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Standard Guide for Preparation of Metallographic Specimens¹

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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 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, 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

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. 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 two component castable plastic. One component is the resin and the other hardener. Both components can be liquid or one liquid and a powder. Castable mounts generally do not require heat and 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 first grinding step in a preparation procedure used to bring all specimens into the same plane of polish. It is unique to 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.

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² *Annual Book of ASTM Standards*, Vol 03.01.

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

5.1 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:

5.1.1 *General Studies or Routine Work*—Specimens should be chosen from locations most likely to reveal the maximum variations within the material under study. For example, specimens 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 could be at a minimum. In the examination of strip or wire, test specimens could be taken from each end of the coils.

5.1.2 *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 from a sound area for a comparison of structures and properties.

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

5.2 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 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.

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

5.3.1 Variations in structure from center to surface,

5.3.2 Distribution of nonmetallic impurities across the section,

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

5.3.4 Depth of surface imperfections,

5.3.5 Depth of corrosion,

5.3.6 Thickness of protective coatings, and

5.3.7 Structure of protective coating.

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

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

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

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

5.4.4 The microstructure attained with any heat treatment.

5.5 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.

6. Size of Metallographic Specimens

6.1 For convenience, 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 cylindrical. The height of the specimen should be no greater than necessary for convenient handling during polishing.

6.1.1 Larger specimens are generally more difficult to prepare.

6.1.2 Specimens that are, fragile, oddly shaped or too small to be handled readily during polishing should be mounted to ensure a surface satisfactory for microscopical study. There

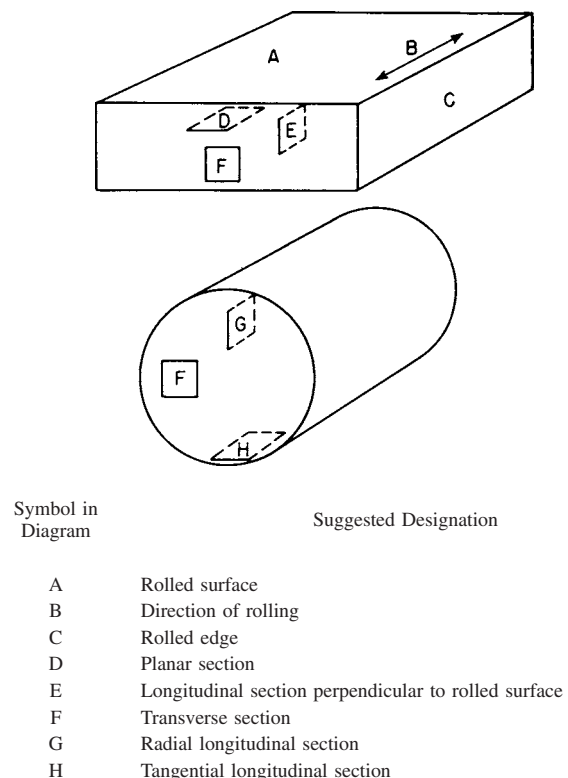


FIG. 1 Method of Designating Location of Area Shown in Photomicrograph.

are, based on technique used, three fundamental methods of 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

8.1 Cleanliness (see Appendix X1) during specimen preparation is essential. All greases, oils, coolants and residue from cutoff blades on the specimen should be removed by some suitable organic solvent. Failure to clean thoroughly can prevent cold mounting resins from adhering to the specimen

surface. Ultrasonic cleaning may be effective in removing the last traces of residues on a specimen surface.

8.2 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 dissolving in cold nitric acid (HNO_3 , sp gr 1.42), in dilute sulfuric acid (H_2SO_4) or in dilute hydrochloric acid (HCl). The HNO_3 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. 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 (polyethylene polyamine) or SbCl_3 are two useful inhibitors.

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

9. Mounting of Specimens

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

9.2 Specimens may be either mechanically mounted, mounted in plastic, or a combination of the two.

9.3 Mechanical Mounting:

9.3.1 Strip and sheet specimens may be mounted by binding or clamping several specimens into a pack held together by two end pieces and two bolts.

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

9.3.3 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.

9.3.4 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.

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

9.3.6 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.

9.3.7 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.

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_2O_3	P or R	hard
up to 500	medium soft ferrous	Al_2O_3	P or R	med. hard
up to 600	medium hard ferrous	Al_2O_3	P or R	medium
up to 700	hard ferrous	Al_2O_3	P or R&R	med. soft
up to 800	very hard ferrous	Al_2O_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

9.3.8 Exercise care in clamping the specimen. Excessive clamping pressure may damage soft specimen.

9.4 Plastic Mounting:

9.4.1 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 and castable.

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

9.4.3 *Compression Mounting*—There 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 (140–180°C) and force (27–30 MPa). Thermosetting plastics can be ejected hot but the best results are obtained when the cured mount is cooled under pressure. Thermoplastic compounds do not harden until cooled and therefore should not be ejected while hot. Regardless of the resin used, the best results are obtained when (1) the specimen is clean and dry, and (2) the cured mount is cooled under full pressure to below 40°C before ejection from the press. This will ensure minimal shrinkage gap formation.

