



Designation: **E 3 – 9501**

An American National Standard

Standard Practice Guide for Preparation of Metallographic Specimens¹

This standard is issued under the fixed designation E 3; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 The primary objective of metallographic examinations is to reveal the constituents and structure of metals and their alloys by means of ~~the~~ a light optical or scanning electron microscope. In special cases, the objective of the examination may require the development of less detail than in other cases but, under nearly all conditions, the proper selection and preparation of the specimen is of major importance. Because of the diversity in available equipment and the wide variety of problems encountered, the following text presents for the guidance of the metallographer only those practices which experience has shown are generally satisfactory; it cannot and does not describe the variations in technique required to solve individual specimen preparation problems.

NOTE 1—For a more extensive description of various metallographic techniques, refer to Samuels, L. E., *Metallographic Polishing by Mechanical Methods*, American Society for Metals (ASM) Metals Park, OH, 3rd Ed., 1982; Petzow, G., *Metallographic Etching*, ASM, 1978; and VanderVoort, G., *Metallography: Principles and Practice*, McGraw Hill, NY, ~~1984~~, 2nd Ed., 1999.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

A 90/A 90M Standard Test Method for Weight (Mass) of Coating on Iron and Steel with Zinc or Zinc-Alloy Coatings

¹ This practice guide is under the jurisdiction of ASTM Committee ~~E-4~~ E04 on Metallography and is the direct responsibility of Subcommittee E04.01 on Sampling, Specimen Preparation, and Photography.

Current edition approved ~~Jan. 15, 1995~~, April 10, 2001. Published ~~March 1995~~, July 2001. Originally published as E 3 – 21 T. Last previous edition ~~E 3 – 80 (1986)~~, E 3 – 95.

E 7 Terminology Relating to Metallography²

E 45 Practice for Determining the Inclusion Content of Steel²

E 340 Test Method for Macroetching Metals and Alloys²

E 407 Test Methods for Microetching Metals and Alloys²

E 768 Practice for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel²

E 1077 Test Method for Estimating the Depth of Decarburization of Steel Specimens²

E 1122 Practice for Obtaining JK Inclusion Ratings Using Automatic Image Analysis²

E 1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis²

E 1268 Practice for Assessing the Degree of Banding or Orientation of Microstructures²

E 1558 Guide to Electrolytic Polishing of Metallographic Specimens²

E 1920 Guide for Metallographic Preparation of Thermal Sprayed Coatings²

3. Significance and Use

3.1 Microstructures have Terminology

3.1 Definitions:

3.1.1 For definitions used in this practice, refer to Terminology E 7.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 castable mount—a metallographic mount generally made from a ~~strong influence on~~ two component castable plastic. One component is the ~~properties resin and successful application of metals the other hardener.~~ Both components can be liquid or one liquid and alloys. ~~Determination a powder.~~ Castable mounts generally do not require heat and ~~control of microstructure pressure to cure.~~

3.2.2 compression mount—a metallographic mount made using plastic that requires both heat and pressure for curing.

3.2.3 planar grinding—is the use of metallographic examination.

3.2 Many specifications contain first grinding step in a requirement regarding microstructure; hence, a major use for metallographic examination is inspection preparation procedure used to ensure that bring all specimens into the requirement is met. Other major uses for metallographic examination are in failure analysis, and in research and development.

3.3 Proper choice same plane of specimen location and orientation will minimize the number of specimens required and simplify their interpretation. ~~polish. It is easy unique to take too few specimens for study, but it is seldom semi or fully automatic preparation equipment that utilize specimen holders.~~

3.2.4 rigid grinding disc—a non-fabric support surface, such as a composite of metal/ceramic or metal/polymer charged with an abrasive (usually 6 to 15µm diamond particles), and used as the fine grinding operation in a metallographic preparation procedure.

4. Significance and Use

4.1 Microstructures have a strong influence on the properties and successful application of metals and alloys. Determination and control of microstructure requires the use of metallographic examination.

4.2 Many specifications contain a requirement regarding microstructure; hence, a major use for metallographic examination is inspection to ensure that the requirement is met. Other major uses for metallographic examination are in failure analysis, and in research and development.

4.3 Proper choice of specimen location and orientation will minimize the number of specimens required and simplify their interpretation. It is easy to take too few specimens for study, but it is seldom that too many are studied.

5. Selection of Metallographic Specimens

45.1 The selection of test specimens for metallographic examination is extremely important because, if their interpretation is to be of value, the specimens must be representative of the material that is being studied. The intent or purpose of the metallographic examination will usually dictate the location of the specimens to be studied. With respect to purpose of study, metallographic examination may be divided into three classifications:

45.1.1 General Studies or Routine Work—~~Specimens should be chosen from locations that are most likely to reveal the maximum variations within the material under study should be chosen.~~ study. For example, specimens ~~should~~ could be taken from a casting in the zones wherein maximum segregation might be expected to occur as well as specimens from sections where segregation ~~should~~ could be at a minimum. In the examination of strip or wire, test specimens ~~should~~ could be taken from each end of the coils.

45.1.2 Study of Failures—Test specimens should be taken as closely as possible to the fracture or to the initiation of the failure. Before taking the metallographic specimens, study of the fracture surface should be complete, or, at the very least, the fracture surface should be documented. ~~§~~ In many cases, specimens should be taken in many cases from a sound area for a comparison of structures and properties.

² Annual Book of ASTM Standards, Vol 03.01.

45.1.3 *Research Studies*—The nature of the study will dictate specimen location, orientation, etc. Sampling will usually be more extensive than in routine examinations.

45.2 Having established the location of the metallographic samples to be studied, the type of section to be examined must be decided.

5.2.1 For a casting, a section cut perpendicular to the surface will show the variations in structure from the outside to the interior of the casting.

5.2.2 In hot-worked or cold-worked metals, both transverse and longitudinal sections should be studied. Special investigations may at times require specimens with surfaces prepared parallel to the original surface of the product.

5.2.3 In the case of wire and small rounds, a longitudinal section through the center of the specimen proves advantageous when studied in conjunction with the transverse section.

4.3 Cross

5.3 Transverse sections or transverse cross sections taken perpendicular to the main axis of the material are more suitable often used for revealing the following information:

45.3.1 Variations in structure from center to surface,

45.3.2 Distribution of nonmetallic impurities across the section,

45.3.3 Decarburization at the surface of a ferrous material (see Test Method E 1077),

45.3.4 Depth of surface imperfections,

45.3.5 Depth of corrosion,

45.3.6 Thickness of protective coatings, and

45.3.7 Structure of protective coating.

45.4 Longitudinal sections taken parallel to the main axis of the material are more suitable often used for revealing the following information:

45.4.1 Inclusion content of steel (see Practices E 45, E 768, E 1122, and E 1245),

45.4.2 Degree of plastic deformation, as shown by grain distortion,

45.4.3 Presence or absence of banding in the structure (see Practice E 1268), and

4.4.4 ~~The quality~~

5.4.4 The microstructure attained with any heat treatment.

45.5 The locations of surfaces examined should always be given in reporting results and in any illustrative micrographs. A suitable method of indicating surface locations is shown in Fig. 1.

5. Size of Metallographic Specimens

5.1 The specimens to be polished for metallographic examination are generally not more than about 12 to 25 mm (0.5 to 1.0 in.) square, or approximately 12 to 25 mm in diameter if the material is round. The height of the specimen should be no greater than necessary for convenient handling during polishing.

5.2 It is not always possible to secure specimens having the dimensions given in 5.1, when the material to be examined is smaller than the ideal dimensions. For example, in the polishing of wire, strip, and other small articles, it is necessary to mount the specimens because of their size and shape.

5.2.1 Larger samples may be mounted or not, as the available equipment dictates. However, the larger the specimen, the more difficult it is to prepare, especially by manual methods.

5.2.2 Specimens that are too small to be handled readily during polishing should be mounted to ensure a surface satisfactory for microscopical study. There are, based on technique used, three fundamental methods of mounting specimens (see Sections 7-9).

