



Standard Practice 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 light 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 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.

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:

- 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 1077 Test Method for Estimating the Depth of Decarburization of Steel Specimens²
- E 1268 Practice for Assessing the Degree of Banding or Orientation of Microstructures²
- E 1558 Guide to Electrolytic Polishing of Metallographic Specimens²

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

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

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

3. Significance and Use

3.1 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.

3.2 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.

3.3 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.

4. Selection of Metallographic Specimens

4.1 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:

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

4.1.2 *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. Specimens should be taken in many cases from a sound area for a comparison of structures and properties.

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

4.2 Having established the location of the metallographic samples to be studied, the type of section to be examined must

be decided. 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. 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. 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 sections or transverse sections taken perpendicular to the main axis of the material are more suitable for revealing the following information:

- 4.3.1 Variations in structure from center to surface,
- 4.3.2 Distribution of nonmetallic impurities across the section,
- 4.3.3 Decarburization at the surface of a ferrous material (see Test Method E 1077),
- 4.3.4 Depth of surface imperfections,
- 4.3.5 Depth of corrosion,
- 4.3.6 Thickness of protective coatings, and
- 4.3.7 Structure of protective coating.

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

- 4.4.1 Inclusion content of steel (see Practice E 45),
- 4.4.2 Degree of plastic deformation, as shown by grain distortion,
- 4.4.3 Presence or absence of banding in the structure (see Practice E 1268), and
- 4.4.4 The quality attained with any heat treatment.

4.5 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.

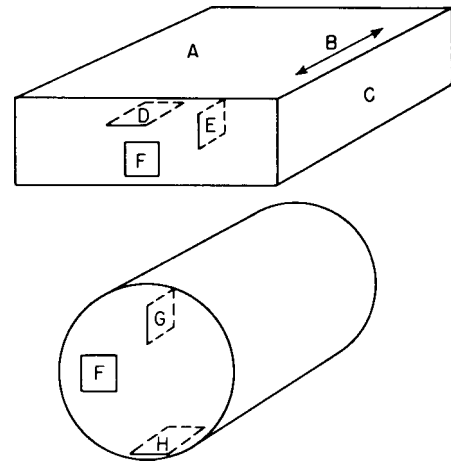
5.2 It is not always possible to secure specimens having the dimensions given in 5.1, when the material to be examined is smaller than the ideal dimensions. For example, in the polishing of wire, strip, and other small articles, it is necessary to mount the specimens because of their size and shape.

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 of Metallographic Specimens

6.1 In cutting the metallographic specimen from the main



| 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 | 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.

body of the material, care must be exercised to minimize altering the structure of the metal. Three common types of sectioning are as follows:

6.1.1 Sawing, whether by hand 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 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 is normally faster than sawing. The choice 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 for ferrous metals and silicon carbide wheels 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 more effectively cut using diamond-impregnated cutting wheels. Manufacturer's instructions should be followed as to the choice of wheel and speeds.

6.1.3 Flame cutting completely alters the structure of the metal at the flame cut edge. If flame cutting is necessary to remove the specimen, it should be cut sufficiently large so that it can be recut 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 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.

7. Cleanliness

7.1 Cleanliness (see Appendix X1.) during specimen preparation is essential. All greases and oils 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 effective in removing the last traces of residues on a specimen surface.

7.2 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. The coating can be removed by digestion 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.

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

8. Mounting of Specimens

8.1 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, flimsy, or oddly shaped specimens, fractures, or in instances where the specimen edges are to be examined.

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

8.3 Mechanical Mounting:

8.3.1 Strip and sheet specimens are frequently 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 specimens should be tightly bound together to prevent absorption and subsequent exudation of polishing materials or etchants.

8.3.3 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.

8.3.4 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.

8.3.5 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.

8.3.6 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.

8.3.7 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.

8.3.8 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 Plastic Mounting:

8.4.1 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 mounting specimens in plastic, exercise care in order to avoid rounding of specimen edges during the grinding operation. There are several methods available that prevent rounding. The specimens may be surrounded by hard shot, small rivets, rings, etc., of approximately the same hardness or, when using casting resin, a slurry of resin and alumina may be poured around the specimen to prevent rounding. The specimens may also be plated before mounting (see Section 9).

8.4.3 *Compression Mounting*—Thermosetting plastics require the use of a mounting press providing heat (up to approximately 160°C) and pressure (up to approximately 30 MPa). *The finished mounts can be ejected hot but the best results are obtained when the finished mount is cooled under pressure.* There are three types of thermosetting compression mounting plastics used predominantly in the metallographic laboratory. Regardless of the resin used to compression mount specimens, the best results are obtained when (1) the specimens are clean and dry, and (2) the cured mount is cooled under full pressure to below 30°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 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 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 *Castable Plastics*—Castable resins are used 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 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 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 molds for castable plastics are 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.

8.5 *Mounting Porous Specimen:*

8.5.1 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 a vacuum chamber fitted with a funnel and a stopcock, or a similar commercially available evacuation device, so that the resin can be poured into the mold from outside. A low-viscosity resin will produce the best results but ordinary metallographic resins will work well. The vacuum chamber is then evacuated. The pressure in the chamber must remain above the critical vapor pressure of the hardener to avoid evaporating 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. Very porous specimens

may be turned using 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 resin is placed in the cup 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 the specimen over to ensure impregnation of the bottom side. Remember to turn the specimen back over again before the resin starts to set.

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

8.5.4 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.