

Designation: E2015 - 04 (Reapproved 2021)

Standard Guide for Preparation of Plastics and Polymeric Specimens for Microstructural Examination¹

This standard is issued under the fixed designation E2015; 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.

1. Scope

1.1 This guide covers recommended procedures and guidelines for the preparation of plastic and polymeric specimens for microstructural examination by light and electron microscopy.

1.2 This guide is applicable to most semi-rigid and rigid plastics, including engineering plastics. This guide is also applicable to some non-rigid plastics.

1.3 The procedures and guidelines presented in this guide are those which generally produce satisfactory specimens. This guide does not describe the variations in techniques required to solve individual problems.

1.4 Many detailed descriptions of grinding and polishing of plastics and polymers are available (1-7).²

1.5 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.6 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:³
D883 Terminology Relating to Plastics
E3 Guide for Preparation of Metallographic Specimens
E7 Terminology Relating to Metallography

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions used in this guide of terms directly related to metallography, refer to Terminology E7.

3.1.2 For definitions used in this guide of terms directly related to plastics and polymers, refer to Engineering Materials Handbook, Vol 2 (8) and Terminology D883.

3.1.3 *plastic(s)*—a material that contains as an essential ingredient one or more organic polymeric substances of large molecular weight; is solid in its finished state; and at some stage in its manufacture or processing into finished articles, can be shaped by flow.

3.1.4 polymer(s)—a substance consisting of molecules characterized by the repetition (neglecting ends, branch junctions, and other minor irregularities) of one or more types of monomeric units.

4. Significance and Use

4.1 One of the fundamental objectives of microstructural examination of manufactured materials, especially plastics and polymers, is to gain a more complete understanding of the relationships between the manufacturing processes, the microstructure and texture of the material, and the product's performance (that is, physical, optical, or mechanical properties, or combination thereof). Under nearly all conditions, the proper selection and preparation of the specimen are of major importance.

4.2 Because of the wide range of available equipment; physical, chemical, and mechanical properties of materials; and the personal element, specimen preparation is an art based upon scientific principles. However, like metallographic specimen preparation, certain methods, practices, and procedures can be used to routinely produce acceptable quality plastic and polymeric specimens for microstructural examination. Acceptable quality means:

4.2.1 The observed microstructure is free of thermal, mechanical, and chemical alterations, artifacts, damage, or defects resulting from the specimen preparation process.

4.2.2 A surface finish appropriate for the microscopical techniques to be used.

4.2.3 The microstructure is reproducibly displayed for a given specimen.

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² The boldface numbers in parentheses refer to the list of references at the end of this standard.

³ 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.

4.3 The mounting, sectioning, grinding, and polishing procedures in this guide may introduce thermal, mechanical, and chemical stresses on the material being prepared for microstructural examination. Thus, knowledge of the material's physical, mechanical, and chemical properties is of importance in selecting the most appropriate technique(s) to reveal its true microstructure and to minimize the total number of steps needed to produce high quality polished specimens.

4.4 The general guidelines presented below will need to be modified for each type of plastic or polymer to be prepared. Table X1.1 presents general procedures for preparing plastics and polymers. Tables X1.2-X1.5 present procedures for preparing four polymers with very different mechanical properties.

5. Selection of Specimens

5.1 The selection of test specimens is extremely important and dependent upon the purpose of the examination, the material, and the microscopical technique to be used. The principles of specimen selection presented in Practice E3 should be used as a primary guide for the selection of a plastic or polymeric test specimen.

5.2 The selection criteria must include the following considerations:

5.2.1 The size or scale of homogeneity/heterogeneity of all structures, textures, and other features within the material being studied;

5.2.2 The size or scale and distribution of the structures to be studied;

5.2.3 The microscopical technique(s) to be used; and

5.2.4 The need for control/reference specimens.

5.3 Once the specimen locations have been selected, these locations should be well documented. Macrographs or micrographs, or both, of the specimen locations along with brief specimen location descriptions accompanying the written results are usually sufficient.

6. Size of Specimens

6.1 The grinding and polishing procedures presented in this guide require the use of automated grinding and polishing equipment. Therefore, the specimen size will be limited by the holders available for the equipment to be used.

7. Cleaning of Specimens

7.1 Most plastics and polymers are very soft and subject to abrasion from debris produced during sectioning, grinding, and polishing. In addition, oils and other surface films inhibit uniform coating and adhesion of the mounting resin to the specimen surface. Therefore, it is essential that the specimen and all specimen preparation surfaces be kept as clean as possible. Thorough cleaning after each grinding and polishing step will minimize contamination from the carry-over of coarser abrasives and debris that may cause damage during the next preparation step.