6. Cutting Size of Metallographic Specimens

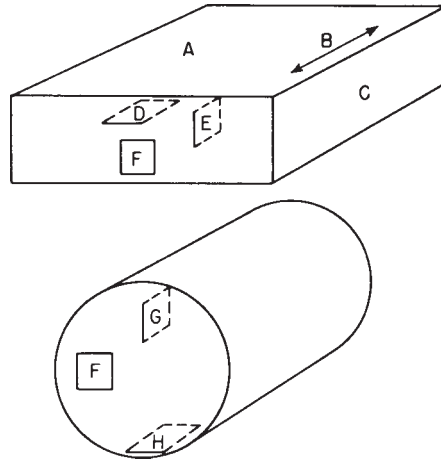
6.1 In cutting the metallographic specimen from the main body of the material, care must be exercised

6.1 For convenience, specimens to minimize altering the structure of the metal. Three common types of sectioning be polished for metallographic examination are as follows:

6.1.1 Sawing, whether by hand generally not more than about 12 to 25 mm (0.5 to 1.0 in.) square, or machine with lubrication, is easy and fast, and relatively cool. It can be used on all materials with hardnesses below approximately 35 HRC. It does produce a rough surface containing extensive plastic flow that must be removed 12 to 25 mm in subsequent preparation.

6.1.2 An abrasive cut-off wheel will produce a smooth surface often ready for fine grinding. This method of sectioning diameter if the material is normally faster than sawing. cylindrical. The choice height of cut-off wheel, lubricant, cooling conditions, and the grade and hardness of metal being cut will influence the quality of the cut. A poor choice of cutting conditions can easily overheat the specimen, producing an alteration of the microstructure. As a general rule, soft materials are cut with a hard bond wheel and hard materials with a soft bond wheel. Aluminum oxide abrasive wheels are preferred specimen should be no greater than necessary for ferrous metals and silicon carbide wheels convenient handling during polishing.

6.1.1 Larger specimens are preferred for nonferrous alloys. Abrasive cut-off wheels are essential for sectioning metals with hardnesses above about 35 HRC. Extremely hard metallic materials and ceramics may be generally more effectively cut using diamond-impregnated cutting wheels. Manufacturer's instructions should be followed as difficult to the choice of wheel and speeds.



Symbol in Diagram	Suggested Designation
A	Rolled surface
B	Direction of rolling
C	Rolled edge
D	Longitudinal (or lengthwise) section parallel to rolled surface
E	Planar section
F	Longitudinal section perpendicular to rolled surface
G	Transverse section
H	Radial longitudinal section
	Tangential longitudinal section

FIG. 1 Method of Designating Location of Area Shown in Photomicrograph.

6.1.3 Flame cutting completely alters the structure of the metal at the flame cut edge. If flame cutting is necessary prepare.

6.1.2 Specimens that are, fragile, oddly shaped or too small to remove the specimen, it be handled readily during polishing should be cut sufficiently large so that it can be recut mounted to the proper size by some other method that will not substantially alter the structure. Exercise care to ensure that the region of interest is not altered by the heat of the cutting flame.

6.2 Other a surface satisfactory for microscopical study. There are, based on technique used, three fundamental methods of sectioning are permitted provided they do not alter the microstructure at the plane of polishing. All cutting operations produce some depth of damage, which will have to be removed in subsequent preparation steps. mounting specimens (see Section 9).

7. Cutting of Metallographic Specimens

7.1 In cutting the metallographic specimen from the main body of the material, care must be exercised to minimize altering the structure of the metal. Three common types of sectioning are as follows:

7.1.1 Sawing, whether by hand or machine with lubrication, is easy, fast, and relatively cool. It can be used on all materials with hardnesses below approximately 350 HV. It does produce a rough surface containing extensive plastic flow that must be removed in subsequent preparation.

7.1.2 An abrasive cut-off blade will produce a smooth surface often ready for fine grinding. This method of sectioning is normally faster than sawing. The choice of cut-off blade, lubricant, cooling conditions, and the grade and hardness of metal being cut will influence the quality of the cut. A poor choice of cutting conditions can easily damage the specimen, producing an alteration of the microstructure. Generally, soft materials are cut with a hard bond blade and hard materials with a soft bond blade. Aluminum oxide abrasive blades are preferred for ferrous metals and silicon carbide blades are preferred for nonferrous alloys. Abrasive cut-off blades are essential for sectioning metals with hardness above about 350 HV. Extremely hard metallic materials and ceramics may be more effectively cut using diamond-impregnated cutting blades. Manufacturer's instructions should be followed as to the choice of blade. Table 1 lists the suggested cutoff blades for materials with various Vickers (HV) hardness values.

7.1.3 A shear is a type of cutting tool with which a material in the form of wire, sheet, plate or rod is cut between two opposing blades.

7.2 Other methods of sectioning are permitted provided they do not alter the microstructure at the plane of polishing. All cutting operations produce some depth of damage, which will have to be removed in subsequent preparation steps.

8. Cleanliness

78.1 Cleanliness (see Appendix X1-) during specimen preparation is essential. All greases, oils, coolants and residue from

TABLE 1 Cutoff Blade Selection

Hardness HV	Materials	Abrasive	Bond	Bond Hardness
up to 300	non-ferrous (Al, Cu)	SiC	P or R	hard
up to 400	non-ferrous (Ti)	SiC	P or R	med. hard
up to 400	soft ferrous	Al ₂ O ₃	P or R	hard
up to 500	medium soft ferrous	Al ₂ O ₃	P or R	med. hard
up to 600	medium hard ferrous	Al ₂ O ₃	P or R	medium
up to 700	hard ferrous	Al ₂ O ₃	P or R&R	med. soft
up to 800	very hard ferrous	Al ₂ O ₃	P or R&R	soft
> 800	extremely hard ferrous	CBN	P or M	hard
	more brittle ceramics	diamond	P or M	very hard
	tougher ceramics	diamond	M	ext. hard

P—phenolic
R—rubber
R&R—resin and rubber
M—metal

cutoff blades on the specimen should be removed by some suitable organic solvent. Failure to clean thoroughly can prevent cold mounting—~~castable~~ resins from adhering to the specimen surface. Ultrasonic cleaning ~~is particularly~~ may be effective in removing the last traces of residues on a specimen surface.

78.2 Any coating metal that will interfere with the subsequent etching of the base metal should be removed before polishing, if possible. If etching is required, when studying the underlying steel in a galvanized specimen, the zinc coating should be removed before mounting to prevent galvanic effects during etching. The coating can be removed by ~~digestion~~ dissolving in cold nitric acid (HNO₃, sp gr 1.42), in dilute sulfuric acid (H₂SO₄) or in dilute hydrochloric acid (HCl). The HNO₃ method requires care to prevent overheating, since large samples will generate considerable heat. By placing the cleaning container in cold water during the stripping of the zinc, attack on the underlying steel will be ~~minimized~~.

7.3 ~~Oxidized~~ minimized. More information may be found in Test Method A 90/A 90M.

NOTE 2—Picral etchant produces little or no galvanic etching effects when used on galvanized steel.

NOTE 3—The addition of an inhibitor during the stripping of Zn from galvanized coatings will minimize the attack of the steel substrate. NEP (poethylinepolyamine) or SbCl₅ are two useful inhibitors.

8.3 Oxidized or corroded surfaces may be cleaned as described in Appendix X1.

89. Mounting of Specimens

89.1 There are many instances where it will be advantageous to mount the specimens prior to grinding and polishing. Mounting of the specimen is usually performed on small, ~~filmsy~~, fragile, or oddly shaped specimens, fractures, or in instances where the specimen edges are to be examined.

89.2 Specimens may be either mechanically mounted, mounted in plastic, or a combination of the ~~two can be used to provide optimum results~~.

8.3— ~~two~~.

9.3 Mechanical Mounting:

89.3.1 Strip and sheet specimens ~~are frequently~~ may be mounted by binding or clamping several specimens into a pack held together by two end pieces and two bolts. ~~Clamp mounting generally affords a means of rapid mounting with very good edge retention~~.

8.3.2 ~~The bolts~~.

9.3.2 ~~The~~ specimens should be tightly bound together to prevent absorption and subsequent exudation of polishing materials or etchants.

89.3.3 The use of filler sheets of a softer material alternated with the specimen may be used in order to minimize the seepage of polishing materials and etchants. Use of filler material is especially advantageous if the specimens have a high degree of surface irregularities.

89.3.4 Filler material *must* be chosen so as not to react electrolytically with the specimen during etching. Thin pieces of plastic, lead, or copper are typical materials that are used. Copper is especially good for steel specimens since the usual etchants for steels will not attack the copper.

