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An American National Standard

Standard Practice for
Preparation of Metallographic Specimens[1]

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 Vander-Voort, 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 Metallography[2]

E 45 Practice for Determining the Inclusion Content of Steel[2]

E 340 Test Method for Macroetching Metals and Alloys[2]

E 407 Test Methods for Microetching Metals and Alloys[2]

E 1077 Test Method for Estimating the Depth of Decarburization of Steel Specimens[2]

E 1268 Practice for Assessing the Degree of Banding or Orientation of Microstructures[2]

E 1558 Guide to Electrolytic Polishing of Metallographic Specimens[2]

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

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[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.

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

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).

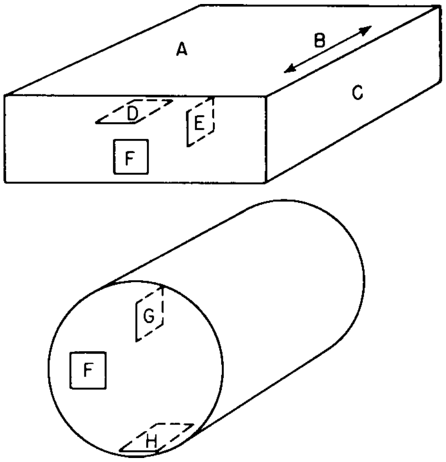
6. Cutting of Metallographic Specimens

6.1 In cutting the metallographic specimen from the main 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



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.

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.

10. Grinding and Polishing

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

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

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

10.2.1.1 Flatten an irregular or damaged cut surface,

10.2.1.2 Remove scale and other surface conditions prior to mounting,

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

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

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

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

10.2.2.1 Grinding can be done in a number of ways, ranging from rubbing the specimen on a stationary piece of abrasive paper to the use of automatic devices. The choice of method depends on the number of specimens to be done, financial considerations, and requirements such as flatness, uniformity, and so forth. Grinding on abrasive-coated rotating disks using hand-held specimens is the traditional method. Mechanical devices to hold the specimen against abrasive-covered rotating disks are becoming increasingly common. Many of these machines permit automated grinding, providing surfaces superior to hand-held specimens.

10.2.2.2 Grinding should start with the finest paper capable of flattening the specimen and removing the effects of prior operations, such as sectioning. The next paper should remove the effects of the prior paper in a short time. A typical sequence of papers might be 240, 320, 400, and 600-grit abrasive papers. Depending on the smoothness of the specimen surface, some of the coarser paper grades can be skipped.

10.2.2.3 In the hand-held methods, the specimen is rotated 90° between papers to determine when the prior set of scratches have been removed. The specimen should also be moved back and forth across the paper to prevent grooving of the specimen. At the end of the grinding on each paper, the surface of the specimen and its mount, if any, should be flat with one set of unidirectional grinding scratches.

10.2.2.4 Most of the devices for automatic grinding 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 scratch pattern now consists of random arcs. Deciding when the previous scratches have been removed is more difficult than with directional grinding. The specimen surface should show uniform scratches before proceeding to the next step. Cleaning between stages may be necessary to prevent carryover of abrasives and contamination of grinding surfaces. Manufacturer's instructions will include suggested machine settings for grinding various metals.

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

10.3 *Polishing*—Polishing is usually distinguished from grinding by the use of loose abrasive embedded in an appropriately lubricated supporting surface. The choice of abrasive, lubricant, and polishing surface support is often specific to the metal and the object of the investigation.

10.3.1 The use of graded diamond paste or suspensions as the abrasive can reduce the number of combinations markedly. Those supports most commonly used are non-napped cloths such as nylon or nonwoven textiles available for metallographic purposes.

10.3.2 The final polish may be 1-µm diamond abrasive. For high-resolution work, this diamond polish may be followed by polishing on a short nap synthetic suede using an aqueous suspension of 0.05-µm gamma alumina or colloidal silica. Other abrasives and supports often are required depending on the particular task in hand. This step should be kept to the shortest time possible to prevent edge rounding, pitting, or other artifacts. Twenty to forty seconds should be sufficient if the previous steps have been correctly performed.

10.3.3 Careful cleaning of the specimen between stages is mandatory to prevent contamination by coarser abrasive. Ultrasonic cleaning is recommended between each polishing step.

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

10.3.4.1 Hand methods consist of holding the specimen by hand against an abrasive-charged rotating wheel and moving the specimen in an elliptical path around the wheel against the direction of rotation of the wheel. The specimen should be held firmly in contact with the wheel. Just how firm and just how fast to go around the wheel is a matter of experience and personal preference. In the preparation of advanced materials, the operating parameters must be strictly controlled.

