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Standard Guide for Preparation of Metallographic Specimens[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. 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 prep

NOTE 1—For a more exter ... techniques, refer to Samuels, L ... cal Methods, American Socie ... 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.*

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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[2]
E7 Terminology Relating to Metallography
E45 Test Methods for Determining the Inclusion Content of Steel
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 (Withdrawn 2006)[3]
E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis
... Degree of Banding or
... shing of Metallographic
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 resin and the other hardener. Both components can he 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

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

[3] The last approved version of this historical standard is referenced on www.astm.org.

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.

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
tation is to be of value, th
the material that is being
metallographic examinati
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,

ding in the structure (see
with any heat treatment.
mined 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.

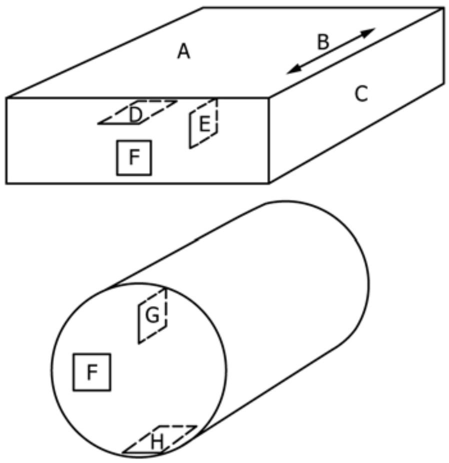
6.1.1 Larger specimens are generally more difficult to prepare.

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 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



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

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

a rough surface containin

removed in subsequent p

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

emoved by dissolving in

), in dilute sulfuric acid

acid (HCl). The HNO_3

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 (polethylinepolyamine) 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.