89.3.5 Alternatively, the specimens may be coated with a layer of ~~phenolic or~~ epoxy resin before being placed in the clamp in order to minimize the absorption of polishing materials or etchants.

89.3.6 The clamp material should be similar in composition to the specimen to avoid galvanic effects that would inhibit etching. The specimen will not etch if the clamp material is more readily attacked by the etchant.

89.3.7 The clamp should preferably be of similar hardness as the specimens to minimize the rounding of the edges of the specimens during grinding and polishing.

89.3.8 Exercise care in clamping the specimen. Excessive clamping pressure may damage soft ~~specimens; however, good sealing is required to prevent absorption of polishing materials or etchants~~.

8.4— ~~specimen~~.

9.4 Plastic Mounting:

89.4.1 Specimens may be embedded in plastic to protect them from damage and to provide a uniform format for both manual and automatic preparation. This is the most common method for mounting metallographic specimens. Mounting plastics may be divided into two classes—compression mounting and castable.

8.4.2 When

9.4.2 The choice of a mounting specimens in plastic, exercise care in order to avoid rounding compound will influence the extent of specimen edges edge rounding observed during the grinding and polishing operations. There are several methods available that prevent minimize rounding. The specimens may be surrounded by hard shot, small rivets, rings, etc., of approximately the same hardness or, when using casting a castable resin, a slurry of resin and alumina may be poured around the specimen to prevent rounding. specimen. The specimens may also be plated before mounting (see Section 9 10). Many mounting procedures result in sharp edges on the mount corners. The corners should be beveled to remove any plastic mounting flash.

89.4.3 Compression Mounting—Therme are four types of compression mounting plastics used predominantly in the metallographic laboratory (see Table 2). These plastics require the use of a mounting press providing heat (up to approximately 160°C) (140-180°C) and pressure (up to approximately 30 force (27-30 MPa). The finished mounts Thermosetting plastics can be ejected hot but the best results are obtained when the finished cured mount is cooled under pressure. There are three types of thermosetting compression mounting plastics used predominantly in the metallographic laboratory. Thermoplastic compounds do not harden until cooled and therefore should not be ejected while hot. Regardless of the resin used to compression mount specimens, used, the best results are obtained when (1) the specimen is are clean and dry, and (2) the cured mount is cooled under full pressure to below 3 40°C before ejection from the press:

8.4.3.1 Wood-filled bakelite resins cure in 5 to 10 min, are relatively inexpensive, can be obtained in several colors, and are opaque. These resins have a tendency to pull away from the specimen leaving a crevice, which press. This will trap liquids that later can smear, stain, and obscure a portion of the specimen.

8.4.3.2 Diallyl phthalate resins are less likely to shrink and are more resistance to attack by etchants. They are more expensive than the phenolic resins with about the same hardness.

8.4.3.3 Filled dry epoxy resins provide ensure minimal shrinkage. Commercial resins intended for metallography are usually filled with hard material, minimizing edge rounding during preparation. These resins are the most expensive of the three types of thermosetting plastics. Cost can be reduced by first adding a layer of filled epoxy resin and filling up the remainder of the press cavity with phenolic resin.

8.4.3.4 Resins are used in a similar fashion. Because of the adhesive characteristics of the resins, a mold release agent should be applied to the surface of the mold. Do not apply the release agent to the specimen. The specimen is placed in a heated mold face down (the surface to be ground). The appropriate amount of resin is poured over the specimen, the mold is closed, and pressure is applied. The pressure is released at the end of the cure, the mold opened, and the finished mount ejected. As noted in 8.4.3, shrinkage can be minimized by cooling to room temperature under pressure. Modern automated mounting presses can apply pressure and heat, time the cure, and cool the mount under pressure.

8.4.3.5 Acrylic thermosetting resins produce transparent mounts. They require cooling under pressure. Heat and pressure must be carefully applied to avoid formation of “cotton ball” defects in the center of the mount.

8.4.4 gap formation.

9.4.4 Castable Plastics—Castable resins mounts are usually prepared at room temperature. Some may require an external heat source or applied pressure in order to cure. These resins consist of two or more components which must be mixed just prior to use. There are three four kinds of castable plastics in common use:

8.4.4.1 Acrylic resins consist of a powder and liquid, and cure rapidly (from 8 to 15 min) to a moderate hardness. These resins exhibit low abrasion resistance and a tendency to pull away from the specimen. They also tend to give off an unpleasant odor and enough heat during curing to alter the microstructure of some as-quenched steels.

8.4.4.2 Polyesters consist of two liquids, and cure to form water-clear mounts with little heat evolution, low shrinkage, and low hardness. The cure takes 1 to use (see Table 3-h and the mixing ratio is critical. They are more expensive than the acrylic resins.

8.4.4.3 Epoxy resins have the best properties concerning transparency, heat generation, shrinkage, adhesion to the specimen, and hardness of the three castable resins. They are expensive. Cure times vary broadly, from 1 to 1½ h for some formulations to 4 to 8 h for others. Some formulations require cooling and others heating.

8.4.4.4 The).

9.4.5 The molds for castable plastics are often simple cups that hold the resin until it cures. They may be reusable or not; the choice is a matter of convenience and cost. Handling castable resins requires care. They all can cause dermatitis. Manufacturers’

TABLE 2 Characteristics of Hot-Compression Mounting Compounds

Type of Compound	Characteristics
Acrylic	thermoplastic, cure time 10-15 min, optically clear, moderate shrinkage, low abrasion resistance, degraded by hot etchants
Diallyl phthalate ^A	thermosetting, cure time 5-10 min, opaque, minimal shrinkage, good resistance to etchants, moderate abrasion resistance
Epoxy ^A	thermosetting, cure time 5-10 min, opaque, very low shrinkage, good resistance to etchants, high abrasion resistance
Phenolic ^A (Bakelite)	thermosetting, cure time 5-10 min, opaque, moderate shrinkage, degraded by hot etchants, moderate abrasion resistance

^A These compounds may be filled with wood flour, glass fiber or mineral particulate.

TABLE 3 Characteristics of Castable Mounting Compounds

Type of Compound	Characteristics
Acrylic	Cure time 8-15 min, moderate shrinkage, peak curing temperature can reach 90-120°C during polymerization, low abrasion resistance, opaque to transparent
Polyester-acrylic (quartz-filled)	Cure time 8-15 min, very low shrinkage, peak curing temperature can reach 90-120°C during polymerization, high abrasion resistance, opaque
Polyester	Cure time 30-60 min, high shrinkage, peak curing temperature can reach 90- 120 C during polymerization, moderate abrasion resistance, transparent
Epoxy	Cure time ½-20 h, very low shrinkage, good adhesion, low heat generation during polymerization, moderate abrasion resistance, low viscosity (good for vacuum impregnation), transparent

recommendations for mixing and curing must be followed to obtain best results.

89.5 Mounting Porous Specimen:

89.5.1 Porous or intricate specimens may be vacuum impregnated in order to fill voids, prevent contamination and seepage, and prevent loss of friable or loose components. Impregnation is accomplished by placing the specimen in a mold ~~into~~ in a vacuum chamber ~~fitted with a funnel and~~ then introducing the resin into the mold after the chamber has been evacuated. The introduction of the resin into the mold can be accomplished either by having ~~a stopcock, funnel or stopcock~~ a stopcock fitted to the vacuum chamber ~~or by having a similar commercially available evacuation device, so that basin of the resin can be poured into present inside the mold from outside.~~ chamber. A low-viscosity resin will produce the best ~~results but ordinary metallographic resins will work well.~~ results. The vacuum chamber is then evacuated. The pressure in the chamber must remain above the critical vapor pressure of the hardener to avoid ~~evaporating~~ boiling away the hardener. After the pressure has equilibrated, the resin is introduced into the mold and the vacuum is released and air admittred to the chamber. Atmospheric pressure will force the resin into fine pores, cracks, and holes. ~~Very porous specimens may be turned using holes.~~

9.5.2 ~~If a wooden applicator after opening to the atmosphere to ensure the impregnation of the face-down side. The surface to be polished must be returned to the down-side position before the resin starts to set.~~

8.5.2 ~~If a low-viscosity resin is used, the funnel and stopcock may be eliminated. The specimen and resin is are placed in the cup mold prior to evacuation. The air in the specimen will bubble out through the resin. Exercise care to ensure the hardening agent is not evaporated during evacuation. Again, turn~~ Dipping the specimen ~~over to ensure impregnation of in the bottom side. Remember to turn the specimen back over again before the resin starts prior to se~~ placing it in the mold may help in filling voids.

