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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Preparation of Compression-Molded Polyethylene Test Sheets and Test Specimens[1]

This standard is issued under the fixed designation D 1928; 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 This practice covers the preparation of compression-molded test sheets of Types I, II, III, and IV polyethylene plastics (Specification D 1248). This practice includes both branched and linear polyethylenes. Two of the procedures given provide for compression-molded sheets to be conditioned by first heating each material above its melting point for a period of time sufficient to erase its prior thermal history and then cooling it from the melt state at a controlled rate while maintaining the required dimensions of the sheet. The third procedure provides for molded test sheets to be prepared by cooling the platens of the compression press, and hence the molten polyethylene plastic, at a controlled rate. Three cooling schedules are provided for as follows:

1.1.1 *Procedure A*, in which the temperature of the initially molten plaque is lowered at a rate of 5 ± 0.5°C/h.

NOTE 1—It is recognized that Procedure A may not be applicable to material containing carbon black due to difficulties, at times, with sheets containing voids. If it is not possible to mold a void-free sheet, Procedure B or C should be selected.

1.1.2 *Procedure B*, in which the initially molten plaque is chilled very rapidly under specified conditions in water, and

1.1.3 *Procedure C*, in which the temperature of the platens of the molding press, and hence of the initially molten plaque, is lowered at a rate of 15 ± 2°C/min.

1.2 This practice method also covers the preparation of test specimens from compression molded test sheets.

1.3 *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.* Specific precautionary statements are given in 7.5.1.

NOTE 2—Although this practice and ISO 293-1986(E) differ in some details, data obtained using either method should be technically equivalent.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]

D 638 Test Method for Tensile Properties of Plastics[2]

D 1248 Specification for Polyethylene Plastics Molding and Extrusion Materials[2]

D 4703 Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets[3]

D 4976 Specification for Polyethylene Plastics Molding and Extrusion Materials[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *picture frame mold*—a flat piece of metal of some dimensions usually of brass or steel that has its center portion removed to give a shape and dimension of the final molding. The thickness of the metal is dependent on the desired thickness of the finished molding, taking into consideration the shrinkage of the material to be molded.

4. Significance and Use

4.1 The conditions under which a polyethylene plastic is formed into a test sheet, particularly the rate of cooling, influence some of the properties of test specimens taken from the sheet. It is, therefore, necessary to control the cooling rate of the test sheet. This practice is intended to minimize interlaboratory variability of test results on compression-molded specimens arising from differences in rate of cooling.

4.1.1 Procedures A and B are designed also to erase moderate differences in the prior thermal history by employing a conditioning period of 1 h at a temperature about 25°C above the melting point of the polyethylene plastic prior to cooling at a controlled rate. For Procedures A and B, the conditioning temperature may have to be raised above this minimum value in some cases to promote adhesion to the aluminum foil parting sheets. The following temperatures, which depend on the type of polyethylene plastic as classified in Specification D 1248, have been found to be generally useful:

Type I 140°C

[1] This practice is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.12 on Olefin Plastics.

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

[3] *Annual Book of ASTM Standards*, Vol 08.03.

Type II 150°C
Type III 155°C
Type IV 155°C

4.1.2 Values obtained on specimens from test sheets prepared in accordance with Procedure C of this practice are useful for the identification of type and the classification by grade of polyethylene plastics in accordance with Specification D 1248.

4.2 Results obtained on specimens from test sheets prepared by any of the procedures described do not necessarily reflect the properties of articles fabricated by other methods, such as extrusion and injection molding.

5. Apparatus, Test Sheet Preparation

5.1 *Two-Roll Mill* that can be heated to a temperature high enough to flux the materials to be tested. Heating may be by steam or electricity.

5.2 *Press*, with platens that can be heated to at least 150°C for Type I, 160°C for Type II, and 177°C for Types III and IV polyethylene plastics.