7.2 The least aggressive solution, which effectively cleans the specimen surface, should be used. This requires knowledge of the specimen's reactivity in potential cleaning solutions. For many plastic and polymeric materials, cleaning with an aqueous solution of dish soap is very effective. However, some plastics and polymers are subject to physical and chemical changes when placed in contact with aqueous solutions.

7.3 The use of ultrasonic baths to promote cleaning is usually an acceptable practice. However, materials such as partially cured resins may be damaged by excessive cavitation in ultrasonic cleaning.

8. Preliminary Sectioning and Mounting of Specimens

8.1 Contrary to traditional metallographic procedures, small specimens or parts, or both, with the plane of interest not parallel to a flat surface may require mounting prior to sectioning to facilitate sectioning of the specimen parallel to the desired plane to be polished. Also, laminated, friable, or very ductile materials may be mounted prior to section to minimize damage during sectioning.

8.2 In general, specimens should be mounted for sectioning, grinding, and polishing. Mounted specimens are typically easier to handle and less susceptible to damage. Specimens are usually mounted in castable resins but may also be mechanically mounted. For very soft, flexible materials, it is often necessary to use a combination of mechanical mounting and mounting in a castable resin. Compression mounting in thermoplastic or thermosetting plastic is generally not recommended but may be suitable for high temperature engineering plastics.

8.3 Preliminary sectioning may be necessary prior to mounting. This is usually accomplished by cutting or sawing of the unmounted part (see Section 9). These cuts should be made sufficiently far from the area of interest to minimize damage due to sectioning yet close enough to minimize the next material removal step.

8.4 The pre-sectioned specimen must be thoroughly cleaned and dried to remove any debris and oils from the suface that might inhibit the wetting and adhesion of the mounting medium to the specimen surface.

8.5 In many cases, there may be some reactivity between the mounting medium and the specimen. Coating the specimen with a 20 nm to 60 nm thick metal film of gold or gold/ palladium provides an excellent barrier between the mounting medium and the specimen. This metal coating also acts as an interface that will improve the adhesion of the mounting medium to the specimen. The sputter coaters and vapor deposition coaters used to prepare conductive coatings for electron microscopy specimens work very well for this application. In some cases, electroless plating can be used to produce metal coatings on the plastics and polymers.

8.6 Room temperature-cured, castable resins are generally used to encapsulate plastic and polymeric specimens.

8.6.1 It is critical that the manufacturer's recommended mixing proportions be followed precisely and that mixing of the components be thorough so that uniform and reproducible results will be achieved.

8.6.2 Molds for castable resins can be easily produced in the laboratory and a wide variety of shapes and compositions are

available from various manufacturers. The molds may be reusable or not; the choice is a matter of convenience and cost. Handling of these resins requires care. They all can cause dermatitis as well as other problems.

8.6.3 Styrene, latex, or other plastic spheres or particles can be mixed into the mounting resin to modify the mechanical properties of the cured resin to more closely match those of the specimen.

8.6.4 Many plastics and polymers tend to float in the mounting resins. Floating can be inhibited by placing a phenolic or other ringform on adhesive tape or by placing double-sided adhesive tape on the interior bottom of the mold, then attaching the specimen to the adhesive inside the ringform or mold and covering it with the mounting resin. Floating can also be inhibited by partially surrounding the specimen with the mounting resin and allowing the resin to partially cure, then repeating this step one or more times until the specimen is completely encased in mounting resin.

8.6.5 Many plastic and polymeric materials may be damaged by the heat produced during curing of castable resins. This can be minimized or eliminated by using the smallest volume of resin necessary to encapsulate the specimen and by placing the mounted specimen in a refrigerator or ice bath while the resin cures.

8.7 Vacuum impregnation is a recommended method for ensuring high quality mounts.

8.8 The contrast between the specimen and castable mounting resin is often quite poor, making it difficult to identify edges or study edge structures. A thick (>100 nm) metal coating (see section 8.5) will help improve the contrast at the specimen-resin interface. Another approach is to charge the resin with a colorant or fluorescent dye, such as fluorescein.

9. Cutting or Sectioning of Specimens

9.1 In general, sectioning should produce a flat, relatively damage-free surface very near to the region of interest.

9.1.1 Cutting with a sharp blade, scalpel, knife, or scissors is one of the fastest and most common methods for sectioning plastic and polymer films, tubing, and thin flexible parts. This technique will introduce a strain (typically dominated by ductile deformation) in the region near the cut face. The width of the strain region can be minimized by properly securing the sample during cutting, using a sharp instrument, making the cut with uniform speed and force, and making the cut at the appropriate temperature (often below room temperature). The cut face from a (cryogenically)microtomed specimen is often ready for microstructural examination with minimal final polishing or without additional preparation.