89.5.3 Vacuum impregnation is an effective method for ensuring optimal results for porous metallographic mounts. It is imperative that ~~porous~~ the specimens be completely dry prior to impregnation.

89.5.4 A more rapid technique but less effective method is to lacquer the specimens with one of the formulations used by the canning industry to line food containers. The formulations are highly penetrating and the cure is a short time at low temperatures. After lacquering, the specimens are mounted in the usual fashion.

9. Plating of Specimens

9.1 Specimens such as fractures or those where it is necessary to examine the edges, are often plated to obtain good edge retention. Plating can be done electrolytically or with electroless solutions. These specimens are invariably mounted prior to the grinding and polishing procedures.

9.2 Chromium, copper, iron, nickel, gold, silver, and zinc may be electrolytically deposited although copper and nickel are predominantly used in metallographic laboratories.

9.3 ~~Thoroughly clean the specimen surface prior to plating so as to ensure good adhesion of the plating. Avoid industrial cleaning treatments that are too harsh and may cause damage to the specimen surface. Milder cleaning treatments that involve detergents, solvents, mild alkaline, or acidic solutions are recommended.~~

9.4 Ferrous metals are commonly plated electrolytically with nickel or copper. A flash coat in a copper or electroless nickel bath can be first applied for specimens that are difficult to electroplate.

9.5 Nonferrous metals may be plated with silver and the precious metals may be plated with nickel, gold, or silver.

9.6 The plating material should be softer, but not much softer, than the specimen in order to avoid differential polishing that may mask the specimen edge. The plating material should not react electrolytically with the base metal of the specimen during plating, polishing, or etching.

9.7 Electroless plating is preferred to electrolytic plating for specimens with rough, porous, or irregular surfaces, because the electroless solution provides better surface coverage and penetration.

9.8 Active metals such as zinc and aluminum are difficult to plate. Sometimes a flash cyanide copper plate can be deposited, which then can be followed by normal plating from a sulfate bath. Evaporated coatings of copper, gold, or chromium may also be used as starter coatings.

10. Plating of Specimens

10.1 Specimens such as fractures or those where it is necessary to examine the edges, are often plated to obtain good edge retention. Plating can be done electrolytically or with electroless solutions. These specimens are invariably mounted prior to the grinding and polishing procedures. Electroless plating solutions can be purchased commercially.

10.2 Thoroughly clean the specimen surface prior to plating to ensure good adhesion of the plating. Avoid industrial cleaning treatments that are too harsh and may cause damage to the specimen surface. Milder cleaning treatments that involve detergents, solvents, mild alkaline, or acidic solutions are recommended.

10.3 Chromium, copper, iron, nickel, gold, silver, and zinc may be electrolytically deposited although copper and nickel are predominantly used in metallographic laboratories.

10.3.1 Ferrous metals are commonly plated electrolytically with nickel or copper. A flash coat in a copper or electroless nickel bath can be first applied for specimens that are difficult to electroplate.

10.3.2 Nonferrous metals may be plated with silver and the precious metals may be plated with nickel, gold, or silver.

10.4 The plating material should not react galvanically with the base metal of the specimen during plating, polishing, or etching.

10.5 Electroless plating is preferred to electrolytic plating for specimens with rough, porous, or irregular surfaces, because the electroless solution provides better surface coverage and penetration.

10.6 Active metals such as zinc and aluminum are difficult to plate. Sometimes a flash cyanide copper plate can be deposited, which then can be followed by normal plating from a sulfate bath. Evaporated coatings of copper, gold, or chromium may also be used as starter coatings.

10.7 It is recommended that the plating thickness be at least 5µm.

11. Grinding and Polishing

10.1 *General*—Many

General Information

11.1 Many metals and alloys can be prepared using a similar sequence of grinding and polishing. Hard alloys may require greater pressure than soft alloys. The major differences, though, differences will be in the final polishing. Some metals and alloys will require specific combinations of abrasive and support material, but a surprising number can be handled by the same procedure. Some composite materials, however, require lapping as a critical step when traditional approaches are not satisfactory. Supplies and instructions for grinding, lapping, and polishing are readily obtainable from laboratory supply houses.

10.1.2 *Grinding*—Grinding consists of two stages—rough and fine.

10.2.1 *Rough Grinding*—Rough grinding (180 grit and coarser) is used to accomplish the following:

10.2.1.1 Flatten an irregular or damaged cut surface;

10.2.1.2 Remove scale and other surface conditions prior to mounting;

10.2.1.3 Remove substantial amounts of specimen material to reach a desired plane for polishing;

10.2.1.4 Remove plastic mounting flash, level the mount surface, and bevel mount edges before fine grinding; and

10.2.1.5 Rough grinding may be performed on belts or rotating wheels. In some methods 45 or 30 µm diamond abrasives are used on hard platens:

10.2.2 *Fine Grinding*—In fine grinding, the specimen is ground on successively finer abrasive papers using water to wash away grinding debris and to act as a lubricant. The specimen should be cleaned between successive papers to prevent carryover of coarser abrasive.

10.2.2.1 *Grinding* can be done in a number of ways, ranging from rubbing the specimen on a stationary piece of abrasive paper to the use of automatic devices. The choice of method depends on the number and type of specimens to be done, financial considerations; and requirements such as flatness, uniformity, flatness and so forth. Grinding on abrasive-coated rotating disks using hand-held specimens is uniformity.

11.2.1 Abrasive grit size designations in this practice are expressed in the traditional method. Mechanical devices to hold ANSI (American National Standards Institute) or CAMI (Coated Abrasives Manufacturers Institute) system units with the specimen against abrasive-covered rotating disks are becoming increasingly common. Many corresponding FEPA (European Federation of Abrasive Producers) numbers in parentheses. Table 4 provides a correlation between these machines permit automated grinding, providing surfaces superior to hand-held specimens:

10.2.2.2 *Grinding* two systems and the approximate median particle diameter for a given size in micrometres.

11.2.2 Grinding should start with the finest paper, platen or stone capable of flattening the specimen and removing the effects of prior operations, such as sectioning. The next paper subsequent steps should remove the effects of the prior paper previous ones in a short time. A typical sequence Grinding consists of papers might be 240, 320, 400, two stages- planar (rough) and 600-grit abrasive papers. Depending fine.

11.2.3 Planar or rough grinding [240 grit (P220) and coarser] may be performed on the smoothness of the specimen surface, belts, rotating wheels or stones. In some of the coarser paper grades can be skipped:

10.2.2.3 In the hand-held methods, the specimen is rotated 90° between papers diamond abrasives are used on rigid platens. Planar grinding may be used to determine when accomplish the prior set of scratches have been removed. The specimen should also be moved back following:

11.2.3.1 Flatten an irregular or damaged cut surface;

11.2.3.2 Remove sectioning damage, scale and forth across the paper other surface conditions prior to prevent grooving mounting;

11.2.3.3 Remove substantial amounts of the specimen. At the end of the grinding on each paper, the surface of the specimen

TABLE 4 European/USA Grit Grade Comparison Guide

Grit Number	FEPA	ANSI/CAMI	
	Size (µm)	Grit Number	Size (µm)
P120	125.0	120	116.0
P150	100.0	180	78.0
P220	68.0	220	66.0
P240	58.5
P280	52.2	240	51.8
P320	46.2
P360	40.5	280	42.3
P400	35.0	320	34.3
P500	30.2
P600	25.8	360	27.3
P800	21.8	400	22.1
P1000	18.3	500	18.2
P1200	15.3	600	14.5
P1500	12.6	800	11.5
P2000	10.3	1000	9.5
P2500	8.4	1500	8.0
P4000 ^A	5.0

^A Not found in the FEPA grading system.