5.2.1 The press shall have a clamping force capable of applying a pressure (conventionally given as the ratio of the clamping force to the area of the mold cavity) of at least 10 MPa, and shall be capable of maintaining pressure within 10 % of the specified pressure during the molding cycle. (Practice D 4703)

NOTE 3—Pressure is not a key parameter of the molding of polyethylene. Available data shows no additional variability imparted to low, medium or high density polyethylene density measurements when ram force was varied from ten tons to twenty-eight tons.

5.2.2 The platens of the press shall be capable of being heated to at least 240°C and being cooled at a rate consistent with the cooling method selected from 7.10.

5.2.3 The platens or mold shall be heated either by high-pressure steam, by a heat-conducting fluid in an appropriate channel system, or by using electric-heating elements. The platens or mold are cooled by a heat-conducting fluid (usually cold water) in a channel system.

5.2.4 The heating and cooling system in the mold platens shall be such that, when used with a particular mold, they shall be capable of maintaining a temperature difference between points on the mold surfaces of no more than ±5°C during heating or cooling.

5.2.5 For quench cooling (Method C in Table 1), two presses shall be used, one for heating during molding and the other for cooling unless it can be demonstrated that the press used for heating can cool at the specified rate.

5.3 *Molds*:

5.3.1 *Flash-Type Mold*—The flash-type mold may be of the *picture-frame* type, where a steel chase (the picture frame) is sandwiched between two thin steel ferrotype plates (see Fig. 1), or it may be of the machined-cavity type (see Fig. 2), where the mold consists of a cavity machined in a steel plate, with a single steel ferrotype plate used as a top or cover. The cavity, or cavities, in the flash-type mold may be constructed to mold a single plaque from which test specimens may be stamped or machined, or the mold may be built to mold one or more specimens to finished dimensions. Flash molds permit excess molding material to be squeezed out and do not exert molding pressure on the material during cooling. This type of mold is useful for preparing test specimens or panels of similar thickness or comparable levels of low internal stress.

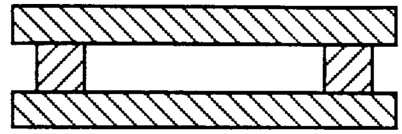

Figure 1

FIG. 1 Flash Picture-Frame Mold

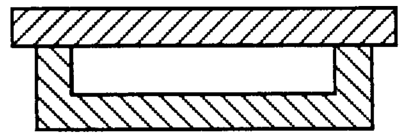

Figure 2

FIG. 2 Flash Mold with Machined Cavity

5.3.2 *Chases*, single-cavity picture frame molds with appropriate dimensions for the particular test specimens for which the molded test sheets are intended.

5.3.3 *Backing Plates,* , flat, for the chases. The backing plates should be strong enough to resist warping or distortion under the molding conditions. Plates made from high thermal conductivity materials such as aluminum, copper or bronze plates or polished steel, of 3.2 to 12.7 mm thick, are suitable.

5.4 *Aluminum Foil,* , 0.05 to 0.2 mm thick, for use as a parting agent in the molding operation.

NOTE 4—It is necessary to use aluminum foil of the specified thickness as a parting agent in the molding operation in Procedures A and B. Much thinner foil is not stiff enough to resist the tendency of the plastic surface to wrinkle on fluxing, while thicker foil does not conform well enough to the plastic surface. Aluminum alloy 1100, Temper O, is suitable.

5.5 *Oven*, capable of maintaining a temperature of at least 175°C. Temperature variation within the oven should be less than 4°C, and the oven should be large enough to accommodate one day's production of molded sheets conveniently.

For Procedure A only:

5.6 *Device* for lowering the oven temperature at a rate of 5.0 ± 0.5°C (9.0 ± 0.9°F)/h.

NOTE 5—A suitable device may be made by connecting a motor-driven thermoregulator or a programmer through a relay to the oven heaters.[4] An alternative and equally satisfactory device comprises a controller and a small electric motor geared to lower the oven temperature at the specified rate.[5]

For Procedure B only:

5.7 *Cooling Tanks*—Tanks into which water may run and continuously overflow and which are large enough to accommodate the chases but small enough to fit into a laboratory sink. Each tank shall contain a mesh basket to hold the chases

[4] Detail drawings of a suitable device are available from ASTM Headquarters. Request Adjunct No. 12-419280-00.