9.1.2 Sawing either manually or by machine is generally a convenient method for sectioning rigid plastics. Sawing produces a rather rough surface with a region of non-uniform strain that is generally wider than that produced by cutting. The deformation is often easily removed by the subsequent grinding and polishing steps. The width of the deformation region can be minimized by choosing a sharp, fine, short-toothed blade; a feed rate equal to the material removal rate; a coolant/lubricant that is non-reactive with the specimen; and a blade speed that does not cause a significant temperature rise in

the specimen and by presenting a minimum cross-sectional area of the part to the saw blade.

9.1.3 Cutting or sectioning may also be accomplished by the use of an abrasive cut-off wheel. This technique generally produces a cut surface with deformation that can be removed by fine grinding and polishing. Abrasive wheels with 80 grit to 120 grit abrasive cut soft epoxies quickly but leave a rough finish, often with a relatively thick layer of ductile deformation. Finer grit (240 and above) abrasive wheels cut soft epoxies quite slowly, tend to be quickly clogged with plastic or polymer, and tend to wander. The force or load should be sufficient to ensure a cutting or feed rate that is equal to the removal rate. The blade speed should provide high removal rate without causing a significant temperature rise in the specimen. A non-reactive coolant/lubricant, which contains a surfactant, will allow for high blade speeds, faster cutting, and minimal damage. The effectiveness of abrasive cut-off wheels can be greatly improved by rotating the specimen about an axis that is parallel to the axis of rotation of the cut-off wheel.

9.2 For machine assisted cutting or sectioning, it is always advisable to orient the specimen so that the blade, cutting tool, or abrasive wheel moves from the weakest or least supported to strongest or best supported portion of the specimen while presenting the smallest cross-sectional area to the cutting tool.

9.3 Carefully inspect the cleaned, cut face of mounted porous specimens. If the cut face exhibits open pores, reimpregnate the surface with a small amount of the mounting resin.

10. Grinding

10.1 The principles of grinding and polishing presented in Practice E3 should be used for plastics and polymers to produce a flat polished surface that allows the true microstructure of the specimen to be examined. In general, grinding is used to remove material in order to expose the region of interest while producing a flat surface and removing the deformation caused by the preceding sectioning and mounting steps.

10.2 Hand polishing may be used in some instances for rigid engineering plastics. Automated polishing systems with specimen holders that hold the specimen against rotating disks permit automated grinding and polishing to yield surfaces that are superior to hand polished specimen surfaces.

10.3 The mounted specimen should be examined frequently during grinding to ensure that material removal does not go beyond the region of interest.

10.4 Grinding is often separated into two steps: rough and fine. However, rough grinding (240[P220] grit and coarser) is usually too aggressive for plastics and polymers.

10.4.1 Typically a single grinding step using 15 μ m fixed abrasive, such as 600(P1200)-grit SiC abrasive papers, is sufficient to produce flat surfaces ready for polishing.

10.4.2 With proper sectioning and mounting, only moderate pressures and short times are needed to complete the grinding. Pressures from 13 800 N/m² to 34 500 N/m² (2 psi to 5 psi) and effective wheel (12 in. diameter) speeds from 90 rpm to 150 rpm are common.

10.4.3 The specimen surface should be inspected after every 15 s to 30 s of grinding to monitor material removal and specimen surface flatness.

10.4.4 Water is most commonly used as a coolant/lubricant for grinding. Some plastics and polymers may react with water. For these materials, a knowledge of the reactivity in other fluids is necessary to choose the most appropriate coolant/ lubricant for grinding and polishing. Perfluorinated liquids, such as those used as diffusion pump oil or as cooling liquids for active electronic circuits, although expensive, are often appropriate for use with water soluble plastics and polymers. Oil based solutions may also be used and are relatively low-cost but require special care for handling, cleaning, and disposal.

10.4.5 Specimens must be thoroughly cleaned after each grinding step to prevent carryover of debris and abrasive grit, which may cause damage during the next step of the preparation.

11. Polishing

11.1 Polishing removes the damage produced during grinding and is used to expose the true microstructure of the specimen with minimal material removal. In metallography, this is generally accomplished by using free abrasive carried on, or partially embedded in, a lubricated supporting surface.