ANSI—American National Standards Institute

CAMI—Coated Abrasives Manufacturers Institute

FEPA—European Federation of Abrasive Producers

and its mount, if any, should be flat with one set of unidirectional grinding scratches:

10.2.2.4 Most of the device material to reach a desired plane for automatic grinding move polishing.

11.2.3.4 Level the specimen around a rotating wheel covered with abrasive so that mount surface.

11.2.4 In fine grinding, damage to the specimen follows an epicycloid path. In some devices, incurred from the specimen rotates on its own axis as well. planar or rough grinding step must be removed. The scratch pattern now consists of random arcs. Deciding when the previous scratches have been removed is more difficult than with directional grinding. The specimen surface should show uniform scratches before proceeding is either ground on successively finer abrasive papers (using water to the next step. Cleaning between stages may be necessary to prevent carryover of abrasives and contamination of wash away grinding surfaces. Manufacturer's instructions will include suggested machine settings for grinding various metals.

10.2.2.5 After debris and to act as a coolant) or on a rigid disc or cloth charged with a suitable abrasive.

11.2.5 After all grinding is done, the specimen must be cleaned thoroughly. Ultrasonic cleaning in a water-detergent bath is recommended. In hand operations, the hands must be washed also, exercising care to clean beneath fingernails:

10.3- water/soap solution containing a corrosion inhibitor may prove beneficial.

11.3 Polishing—Polishing is usually distinguished from grinding by the use of loose abrasive ($\leq 6\mu\text{m}$) embedded in an appropriately lubricated supporting surface. The choice of abrasive, lubricant, and polishing surface support is often specific to the metal and the object of the investigation.

10.3.1 The use of graded diamond paste or suspensions as the abrasive investigation. Polishing can reduce the number of combinations markedly. Those supports most commonly used are non-napped cloths such as nylon or nonwoven textiles available be divided into rough and fine (final) stages.

11.3.1 Rough polishing is often sufficient for metallographic purposes.

10.3.2 The final polish routine evaluations like microindentation hardness and grain size.

11.3.2 When fine polishing is required, it may be 1-µm performed with diamond or an oxide slurry step or both. The choice of final polishing abrasive type and size is dictated by the hardness of the specimen. For high-resolution work, this instance, a 1µm diamond final polish may be followed by is often sufficient for many grades of steel, however, softer steels and non-ferrous materials often require an additional polishing on a short nap synthetic suede using step with an aqueous oxide slurry or suspension of 0.05-µm gamma alumina SiO_2 or colloidal silica. Other abrasives and supports often Al_2O_3 . Final polishing cloths are required depending on the particular task generally softer and higher in hand. This step should nap than rough polishing cloths. Therefore, polishing time and force must be kept to the shortest time possible a minimum to prevent avoid artifacts such as edge rounding, pitting, or other artifacts. Twenty to forty seconds should be sufficient if the previous steps have been correctly performed.

10.3.3 Careful rounding and relief.

11.3.3 Careful cleaning of the specimen between stages is mandatory to prevent contamination by coarser abrasive. Ultrasonic cleaning is recommended between each may be effective.

11.3.4 The polishing step.

10.3.4 The polishing operations may be conducted by hand manual or by automatic methods.

10.3.4.1 Hand automated methods (preferred).

Manual (Hand-held) Methods

11.4 When grinding manually, the specimen should be moved back and forth across the paper to allow for even wear. Between grinding steps, the specimen should be rotated 45-90°. At the end of grinding on each paper, the surface of the specimen and its

mount, if any, should be flat with one set of unidirectional grinding scratches.

11.5 Manual polishing methods consist of holding the specimen by hand against an abrasive-charged rotating wheel and moving the specimen in an elliptical or a circular path around the wheel against the direction of rotation of the wheel. The specimen should be held firmly in contact with the wheel. Just how firm and just how fast to go around wheel.

11.6 The amount of force applied along with the rate of movement of the specimen during grinding and polishing is a matter of experience and personal preference and experience. In the preparation of advanced materials, difficult materials such as thermally sprayed coatings or composites, the operating parameters must be strictly controlled.

10.1.47 A traditional manual preparation sequence consists of a series of grinding and polishing:

10.4.1 Many polishing steps and may be similar to those listed in Table 5.

Automated Methods

11.8 Many styles of automated specimen preparation machinery are available. The most common Most units can perform all grinding and polishing steps. They Many use disk fixtures holders capable of accommodating multiple specimens; the specimens remain in the fixture throughout the preparation. Major advantages in using automatic of automated grinding and polishing procedures are the consistent quality of specimen preparation and the substantial decrease in time required for preparing large number time. Therefore, automated techniques are recommended over manual techniques.

11.9 Most of specimens. Careful attention to cleanliness the devices for automated grinding and polishing move the specimen around a rotating wheel covered with abrasive so that the specimen follows an epicycloid path. In some devices, the specimen rotates on its own axis as well. The resulting scratch pattern now consists of cross-contamination from abrasives and residues randomly oriented arcs. Deciding when the previous scratches have been removed is more difficult than with directional (manual) grinding. The specimen surface should show uniform scratches before proceeding to the next step. Cleaning between steps, particularly in polishing, stages is required. Ultrasonic cleaning is recommended.

10.4.2 An initial coarse grinding step is required to make all specimen surfaces co-planar prevent carryover of abrasives and contamination of subsequent preparation surfaces.

11.10 Table 5 illustrates a traditional automated preparation method. This method uses conventional SiC papers for grinding and is usually used to remove damage to suitable for all but the specimens from sectioning operations. Fine hardest of materials. Tables 6 and 7 are preparation methods that utilize rigid grinding uses either a graded abrasive paper series discs or a single step with an intermediate (9 or cloths for fine grinding. The method in Table 6 μm) diamond abrasive on a permanent, grooved wheel has been shown to prepare the specimens for polishing. As an alternative, lapping platens may be used to replace effective for the finegrinding paper sequence. This approach offers the greatest benefits when very hard or highly dissimilar preparation of materials must harder than HRC45. The method in Table 7 may be prepared.

10.4.3 One polishing step will often suffice for examination up to 100X, using a 3 or 1 μm diamond abrasive on napless cloth. Two steps will suffice for almost all requirements, with the second step using a finer diamond on a napless or low-nap cloth. A third step with fine alumina or colloidal silica on a low-nap cloth can be used for final polishing but relief polishing will be encountered proportionate to the specimen load and polishing time used.

11. preparation of materials softer than HRC45. These procedures may produce excellent results outside of the recommended hardness ranges.

12. Special Procedures

11.2.1 Occasionally, the very metallographer is faced with the preparation of unusual unfamiliar specimens or with special

TABLE 5 Preparation Method 1 (General Use)

Surface	Lubricant	Abrasive Type/Size ANSI (FEPA)	Time sec.	Force ^A N(lbf)	Platen RPM ^B	Rotation
Planar Grinding paper/stone	water	120–320 (P120–400) grit SiC/Al ₂ O ₃	15–45	20–30 (5–8)	200–300 ^C	CO ^D
Fine Grinding						
paper	water	240 (P220) grit SiC	15–45	20–30 (5–8)	200–300	CO
paper	water	320 (P500) grit SiC	15–45	20–30 (5–8)	200–300	CO
paper	water	600 (P1200) grit SiC	15–45	20–30 (5–8)	200–300	CO
Rough Polishing low/no nap cloth	compatible lubricant	6μm diamond	120–300	20–30 (5–8)	100–150	CO
Final Polishing						
med./high nap cloth	compatible lubricant	1μm diamond	60–120	10–20 (3–5)	100–150	CO
synthetic suede ^E	water	0.04μm colloidal silica or 0.05μm alumina	30–60	10–20 (3–5)	100–150	CONTRA ^F

^A Force per 30 mm (1¼ in.) diameter mount.

^B Power heads generally rotate between 25 and 150 rpm.

^C High-speed stone grinders generally rotate at greater than 1000 rpm.

^D Complimentary rotation, surface and specimen rotate in same direction.

^E Optional step.

^F Contra rotation, surface and specimen rotate in opposite directions.

