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## Standard Guide for Preparation of Metallographic Specimens<sup>1</sup>

This standard is issued under the fixed designation E3; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

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~~<sup>e1</sup>Note—Section 13, Precision and Bias was editorially removed from the standard in March 2009.~~

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### 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:

- A90/A90M Test Method for Weight [Mass] of Coating on Iron and Steel Articles with Zinc or Zinc-Alloy Coatings<sup>2</sup>
- E7 Terminology Relating to Metallography
- E45 Test Methods for Determining the Inclusion Content of Steel
- ~~E340 Test Method for Macroetching Metals and Alloys~~
- ~~E407 Practice for Microetching Metals and Alloys~~
- E768 Guide for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel
- E1077 Test Methods for Estimating the Depth of Decarburization of Steel Specimens
- E1122 Practice for Obtaining JK Inclusion Ratings Using Automatic Image Analysis
- E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis
- E1268 Practice for Assessing the Degree of Banding or Orientation of Microstructures
- E1558 Guide for Electrolytic Polishing of Metallographic Specimens
- E1920 Guide for Metallographic Preparation of Thermal Sprayed Coatings

### 3. Terminology

#### 3.1 Definitions:

3.1.1 For definitions used in this practice, refer to Terminology E7.

#### 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

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<sup>2</sup> "Manual Electron Metallography Techniques," 1973. Available from ASTM Headquarters. Request STP 547.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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 15 $\mu$ m 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

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 E1077),
- 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. See Guide E1920.

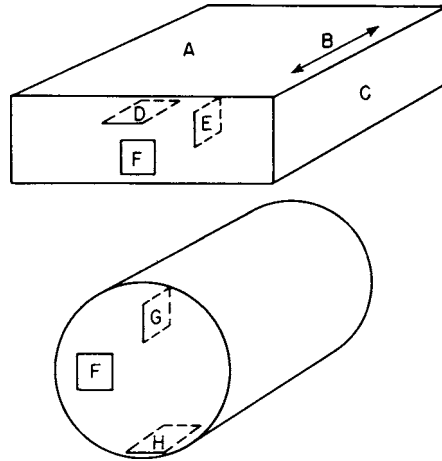
5.4 Longitudinal sections taken parallel to the main axis of the material are often used for revealing the following information:

- 5.4.1 Inclusion content of steel (see Practices E45, E768, E1122, and E1245),
- 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 E1268), 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



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

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

(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 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.

**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 <sub>2</sub> O <sub>3</sub>	P or R	hard
up to 500	medium soft ferrous	Al <sub>2</sub> O <sub>3</sub>	P or R	med. hard
up to 600	medium hard ferrous	Al <sub>2</sub> O <sub>3</sub>	P or R	medium
up to 700	hard ferrous	Al <sub>2</sub> O <sub>3</sub>	P or R&R	med. soft
up to 800	very hard ferrous	Al <sub>2</sub> O <sub>3</sub>	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

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<sub>3</sub>, sp gr 1.42), in dilute sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) or in dilute hydrochloric acid (HCl). The HNO<sub>3</sub> 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 A90/A90M.

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 (polylethylene polyamine) or SbCl<sub>3</sub> 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.

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.

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.

**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 <sup>A</sup>	thermosetting, cure time 5-10 min, opaque, minimal shrinkage, good resistance to etchants, moderate abrasion resistance
Epoxy <sup>A</sup>	thermosetting, cure time 5-10 min, opaque, very low shrinkage, good resistance to etchants, high abrasion resistance
Phenolic <sup>A</sup> (Bakelite)	thermosetting, cure time 5-10 min, opaque, moderate shrinkage, degraded by hot etchants, moderate abrasion resistance

<sup>A</sup> 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

## 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:

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.

**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 <sup>A</sup>	5.0	...	...

<sup>A</sup> Not found in the FEPA grading system.

ANSI—American National Standards Institute

CAMI—Coated Abrasives Manufacturers Institute

FEPA—European Federation of Abrasive Producers