10.4 *Automatic Grinding and Polishing*:

10.4.1 Many styles of automatic specimen preparation machinery are available. The most common units can perform all grinding and polishing steps. They use disk fixtures accommodating multiple specimens; the specimens remain in the fixture throughout the preparation. Major advantages in using automatic grinding and polishing procedures are the consistent quality of specimen preparation and the substantial decrease in time required for preparing large number of specimens. Careful attention to cleanliness and prevention of cross-contamination from abrasives and residues between steps, particularly in polishing, is required. Ultrasonic cleaning is recommended.

10.4.2 An initial coarse-grinding step is required to make all specimen surfaces co-planar and parallel to the fixture. This step is usually used to remove damage to the specimens from sectioning operations. Fine grinding uses either a graded abrasive paper series or a single step with an intermediate (9 or 6 µm) diamond abrasive on a permanent, grooved wheel to prepare the specimens for polishing. As an alternative, lapping platens may be used to replace the finegrinding paper sequence. This approach offers the greatest benefits when very hard or highly dissimilar materials must be prepared.

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

11. Special Procedures

11.1 Occasionally, every metallographer is faced with the preparation of unusual specimens or with special situations. Anticipation of every possible situation is, of course, impossible but some guidance can be offered. Common sense is imperative.

11.1.1 Electrolytic polishing produces totally 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.

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

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

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

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

11.4 *Coated Materials*:

11.4.1 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).

11.4.2 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.

11.4.3 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. 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.

11.4.4 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.

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

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

12. Precision and Bias

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

13. Keywords

13.1 alloys; grinding; metallography; metals; mounting; polishing; specimen preparation (metallographic)

APPENDIX

(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 547[3] 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, there are several approaches that can be used. Always try the simplest, safest methods first. For example, use a blast of compressed air to remove any loosely adherent material. A soft camel-hair brush or a soft toothbrush may also be useful for removing loosely adherent matter.

X1.3.1 If the techniques in X1.3 do not suffice, try aqueous solutions, organic solvents, or alcohol with an ultrasonic cleaner. Aqueous solutions (8 g of Alconox per litre of warm water) containing Alconox[4], a detergent, have been found **(1, 2)** to be effective. Follow the Alconox bath with washing 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 214[5] has been found **(1)** to be useful for cleaning heavily rusted steel fractures.

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

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

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.

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[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., 350 Frontage Rd., West Haven, CT 06516. An equivalent can be used.

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

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

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

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

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

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

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

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

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

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

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

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

REFERENCES

(**1**) Yuzawich, P. M., and Hughes, C. W.,“ An Improved Technique for Removal of Oxide Scale from Fractured Surfaces of Ferrous Materials,” *Practical Metallography*, Vol 15, April 1978, pp. 184–195.

(**2**) Zipp, R. D., “Preservation and Cleaning of Fracture for Fractography,” *Scanning Electron Microscopy*, Part I, 1979, pp. 355–362.

(**3**) Russ, J. C., and Miller, G. A., “Effect of Oxidation on the Electron Fractographic Interpretation of Fractures in Steel,” *Journal, Iron and Steel Institute*, Vol 207, December 1969, pp. 1635–1638.

(**4**) Pickwick, K. M., and Smith, E., “The Effect of Surface Contamination in SEM Fractographic Investigations,” *Micron*, Vol 3, No. 2, 1972, pp. 224–237.

(**5**) Lane, G. S., and Ellis, J., “The Examination of Corroded Fracture Surfaces in the Scanning Electron Microscope,” *Corrosion Science*, Vol 11, September 1971, pp. 661–663.

(**6**) Macmillan, J. W., and Flewitt, P. E. J., “Assessment of Methods for Cleaning Oxide from Fracture Surfaces for Examination in a Scanning Electron Microscope,” *Micron*, Vol 6, No. 3/4, 1975, pp. 141–146.

(**7**) ASTM Task Group E24.02, “Clean Surfaces for Fractography,” ASTM STP 600, ASTM, Philadelphia, PA, 1976, pp. 251–253.

(**8**) Hajicek, D. J., et al, “Development of a Cleaning Method for SEM Fractographic Analysis of Impulsively Loaded Cylinders,” *Microstructural Science*, Vol 5, Elsevier, NY, 1977, pp. 413–421.