TABLE 6 Preparation Method 2 for Harder Materials \geq HRC 45 (450 HV)

Surface	Lubricant	Abrasive Type/Size ANSI (FEPA)	Time sec.	Force ^A N(lbf)	Platen RPM ^B	Rotation
Planar Grinding paper/stone	water	120–320 (P120–400) grit SiC/Al ₂ O ₃	15–45	20–30 (5–8)	200–300 ^C	CO ^D
Fine Grinding rigid disc	compatible lubricant	6–15 μ m diamond	180–300	20–30 (5–8)	100–150	CO
Rough Polishing low/no nap cloth	compatible lubricant	3–6 μ m diamond	120–300	20–30 (5–8)	100–150	CO
Final Polishing med./high nap cloth	compatible lubricant	1 μ m diamond	60–120	10–20 (3–5)	100–150	CO
synthetic suede ^E	water	0.04 μ m colloidal silica or 0.05 μ m alumina	30–60	10–20 (3–5)	100–150	CONTRA ^F

^A Force per 30 mm (1¼ in.) diameter mount.

^B Power heads generally rotate between 25 and 150 rpm.

^C High-speed stone grinders generally rotate at greater than 1000 rpm.

^D Complimentary rotation, surface and specimen rotate in same direction.

^E Optional step.

^F Contra rotation, surface and specimen rotate in opposite directions.

TABLE 7 Preparation Method 3 for Softer Materials \leq HRC 45 (450 HV)

Surface	Lubricant	Abrasive Type/Size ANSI (FEPA)	Time sec.	Force ^A N(lbf)	Platen RPM ^B	Rotation
Planar Grinding paper/stone	water	120–320 (P120–400) grit SiC/Al ₂ O ₃	15–45	20–30 (5–8)	200–300 ^C	CO ^D
Fine Grinding heavy nylon cloth	compatible lubricant	6–15 μ m diamond	180–300	20–30 (5–8)	100–150	CO
Rough Polishing low/no nap cloth	compatible lubricant	3–6 μ m diamond	120–300	20–30 (5–8)	100–150	CO
Final Polishing med./high nap cloth	compatible lubricant	1 μ m diamond	60–120	10–20 (3–5)	100–150	CO
synthetic suede ^E	water	0.04 μ m colloidal silica or 0.05 μ m alumina	30–60	10–20 (3–5)	100–150	CONTRA ^F

^A Force per 30 mm (1¼ in.) diameter mount.

^B Power heads generally rotate between 25 and 150 rpm.

^C High-speed stone grinders generally rotate at greater than 1000 rpm.

^D Complimentary rotation, surface and specimen rotate in same direction.

^E Optional step.

^F Contra rotation, surface and specimen rotate in opposite directions.

situations. Anticipation of every possible situation is, of course, impossible but some guidance can be offered. ~~Common sense is imperative.~~

11.1.1 Electrolytic offered.

12.1.1 When used properly, electrolytic polishing ~~produces totally~~ can produce near deformation-free surfaces but works best on solid solution alloys. Once the operating parameters are set, specimens can be prepared quickly. See Guide E 1558.

12.1.2 Vibratory polishing produces excellent results ~~on a number of alloys.~~ many materials. Although slow, a number of specimens can be prepared ~~simultaneously,~~ resulting in a high through-put. ~~simultaneously.~~ It is especially advantageous for soft materials.

12.2 Porous Specimens—Specimens with continuous or open pores can be vacuum-impregnated (see 9.5) with ~~plastic or some other liquid that will solidify.~~ epoxy. Specimens with closed pores are mounted by a suitable method, ground through the fine grinding stage, cleaned, and dried thoroughly. The surface is then wiped with a liquid epoxy mounting compound, usually the same material used to mount the specimen, to seal the pores. After hardening, the last fine-grinding stage is repeated to remove the excess material, and specimen preparation is continued as usual. The choice of liquid epoxy for impregnation ~~or sealing~~ depends on the nature of the ~~sample.~~ specimen. It ~~should,~~ of course, ~~should~~ be inert toward the specimen.

11.2.1 Impregnation can be accomplished by vacuum impregnation (see 8.4.4.5 through 8.4.4.8) in the specimen.

11.3—

12.3 Composite Materials—Composite materials, particularly hard fibers in a soft matrix or wires in a soft insulation, can be particularly difficult to prepare. The best approach is to first seal or impregnate pores or holes. Then grind carefully, using copious lubrication. The grinding surface must be kept flat and firm. In the polishing stages, the substrate should have no nap and should be fairly hard. Diamond abrasive is recommended. Both will minimize rounding of the hard components. Sometimes, a compromise will have to be made between accepting a few ~~defects (scratches)~~ artifacts such as scratches or rounded edges. If automatic methods are available, high-polishing pressures and high-nap substrates may provide an alternative method.

11.4— edges.

12.4 Coated Materials:

142.4.1 Coated metals, such as galvanized steel, electroplated metal, enamel ware, and so forth, can be considered a variety of composite materials. They present problems of their own, such as flaking, chipping, and rounding. For example, some coatings are so thin as to be unresolvable on simple cross sections (tinplate). Other problems are the presence of a soft coating on a harder substrate (galvanized steel) or a hard brittle coating on a soft substrate (porcelain enamel on aluminum).

142.4.21.1 The problem of thin coatings can be handled by using a taper mount. In this method, the specimen is mounted so that the plane of polish is at a small angle to the plane of the surface. For example, a tapered plug is inserted in the mounting press with the taper up. A blank tapered mount is prepared. Masking tape is wrapped around the circumference of the mount to make a well on the tapered end. A small amount of epoxy mounting compound is mixed. The specimen, cut to fit inside the well, is wetted with the epoxy and laid on the face of the tapered mount, coated side up. Using a probe, the specimen is pressed down firmly onto the tapered face. The balance of the epoxy compound is added and allowed to harden. The mounted specimen is ground and polished on the epoxy face in the conventional manner exercising care that the plane of polish is perpendicular to the cylindrical axis of the mount. This is easily done with most automatic grinding machines.

142.4.31.2 The problem of soft coatings can be solved by the use of a suitable backup. A piece of spring steel is useful to hold the backup in place, or the backup may be cemented to the specimen. The cement can act as an insulation to minimize galvanic effects. Caution: some cements will dissolve in epoxy mounting compounds. A particularly suitable backup is another piece of the same material, with the coating sandwiched in. Another solution is to add another coating, for example, electroplate. However, this may introduce undesirable galvanic effects during etching. Galvanic problems may arise also from the interaction of the coating and its substrate. The mounting procedure used must result in excellent adhesion to the coated surface to minimize edge rounding. If edge rounding persists, the polishing time and applied force may have to be decreased.

142.4.41.3 Hard coatings on softer substrates can be mounted with a backup piece or a hard-filled mounting compound. Diamond abrasives on a napless cloth will minimize surface relief during polishing.

142.5 Fragile specimens should be mounted in one of the liquid cold castable mounting formulations. Vacuum impregnation will ensure filling of holes and cavities (see section 8.4.4.5); 9.5). Thin walls can be reinforced by electroless nickel plating, which will alleviate the rounding problem.

142.6 Likewise, friable specimens can be bound together by impregnation with plastic or by electroless nickel plating, or both. Further guidance can be found in texts on preparation of mineralogical specimens.

12. Precision and Bias

~~12.1 Because use of this practice does not produce numerical results, no statement of precision or bias is possible.~~

13. Precision and Bias

~~13.1 Because use of this practice does not produce numerical results, no statement of precision or bias is possible.~~

14. Keywords

~~14.1 alloys; grinding; metallography; metals; mounting; polishing; sectioning; specimen preparation (metallographic)~~

APPENDIXES

(Nonmandatory Information)

X1. CLEANING SPECIMENS

X1.1 Metallographers frequently need to clean specimens. In some instances, the adherent debris, oxidation, or corrosion product must be collected for analysis, for example, by X-ray diffraction. In other cases, the adherent matter is of no interest, it merely needs to be removed. If the underlying surface is of no interest, the surface can be shot blasted, wire brushed, or ground. However, if the underlying surface is important, for example, a fracture surface, then the cleaning operation must do as little damage as possible. These different aims of the cleaning operation must be kept in mind before formulating the cleaning program.

X1.2 When the adherent material is to be analyzed, a variety of procedures may be applied depending upon whether or not the underlying surface can or cannot be damaged.

X1.2.1 In the case of debris or corrosion product on the surface of a part, a stylus, scalpel, or other sharp object can be used to scrape off or pry off enough material for analysis. This will do some damage to the surface, but it will be localized.