11.2 Care must be taken when using free abrasives not to embed the abrasive in the specimen, which can easily occur with softer plastics and polymers. The use of a lubricant/ coolant, which contains a surfactant or wetting agent, can significantly improve the polishing characteristics of a free abrasive and minimize embedding of the abrasive. Likewise, the use of perforated non-napped surfaces that are somewhat softer than the specimen will help to minimize abrasive embedding as well as edge rounding. Fixed, micro-abrasive, lapping films are an excellent alternative to free abrasives and can essentially eliminate the problem of embedded abrasive.

11.3 To ensure uniform and reproducible results, especially for automated polishing, the base surfaces on which polishing cloths and lapping films are supported or the lapping platen surfaces must be flat and clean.

11.4 Polishing, like grinding, is often separated into rough polishing and fine or final polishing. The total number of polishing steps is dependent on the material properties of the mounted specimen, previous preparation steps, the time available for specimen preparation, and the objective of the examination.

11.5 The purpose of rough polishing is to remove all damage produced by the previous preparation steps while maintaining flatness of the surface and to prepare a surface suitable for viewing by incident bright field microscopy at 100X to 500X magnification.

11.5.1 Six or nine micrometer abrasive is often an appropriate starting point for the first step in rough polishing. This abrasive size provides very good removal rates with minimal additional damage to the specimen. Excessive loading of the polishing surface with abrasive can induce embedding of the abrasive in the specimen.

11.5.2 If lapping surfaces or polishing cloths that are harder than the specimen are used, the applied pressure to the specimen should be about 13 800 N/m² to 27 600 N/m² (2 psi to 4 psi). If lapping surfaces or polishing cloths that are softer than the specimen are used, then the applied pressure to the specimen can be increased to approximately 34 500 N/m²to 48 300 N/m² (5 psi to 7 psi).

11.5.3 For automated polishing, effective wheel (305-mm [12-in.] diameter) speeds from 120 rpm to 200 rpm can be used. With these speeds, polishing times are nominally 2 min. to 4 min. Rough polishing times should not exceed 2 min before re-inspecting the specimen surface finish.

11.5.4 If, after the first rough polish step, only a few fine scratches are visible, then proceeding to the fine polish is appropriate. If numerous scratches are visible, then repeat the rough polish, using either 3 or 1 μ m abrasive.

11.6 The purpose of fine polishing is to remove all scratches, expose the true microstructure of the specimen, and prepare a surface suitable for viewing by incident bright field microscopy at magnifications greater than 500X. Fine polishing typically requires only one step.

11.6.1 Fine polishing typically uses 0.05 μ m alumina dispersed in distilled water. However, alumina, colloidal silica, iron oxide, and other materials dispersed in solutions that chemically assist polishing can also be effectively used as a final polish. This requires a knowledge of the chemical interaction between the carrier liquid and the specimen.

11.6.2 A soft, short-nap cloth works well for the final polish but will cause edge rounding and relief. The extent of relief is directly related to the specimen load and the polishing time.

11.6.3 The specimen load for the final polish should be about 13 800 N/m² to 27 600 N/m² (2 psi to 4 psi).

11.6.4 For automated polishing, effective wheel (305-mm [12-in.] diameter) speeds from 120 rpm to 200 rpm can be used. With these speeds, polishing times are nominally 2 min. to 4 min. Final polishing times should not exceed 1 min before re-inspecting the specimen surface finish to ensure minimal relief and edge rounding.

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 grinding; microstructure; mounting; plastics; polishing; polymers; specimen preparation

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APPENDIX

(Nonmandatory Information)

X1. PREPARING PLASTICS AND POLYMERS

X1.1 See Table X1.1 for general procedures for preparing plastics and polymers.

X1.2 See Tables X1.2-X1.5 for procedures for preparing four polymers with very different mechanical properties.

TABLE X1.1 General Procedure for Automatic Grinding and Polishing of Plastic and Polymeric Specimens

NOTE 1-This procedure assumes a properly mounted specimen:

Note 2—This procedure uses two grinding steps. Depending on the material and the mounting of the specimen, only one grinding step may be necessary. Likewise, two rough polishing steps are shown. A jump from 600-grit fine grinding to 6-µm diamond polish is often sufficient to prepare the surface for final polishing with colloidal silica or 0.05-µm alumina.



low nap water colloidal 30, repeat 9-18(2-4) 80 Contra cloth silica as needed

^A American National Standards Institute (ANSI) designation of grit size.

^B Federation of European Producers of Abrasives (FEPA) designation of grit size.

^C Force per 32 mm (1.25 in.) diameter specimen.