9.4.4 *Castable Plastics*—Castable 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 four kinds of castable plastics in common use (see Table 3).

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

9.5 Mounting Porous Specimen:

9.5.1 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 in a vacuum chamber 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 funnel or stopcock fitted to the vacuum chamber or by having a basin of the resin present inside the chamber. A low-viscosity resin will produce the best results. The pressure in the chamber must remain above the critical vapor pressure of the hardener to avoid boiling away the hardener. After the pressure has equilibrated, the resin is introduced into the mold and the vacuum is released and air admitted to the chamber. Atmospheric pressure will force the resin into fine pores, cracks, and holes.

9.5.2 If a low-viscosity resin is used, the funnel and stopcock may be eliminated. The specimen and resin are placed in the 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. Dipping the specimen in the resin prior to placing it in the mold may help in filling voids.

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

9.5.4 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.

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.

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

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

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 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. Supplies and instructions for grinding, lapping, and polishing are readily obtainable from laboratory supply houses.

11.2 *Grinding*—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 and uniformity.

11.2.1 Abrasive grit size designations in this practice are expressed in the ANSI (American National Standards Institute) or CAMI (Coated Abrasives Manufacturers Institute) system units with the corresponding FEPA (European Federation of Abrasive Producers) numbers in parentheses. Table 4 provides a correlation between these 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 subsequent steps should remove the effects of previous ones in a short time. Grinding consists of two stages- planar (rough) and fine.

11.2.3 Planar or rough grinding [240 grit (P220) and coarser] may be performed on belts, rotating wheels or stones. In some methods, diamond abrasives are used on rigid platens. Planar grinding may be used to accomplish the following:

TABLE 4 European/USA Grit Grade Comparison Guide

FEPA		ANSI/CAMI	
Grit Number	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

11.2.3.1 Flatten an irregular or damaged cut surface,

11.2.3.2 Remove sectioning damage, scale and other surface conditions prior to mounting,

11.2.3.3 Remove substantial amounts of specimen material to reach a desired plane for polishing,

11.2.3.4 Level the mount surface.

11.2.4 In fine grinding, damage to the specimen incurred from the planar or rough grinding step must be removed. The specimen is either ground on successively finer abrasive papers (using water to wash away grinding 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/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. Polishing can be divided into rough and fine (final) stages.

11.3.1 Rough polishing is often sufficient for routine evaluations like microindentation hardness and grain size.

11.3.2 When fine polishing is required, it may be 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 instance, a 1µm diamond final polish is often sufficient for many grades of steel, however,

softer steels and non-ferrous materials often require an additional polishing step with an oxide slurry or suspension of SiO_2 or Al_2O_3 . Final polishing cloths are generally softer and higher in nap than rough polishing cloths. Therefore, polishing time and force must be kept to a minimum to avoid artifacts such as edge rounding and relief.

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

11.3.4 The polishing operations may be conducted by manual or by 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 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.

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

11.7 A traditional manual preparation sequence consists of a series of grinding and 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. Most units can perform grinding and polishing steps. Many use holders capable of accommodating multiple specimens. Major advantages of automated grinding and polishing procedures are the consistent quality of specimen

preparation and the substantial decrease in time. Therefore, automated techniques are recommended over manual techniques.

11.9 Most of 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 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 stages is required to 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 suitable for all but the hardest of materials. Tables 6 and 7 are preparation methods that utilize rigid grinding discs or cloths for fine grinding. The method in Table 6 has been shown to be effective for the preparation of materials harder than HRC45. The method in Table 7 may be used for the preparation of materials softer than HRC45. These procedures may produce excellent results outside of the recommended hardness ranges.

12. Special Procedures

12.1 Occasionally, the metallographer is faced with the preparation of unfamiliar specimens or with special situations. Anticipation of every possible situation is, of course, impossible but some guidance can be offered.

12.1.1 When used properly, electrolytic polishing 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 many materials. Although slow, a number of specimens can be prepared 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 epoxy.

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	6–15µm diamond	180–300	20–30 (5–8)	100–150	CO
Rough Polishing low/no nap cloth	compatible lubricant	3–6µm 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 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	6–15µm diamond	180–300	20–30 (5–8)	100–150	CO
Rough Polishing low/no nap cloth	compatible lubricant	3–6µm 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.

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 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 epoxy for impregnation depends on the nature of the specimen. It should be inert toward the specimen.

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 artifacts such as scratches or rounded edges.

12.4 Coated Materials:

12.4.1 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).

12.4.1.1 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.

12.4.1.2 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.

12.4.1.3 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.

12.5 Fragile specimens should be mounted in one of the castable mounting formulations. Vacuum impregnation will

ensure filling of holes and cavities (see 9.5). Thin walls can be reinforced by electroless nickel plating, which will alleviate the rounding problem.

12.6 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.

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 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, several approaches 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 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.

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

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

⁵ Endox 214 is available from Enthone, Inc., 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.

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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.

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.

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