X1.2.2 As an alternative, use cellulose acetate replicating tape to remove surface debris by the extraction replica approach. A number of approaches have been developed and are described in STP 547³ as well as in many textbooks on electron microscopy. Generally, thick (0.127 mm or 0.005 in.) tape is employed. One surface is moistened with acetone and then pressed against the

³ "Manual Electron Metallography Techniques," 1973. Available from ASTM Headquarters. Request STP 547.

debris-coated surface. After it dries, strip off the tape in the same way as you would remove adhesive tape. The debris will adhere to the tape.

X1.3 When the surface is to be examined, but the adherent debris will not be analyzed, there are several approaches that can be used. Always try the simplest, safest methods first. For example, use a blast of compressed air to remove any loosely adherent material. A soft camel-hair brush or a soft toothbrush may also be useful for removing loosely adherent matter.

X1.3.1 If the techniques in X1.3 do not suffice, try aqueous solutions, organic solvents, or alcohol with an ultrasonic cleaner. Aqueous solutions (8 g of Alconox per litre of warm water) containing Alconox⁴, a detergent, have been found (1, 2) to be effective. Follow the Alconox bath with washing rinsing under running water, then dry. Organic solvents, such as acetone, ethyl methyl ketone, toluene, xylene, or alcohol (ethanol is preferable to methanol because of potential health problems with the latter) are also very effective. Before choosing one of these solutions, be sure that it will not adversely affect the material being cleaned. Avoid use of chlorinated organic solvents (such as trichlorethylene or carbon tetrachloride) due to their carcinogenic nature. Repeated replication, as described in X1.2.2, is an effective method for cleaning fractures (3, 4).

X1.3.2 When the procedures in X1.3 and X1.3.1 are unsuccessful, more drastic methods are required. Electrolytic cleaning solutions (Table X1.1), have been found to be quite useful. An inert material (stainless steel, graphite, or platinum, for example) is used as an anode, while the specimen is the cathode in the electrolytic cell. Some of these solutions can generate dangerous fumes, hence they should be used under a hood with care. Endox 214⁵ has been found (1) to be useful for cleaning heavily rusted steel fractures.

X1.3.3 Cathodic cleaning solutions or acid-inhibited baths have also been employed to clean fractures (3, 5). However, as the degree of corrosion or oxidation increases, fracture features will be destroyed to a greater extent and cleaning, while it can remove the surface deposits, cannot restore damaged fracture features.

X1.3.4 A number of proprietary rust removal solutions have been developed. These are premixed and used directly out of the container. Two such products are described in Refs 6 and 7.

⁴ Alconox is available from Alconox, Inc., New York, NY 10003. An equivalent can be used.

⁵ Endox 214 is available from Enthone, Inc., 350 Frontage Rd., West Haven, CT 06516. An equivalent can be used.

TABLE X1.1 Cleaning Solutions for Use When Standard Methods Are Inadequate

6N HCl plus 2 g/L Hexamethylene tetramine ^A	Immerse specimen in solution for 1 to 15 min. Good for steels. Cleaning action can be enhanced by light brushing or by brief (5 s) periods in an ultrasonic cleaner.
3 mL HCl 4 mL 2-Butyne-1, 4 diol inhibitor 50 mL water ^B	Use a fresh solution at room temperature. Use in an ultrasonic cleaner for about 30 s.
49 mL water 49 mL HCl 2 mL Rodine-50 inhibitor ^C	Wash specimen in alcohol for 2 min in an ultrasonic cleaner before and after a 2-min ultrasonic cleaning period with the inhibited acid bath.
6 g sodium cyanide 6 g sodium sulphite 100 mL distilled water ^{DEF}	Electrolytic rust removal solution. Use under a hood with care. Use 100-mA/cm ² current density for up to 15 min.
10 g ammonium citrate 100 mL distilled water ^G	Use solution heated to 30°C (86°F).
70 mL orthophosphoric acid 32 g chromic acid 130 mL water ^H	Recommended for removing oxides from aluminum alloy fractures (some sources claim that only organic solvents should be used).
8 oz endox 214 powder 1000 mL cold water (add small amount of Photo-Flo) ^{I,J}	Use electrolytically at 250-mA/cm ² current density for 1 min with a Pt cathode to remove oxidation products. Wash in an ultrasonic cleaner with the solution for 1 min. Repeat this cycle several times if necessary. Use under a hood.

^A deLeiris, H., et al, "Techniques for Removing Rust from Fractures of Steel Parts that are to be Examined by Electron Microfractography," *Mem. Sci. Rev. Met.*, Vol 63, No. 5, May 1966, pp. 463–472.

^B Dahlberg, E. P., "Techniques for Cleaning Service Failures in Preparation for Scanning Electron Microscope and Microprobe Analysis," *Scanning Electron Microscopy*, 1974, Part IV, pp. 911–918.

^C Brooks, C. E., and Lundin, C. D., "Rust Removal from Steel Fractures—Effect on Fractographic Evaluation," *Microstructural Science*, Vol 3A, Elsevier, NY, 1975, pp. 21–33.

^D deLeiris, H., et al, "Techniques for Removing Rust from Fractures of Steel Parts That Are to be Estimated by Electron Microfractography," *Mem. Sci. Rev. Met.*, Vol 63, No. 5, May 1966, pp. 463–472.

^E Russ, J. C., and Miller, G. A., "Effect of Oxidization on the Electron Fractographic Interpretation of Fractures in Steel," *JISI*, December 1969, pp. 1635–1638.

^F Pickwick, K. M., and Smith, E., "The Effect of Surface Contamination in SEM Fractographic Investigations," *Micron*, Vol 3, No. 2, 1972, pp. 224–237.

^G Interrante, C. G., and Hicho, G. E., "Removal of Iron-Sulfide Deposits from Fracture Surfaces," *ASTM STP 610*, 1976, pp. 349–365.

^H Beachem, C. D., *The Interpretation of Electron Microscope Fractographs*, NRL Report 6360, U.S. Government Printing Office, Jan. 21, 1966.

^I Yuzawich, P. M., and Hughes, C. W., "An Improved Technique for Removal of Oxide Scale from Fractured Surfaces of Ferrous Materials," *Prakt. Met.*, Vol 15, April 1978, pp. 184–195.

^J Goubau, B., and Werner, H., "Microfractographic Investigation of Fracture Surfaces Coated With Magnetite," *Prakt. Met.*, Vol 17, No. 5, May 1980, pp. 209–219.

X1.3.5 Cleaning can also be accomplished by argon-ion bombardment (6) or by use of a glow-discharge method (7, 8). These methods require specialized equipment.

X2. APPLIED LOAD CONVERSIONS

X2.1 Automated preparation machines commonly display force in either pound-force (lbf) or newtons (N). The ability to convert from one unit to the other may be necessary when trying to interpret a documented procedure.

X2.1.1 To convert from pound-force to newton multiply the pound-force value by 4.5.

X2.1.2 To convert from newton to pound-force multiply the newton value by 0.225.

X2.2 When multiple specimens of equal contact area are held in a holder, the applied force must be divided by the number of specimens in the holder to determine the load per specimen.

X2.2.1 Some automated machines apply force individually to each specimen. In this case it is necessary to divide the force by the contact area to determine the load per specimen.

X2.3 Caution should be taken when using automated machines that display pressure in pound-force per square inch (psi). Typically, the machine is displaying the air pressure within the loading cylinder and not the actual pressure applied to either the specimen holder or individual specimen.

X2.4 When converting from a force to a pressure, the surface area of the specimen(s) must be determined. The value of force is then divided by the contact area to determine the required pressure.

X3. PROCEDURE IMPROVEMENT

X3.1 To improve the preparation of a particular material, try one of the preparation methods described in Table 5, Table 6, or Table 7. Following are general guidelines that may help improve results.

X3.2 If a material is being prepared for the first time, the surface should be microscopically examined after every step.

X3.3 Before proceeding to the next step, be sure that all deformation and artifacts from the previous step, such as scratches, pull-outs or embedded grains, are completely removed. It is difficult to identify when an artifact was introduced if the specimen is not examined prior to the final step. You must know when the artifact was introduced in order to improve the method.

X3.4 Keep the preparation times as short as possible. Excessive preparation wastes consumables and may introduce artifacts such as relief and edge rounding.

X3.5 New consumables such as polishing cloths or diamond grinding products may need to be “broken in” for a short period prior to use.

