water, then heat, immerse specimen, and add H_2O_2 in several parts. Do not mix. Make each subsequent addition after foaming from previous addition has stopped.	
Austenitic stainless steels and high-temperature alloys	HCl (conc) . . . . . 50 ml Saturated solution . . . . . 25 ml of $CuSO_4$ in H_2O	Immerse specimen in solution which may be heated up to 77°C (170°F) until desired contrast is obtained. Rinse and dry.	Marble's reagent. Light etch, good for structures. Amount of $CuSO_4$ solution may be increased to 1:1 ratio for difficult alloys.

Table VII—Macroetchants for Lead and Lead Alloys

Alloys	Composition	Procedure	Comments
Lead and lead alloys	A. H_2O . . . . . 250 ml NH_4OH (sp gro. 90) . . . 140 ml HNO_3 (conc) . . . . . 60 ml Molybdic acid (85%) . . 100 ml B. H_2O . . . . . 960 ml HNO_3 (conc) . . . . . 400 ml C. Glacial acetic acid . . . . 100 ml 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. Swab surface of the specimen with mixed solution until desired contrast is obtained. Rinse and dry.	

Table VII—continued

Alloys	Composition	Procedure	Comments
Antimonial lead	A. Glacial acetic acid . . . . 30 ml HNO_3 (conc) . . . . . 40 ml H_2O . . . . . 160 ml B. Glacial acetic acid . . . . 1 ml H_2O . . . . . 400 ml	Prepare surface on silk velvet wheel with Al_2O_3 abrasive at 150 rpm. Etch with solution A at 42°C (108°F) then repolish until bright. Re-etch with B at room temperature for 1 to 2 hours.	
Antimonial lead	A. HNO_3 (conc) . . . . . 80 ml H_2O . . . . . 220 ml B. $(NH_4)_2MoO_4$ . . . . . 45 g H_2O	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.
Antimonial lead	$(NH_4)_2MoO_4$ . . . . . 10 g Citric acid . . . . . 25 g H_2O . . . . . 100 ml	Immerse specimen in solution at room temperature until desired contrast is obtained, then rinse and dry.	Bright etch, grain structure, defects.
Antimonial lead	Acetic acid . . . . . 75 ml H_2O_2 . . . . . 25 ml	Mix with strongest H_2O_2 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 VIII—Macroetchants for Magnesium and Magnesium Alloys

Alloys	Composition	Procedure	Comments
ZK60A	Glacial acetic acid . . . . . 5 ml H_2O . . . . . 95 ml	Immerse specimen in solution at room temperature for 1/2 to 8 minutes until desired contrast is obtained. Desmut with 50% HF (48%) in water. Rinse in rapidly flowing water and dry.	Flow lines in forgings.
AZ61A AZ80A	Glacial acetic acid . . . . . 10 ml H_2O . . . . . 90 ml	As above, but for 1/2 to 5 minutes.	Grain size, surface casting defects.
AZ31B AZ61A AZ80A	Glacial acetic acid . . . . . 20 ml $NaNO_3$. . . . . 5 g H_2O . . . . . 80 ml	As above, but for 1/4 to 5 minutes.	Flow pattern in forgings. Surface casting defects. Glycolic acid may be substituted for acetic acid.
AZ31B	HNO_3 (conc) . . . . . 10 ml H_2O . . . . . 90 ml	As above, but for 1/2 to 5 minutes.	Flow pattern in forgings. Internal defects in cast slabs and ingots.
AZ31B	$Na_2Cr_2O_7$ . . . . . 180 g HNO_3 (conc) . . . . . 180 ml H_2O to make . . . . . 1000 ml	As above, but rinse in hot water.	General etch for defects in sand and die castings.
AZ31B	CrO_3 . . . . . 280 g HNO_3 (conc) . . . . . 25 ml HF (48%) . . . . . 10 ml H_2O to make . . . . . 1000 ml	As above, but rinse in hot water.	Germination on sand cast surfaces. Surface defects of die castings.
AZ61A AZ80A	6% picric acid in alcohol . 100 ml H_2O . . . . . 10 ml Glacial acetic acid . . . . . 5 ml	As above for 1/2 to 3 minutes, or may be swabbed.	Grain size and flow patterns of both cast and wrought forms. Requires fine finish (600 grit).
AZ21 AZ31	6% picric acid in alcohol . . 70 ml H_2O . . . . . 10 ml Glacial acetic acid . . . . . 10 ml	As above for 1/2 to 3 minutes, or may be swabbed.	Grain size. Specimen should be finished on 600-grit paper.
ZK60A	6% picric acid in alcohol . . 50 ml H_2O . . . . . 20 ml Glacial acetic acid . . . . . 20 ml	As above for 1/2 to 3 minutes, or may be swabbed.	Flow pattern and grain size of homogeneous alloy. Increase water to increase strain contrast. Specimen should be finished on 600-grit paper.