^D RPM for a nominal wheel diameter of 305 mm (12 in.).

^E Complimentary rotation—The head rotation is in the same direction as the wheel. Contra rotation—The head rotation is in the opposite direction as the wheel.

^F There are many aqueous solutions with surfactants and additives to reduce surface tension that can be used in place of lapping oil.

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TABLE X1.2 Procedure for Automatic Grinding and Polishing of Urethanes

NOTE 1—Sectioning:

scissors, jewelers saw, or razor

Cleaning (after each step):

wash with mild detergent in an ultrasonic bath,

rinse with distilled water,

final rinse with ethyl alcohol, and

dry with compressed air or by streaming hot air over the specimen

Mounting:

sputter coat with 40 nm of gold, and

encapsulate in a moderately soft epoxy (70 to 75 Shore D hardness) under vacuum cure at room temperature for 24 h

SURFACE	COOLANT/ LUBRICANT	ABRASIVE SIZE/TYPE ANSI ^A (FEPA) ^B	TIME -seconds	FORCE ^C N (lbs)	SURFACE SPEED in rpm ^D	RELATIVE ROTATION		
			Planar Grinding					
SiC or Al ₂ O ₃ paper	water	400(P600) grit	20-30, repeat as needed	18-27(4-6)	200	Complimentary ^E		
			Fine Grinding					
PSA backed lapping film on rigid substrate	lapping oil ^F	15-μm diamond	30, repeat as needed	9-18(2-4)	120	Complimentary		
			Rough Polishing					
PSA backed lapping film on rigid	lapping oil	6-µm diamond	30, repeat as needed	9-18(2-4)	120	Complimentary		
substrate	(https://standards.iteh.ai) Final Polishing							
Low nap cloth	distilled water	0.05-µm gamma Al ₂ O ₃	30, repeat as needed	9-18(2-4)	180	Contra		

^A American National Standards Institute (ANSI) designation of grit size.

^B Federation of European Producers of Abrasives (FEPA) designation of grit size.

^C Force per 32 mm (1.25 in.) diameter specimen.

^{*p*} RPM for a nominal wheel diameter of 305 mm (12 in.). ^{*c*} Complimentary rotation—The head rotation is in the same direction as the wheel. Contra rotation—The head rotation is in the opposite direction as the wheel. F There are many aqueous solutions with surfactants and additives to reduce surface tension that can be used in place of lapping oil.

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TABLE X1.3 Procedure for Automatic Grinding and Polishing of PMMA and Polycarbonates

NOTE 1—Sectioning: high speed abrasive cut-off saw Cleaning (after each step): wash with mild detergent in an ultrasonic bath, rinse with distilled water, final rinse with ethyl alcohol, isopropyl alcohol, or heptane, and dry with compressed air or by streaming hot air over the specimen Mounting:

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sputter coat with 40 nm of gold, and

encapsulate in acrylic or hard epoxy (80 Shore D hardness or greater) under vacuum cure acrylic at less than room temperature; cure epoxy at room temperature for 24 h

NOTE 2-Hard setting epoxy resins are recommended for mounting PMMA since PMMA may react with acrylic mounting resins.

SURFACE	COOLANT/ LUBRICANT	ABRASIVE SIZE/TYPE ANSI ^A (FEPA) ^B	TIME -seconds	FORCE ^C N (lbs)	SURFACE SPEED in rpm ^D	RELATIVE ROTATION
			Planar Grinding			
SiC or Al ₂ O ₃ paper	water	600(P1200) grit	20-30 repeat as needed	13(3)	150	Complimentary ^E
			Rough Polishing			
Soft napless non-woven synthetic cloth	lapping oil ^F	6-µm diamond	30 repeat as needed	18-27(4-6)	150	Complimentary
			Final Polishing			
Soft moderate- napped rayon cloth	distilled water	0.05-µm gamma Al ₂ O ₃	30 repeat as needed	iteh.ai	180	Contra

^A American National Standards Institute (ANSI) designation of grit size.

^B Federation of European Producers of Abrasives (FEPA) designation of grit size.

^C Force per 32 mm (1.25 in.) diameter specimen.

^D RPM for a nominal wheel diameter of 305 mm (12 in.).

^E Complimentary rotation—The head rotation is in the same direction as the wheel. Contra rotation—The head rotation is in the opposite direction as the wheel. ^F There are many aqueous solutions with surfactants and additives to reduce surface tension that can be used in place of lapping oil.

https://standards.iteh.ai/catalog/standards/sist/3bc68c7f-423e-4b78-afbd-6196d9e417db/astm-e2015-042021