X3.6 The following section lists common preparation artifacts and prevention measures.

X3.7 *Scratches*—Scratches are grooves in the surface of the specimen produced by the points of abrasive particles.

X3.7.1 Make sure that after planar grinding the surface of all of the specimens in the holder exhibit the same uniform scratch pattern over the entire specimen. Repeat the planar grinding step if necessary.

X3.7.2 Clean the specimens and holder carefully after each step to avoid contamination.

X3.7.3 If there are still scratches left over from the previous step after finishing the current step, increase the preparation time by 25 to 50 %. If this does not work then you should consider altering the method by inserting an intermediate step.

X3.8 *Deformation*—Deformation can be classified by two types, elastic and plastic. Elastic deformation disappears when the applied load is removed. Plastic deformation, often called cold work, can be induced during sectioning, mounting, grinding, lapping or polishing. Residual plastic deformation can first be seen after etching. Only deformation that was introduced during metallographic preparation can be eliminated with procedure modification. Deformation from manufacturing operations such as bending, drawing and stretching are not considered because they cannot be removed by altering the preparation method.

X3.8.1 If the deformation is visible in brightfield in the unetched condition, please see X3.7.3 Scratches, for tips on how to improve the preparation.

X3.8.2 If after etching, the deformation is restricted to single or a few grains then it is minimal and may be removed by repeating the previous step.

X3.8.3 If after etching, the deformation is well defined covering several grains or even the whole specimen, then it may have been recently introduced. Check and clean the polishing cloth for possible contamination. Replace the cloth if results do not

improve (see section X.2.1.4). Repeat the previous step.

X3.8.4 If after etching, the deformation is in the form of long, blunt lines covering several grains (with possible interruptions) then it may have been introduced from an earlier stage. Repeat the procedure starting from the fine grinding stage.

X3.9 Smearing—Smearing is the flow of material at the surface of the specimen. It is the result of material being “pushed” across the surface instead of being cut.

X3.9.1 Check the amount of lubricant. Smearing most often occurs when lubrication levels are too low. Increase or change the lubricant to eliminate smearing.

X3.9.2 Check the applied load. Excessive loads can result in smearing. Reduce the load to eliminate smearing.

X3.9.3 Check the abrasive size. Abrasives grains that are too small may not be effective in material removal. Increase the abrasive grain size.

X3.10 Edge Rounding—Edge rounding results when the edge of the specimen abrades at a greater rate than the body of the specimen.

X3.10.1 Mount the specimen. Unmounted specimens always exhibit greater edge rounding than mounted specimens.

X3.10.2 Use the correct mounting compound. There should be minimal shrinkage of the mounting compound away from the specimen. Try to match the abrasion resistance of the mounting compound closely to that of the specimen. See Section 9.

X3.10.3 If the edge rounding first occurred during grinding, consider changing the grinding substrate to a less resilient form. Also consider changing the abrasive type. Diamond abrasive is often more effective than SiC at cutting hard materials.

X3.10.4 Reduce polishing times as much as possible. Long polishing procedures often result in excessive edge rounding.

X3.10.5 Reduce applied load. Normally lower loads result in less edge rounding.

X3.10.6 Change the polishing lubricant. Oil or water/oil type lubricants may help preserve edges.

X3.10.7 Change the polishing cloth. Less resilient cloths produce better edges.

X3.10.8 If the preceding steps are ineffective then consider plating the specimen. See Section 10.

X3.11 Relief—Relief results when material from different phases is removed at different rates due to varying hardness or wear rate of individual phases.

X3.11.1 Relief normally first occurs during polishing. However, if there are extreme differences in the hardness between phases it may occur during grinding. If this is the case then an alternative grinding method should be considered. See Tables 5 and 6.

X3.11.2 Polishing time should be kept to a minimum.

X3.11.3 Polishing cloths that have less resiliency produce less relief (see Edge Rounding in X3.10).

X3.11.4 The polishing abrasive should be at least 2.5 times harder (on the Vickers scale) than the hardest phase being polished.

X3.12 Pull-outs—Pullouts are the cavities left in the surface after grains or particles are torn out during preparation.

X3.12.1 Avoid high loads during grinding and polishing.

X3.12.2 Do not use coarse abrasives for Planar or Fine grinding steps.

X3.12.3 Do not make large abrasive size jumps between preparation steps. Insert an intermediate step if necessary.

X3.12.4 Napless polishing cloths produce less pull-out than napped cloths.

X3.12.5 Every step has to remove the damage from the previous step, and has to introduce as little damage as possible.

X3.12.6 Check the specimen after every step in order to find out when the pull-out occurs.

X3.13 Gaps—Gaps are the voids between the mounting compound and the specimen. Gaps can result in a variety of preparation artifacts such as edge rounding, contamination and staining.

X3.13.1 Clean and dry the specimen thoroughly prior to mounting.

X3.13.2 Select a mounting compound with low shrinkage (see Section 9).

X3.13.3 For hot compression mounting, cool the specimen under pressure.

X3.13.4 For castable mounting compounds, avoid high curing temperatures. It may be necessary to cool the specimen during the curing.

X3.13.5 Specimen height should be kept as low as practical to minimize gaps when using hot compression mounting.

X3.14 Contamination—Contamination is material from a source other than the specimen itself which is deposited on the specimen surface during grinding or polishing.

X3.14.1 Thoroughly clean the specimen between preparation steps (see 11.2.5, 11.3.3).

X3.14.2 Store grinding and polishing discs in a clean, dust-free environment.

X3.14.3 Change grinding or polishing substrate/abrasive if necessary.

X3.15 Embedded Abrasive—Embedded abrasive results when loose grinding, lapping, or polishing abrasive sticks into the surface of the specimen.

X3.15.1 Embedded abrasive is most common with soft non-ferrous materials.

X3.15.2 Change to a more resilient grinding substrate.

X3.15.3 Use a block of paraffin or candle to “pick up” loose SiC particles on fine grit papers. This is done by lightly passing the paraffin block across the paper.

X3.15.4 Change to a more resilient polishing substrate when using diamond abrasives that are less than 3µm in diameter.

X3.15.5 Change to an oil or water/oil-based polishing lubricant.

X3.16 *Lapping Tracks*—Lapping tracks are indentations on the specimen surface made by abrasive particles moving freely (rolling) on a hard surface. Lapping tracks can be produced during both grinding and polishing.

X3.16.1 Change to a more resilient grinding or polishing substrate.

X3.16.2 Increase the applied load in 10 % increments until the lapping tracks disappear.

X3.16.3 Employ optimal dynamics.

REFERENCES

- (1) Yuzawich, P. M., and Hughes, C. W., “An Improved Technique for Removal of Oxide Scale from Fractured Surfaces of Ferrous Materials,” *Practical Metallography*, Vol 15, April 1978, pp. 184–195.
- (2) Zipp, R. D., “Preservation and Cleaning of Fracture for Fractography,” *Scanning Electron Microscopy*, Part I, 1979, pp. 355–362.
- (3) Russ, J. C., and Miller, G. A., “Effect of Oxidation on the Electron Fractographic Interpretation of Fractures in Steel,” *Journal, Iron and Steel Institute*, Vol 207, December 1969, pp. 1635–1638.
- (4) Pickwick, K. M., and Smith, E., “The Effect of Surface Contamination in SEM Fractographic Investigations,” *Micron*, Vol 3, No. 2, 1972, pp. 224–237.
- (5) Lane, G. S., and Ellis, J., “The Examination of Corroded Fracture Surfaces in the Scanning Electron Microscope,” *Corrosion Science*, Vol 11, September 1971, pp. 661–663.
- (6) Macmillan, J. W., and Flewitt, P. E. J., “Assessment of Methods for Cleaning Oxide from Fracture Surfaces for Examination in a Scanning Electron Microscope,” *Micron*, Vol 6, No. 3/4, 1975, pp. 141–146.
- (7) ASTM Task Group E24.02, “Clean Surfaces for Fractography,” ASTM STP 600, ASTM, Philadelphia, PA, 1976, pp. 251–253.
- (8) Hajicek, D. J., et al, “Development of a Cleaning Method for SEM Fractographic Analysis of Impulsively Loaded Cylinders,” *Microstructural Science*, Vol 5, Elsevier, NY, 1977, pp. 413–421.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).