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Table VIII—continued

Alloys	Composition	Procedure	Comments
ZK60A	4% picric acid in alcohol . . 100 ml H_2PO_4. . . . . 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 IX—Macroetchants for Nickel and Nickel Alloys

Alloys	Composition	Procedure	Comments
Ni	$CuSO_4$ . . . . . 10 g HCl . . . . . 50 ml H_2O . . . . . 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	HNO_3. . . . . 20 ml H_2O . . . . . 10 ml $CuSO_4$ . . . . . 10 g	Immerse specimen in solution at room temperature for 20 to 30 minutes; rinse and dry.	Grain structure.
High-Ni alloys	HNO_3 (conc) . . . . . 50 ml H_2O . . . . . 50 ml	Immerse specimen in solution at room temperature for 20 to 30 minutes; rinse and dry.	Porosity and flowlines.
Alloys containing Cr, Fe, and other elements	HNO_3. . . . . 50 ml Acetic acid . . . . . 50 ml	Immerse specimen in hot solution. Rinse in hot water and dry.	Grain structure.
Alloys containing Cr, Fe, and other elements	Acetic acid . . . . . 50 ml HNO_3. . . . . 50 ml Swab.	Lepito's reagent II; etchant for	nickel welds.
Alloys containing Cr, Fe, and other elements	Sat soln of $CuSO_4$ in H_2O . 50 ml HCl. . . . . 50 ml	Swab etchant.	Modified Marble's reagent.
Alloys containing Cr, Fe, and other elements	HCl . . . . . 100 ml H_2O . . . . . 100 ml H_2O_2 (30%) . . . . . 40 ml	See Table V.	See Table V.

Table X—Macroetchants for Noble Metals

Metal	Composition	Condition of Use	Comments
Ag	HNO_3. . . . . 10 ml Methyl alcohol . . . . . 90 ml	Room temperature; few minutes.	Grain contrast.
Au Pt alloys Pd alloys	HCl . . . . . 66 ml HNO_3. . . . . 34 ml	Hot; few minutes.	Grain contrast.
Ru and alloys Os and alloys Rh and alloys	Lactic acid. . . . . 50 ml HNO_3. . . . . 20 ml HF . . . . . 30 ml	Room temperature; few minutes.	Grain contrast.
Ru and alloys Os and alloys Rh and alloys	HCl . . . . . 30 ml HNO_3. . . . . 15 ml HF . . . . . 30 ml	Room temperature; few minutes.	Grain contrast.
Pt and alloys	Sat soln of NaCl in H_2O . . 80 ml HCl . . . . . 20 ml	Electrolytic, 6V; few minutes.	Grain contrast.

Table XI—Macroetchants for Refractory Metals

Metal	Composition	Temperature	Time	Comments
Mo, W, V, Cb, and Ta	HCl (conc) . . . . . 30 ml HNO_3 (conc) . . . . . 15 ml HF (48%) . . . . . 30 ml	Room temperature.	5 to 20 minutes.	
Mo, W, and V	HF (48%) . . . . . 15 ml HNO_3 (conc) . . . . . 35 ml H_2O . . . . . 75 ml	Room temperature.	10 to 20 minutes.	Too fast for Mo; 1 to 5 seconds.

Table XI—continued

Metal	Composition	Temperature	Time	Comments
W, V, Cb, and Ta	HF (48%) . . . . . 10 ml HNO_3 (conc) . . . . . 30 ml Lactic acid (85%) . . 50 ml	Room temperature.	1 to 40 minutes.	
Cr	H_2SO_4 . . . . . 10 ml H_2O . . . . . 90 ml	Boiling.	2 to 5 minutes.	

Table XII—Macroetchants for Tin and Tin Alloys

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

Table XIII—Macroetchants for Titanium, Zirconium, Hafnium, and their Alloys

Alloys	Composition	Procedure	Comments
Ti Alloys	HCl (conc) . . . . . 20 ml HF (48%) . . . . . 40 ml H_2O . . . . . 50 ml	Immerse specimen in solution heated to 49° to 66°C (120 to 150°F) for 20 to 30 minutes. Rinse. If smut forms immerse in 30% H_2SO_4 for 3 minutes. Rinse and dry.	Desmut Ti, -13V-11 Cr-3Al alloys. Others do not normally need desmutting.
7Al-4Mo alloys	HNO_3 (conc) . . . . . 42 ml HF (48%) . . . . . 8 ml H_2O . . . . . 50 ml	As above.	
Zr, Hf, and low alloys	H_2O_2 (30%) . . . . . 45 ml HNO_3 (conc) . . . . . 45 ml HF (48%) . . . . . 10 ml	Swab specimen with solution at room temperature. Rinse 10 seconds after yellow fumes form, then dry.	Should be used under a hood; chemical polishing solution.
Zr, Hf, high alloys	H_2O . . . . . 45 ml HNO_3. . . . . 45 ml HF . . . . . 10 ml	As above.	As above.
1.5Sn-0.15Fe-0.10Cr, and Hf	H_2O . . . . . 70 ml HNO_3 (conc) . . . . . 30 ml HF (48%) . . . . . 5 ml	As above.	As above.
Iodide Ti	H_2O_2 (30%) . . . . . 60 ml H_2O . . . . . 30 ml HF (48%) . . . . . 10 ml	Swab with solution at room temperature until desired contrast is obtained. Rinse in cold water and dry.	As above.

Table XIV—Macroetchants for Zinc and Zinc Alloys

Alloys	Composition	Procedure	Comments
Cu-free zinc alloys	HCl (conc) . . . . . 50 ml H_2O . . . . . 50 ml	Immerse in solution for about 15 seconds. Remove smut by wiping under running water. Repeat until desired contrast is obtained, then dry.	Grain structure.
Zn alloys containing Cu	CrO_3 . . . . . 20 g Na_2SO_4 . . . . . 1.5 g or $Na_2SO_4 \cdot 10H_2O$ . . . . . 3.4 g H_2O . . . . . 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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