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Standard Guide for Preparation of Plastics and Polymeric Specimens for Microstructural Examination[1]

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.

[1] This guide is under the jurisdiction of ASTM Committee E04 on Metallography and is the direct responsibility of Subcommittee E04.01 on Specimen Preparation.

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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