[5] Components of this alternative device may be purchased from the Haydon Corp., Tarrington, CT, and the West Instrument Corp., 4363 W. Montrose Ave., Chicago, IL 60641. The West Guardsman Model JP Controller has been found satisfactory as the controller.

suspended in the cooling water. The basket should be made with as open a construction as possible to facilitate circulation of cooling water over all surfaces of the molding.

For Procedure C only:

5.8 *Polyester Film*, or aluminum foil for use as a parting agent in the molding operation.

NOTE 6—Studies have shown that the type of parting sheet, that is, polyester film or aluminum foil, may cause minor but measurable differences in certain properties. In cases of dispute, the type of parting sheet should be agreed upon by the parties involved.

5.9 *Device* for lowering the temperature of the platens at a rate of 15 ± 2°C/min.

6. Milling

6.1 Compression moldings can be made directly from the granules, pellets, or powders.

NOTE 7—Milled crepe can be made to ensure homogeneity of the material and complete fusing of the material when problems arise or as a referee sample. Addition of an antioxidant is desirable if the molded test sheet is not used subsequently for tests of thermal, oxidative, or environmental stress cracking stability where such additives will interfere.

6.2 Mill rolls should be hot enough to flux materials, but not so hot as to cause them to drip. The crepe should be slashed or turned frequently to promote mixing. Materials should not be milled for more than 5 min after gelling to minimize oxidative and thermal changes.

7. Compression Molding

7.1 A double pressing technique may sometimes be required to squeeze entrapped air out of mill-massed material; if the air is not removed, conditioned sheets may develop voids. A single pressing is usually sufficient, however, to produce void-free test sheets from most polyethylene plastics.

7.2 A flash-type molding operation is involved. The subsequent conditioning step will not give good results unless excess material is squeezed out on all sides and both surfaces of the chase.

NOTE 8—The presence of sink marks in the molded sheet, or the failure of the aluminum foil to adhere tightly all around the chase, usually indicates that an insufficient amount of material has been charged. Failure of the aluminum foil to stick to the plastic can often be remedied by pressing and subsequently conditioning at a higher temperature.

7.3 The press temperature should be warm enough to result in good adhesion between the plastic and the aluminum foil. Recommended minimum temperatures for the press platens are 150°C for Type I, 160°C for Type II, and 177°C for Types III and IV polyethylene plastics.

7.4 Weigh out the amount of plastic necessary to fill the blanked-out volume of the chase and provide an excess, for flash, of not less than 2 and not more than 10 % of the weight of the final molding. (For this purpose, densities of 0.92, 0.93, 0.95 and 0.97 may be assumed for Types I, II, III, and IV polyethylene plastics, (Specification D 1248), respectively. The densities of most carbon black formulations are about 0.01 g/cm^3 higher than those of the natural materials.)

For Procedures A and B:

7.5 Compression molding is performed using parting sheets of aluminum foil. The foil should be cleaned by wiping with acetone and then dried with clean, absorbent paper or cloth. Any wrinkles in the foil should be smoothed out before use. While the foil is usually not suitable for re-use, it may be used again if undamaged.

7.5.1 *Safety Precautions*—See acetone Material Safety Data Sheets for specific hazards information on these materials. The OSHA requirements for 8-h time weighted average and acceptable ceiling concentration are found in 29 CFR, CH XVII, Table Z-2.[6]

7.6 Place the polyethylene plastic in the chase between cleaned aluminum foil parting sheets backed by smooth backing plates. With the platens at the correct temperature, insert the assembly between the platens. Allow a heating period of 5 min with the platens closed and no pressure applied. Then bring to full pressure as quickly as possible. The dwell time at full pressure and cooling rate are optional. After the molding has been formed, remove the assembly from the press. Carefully pry the backing plates off without disturbing the aluminum foil, which should be adhering tightly to the chase and polyethylene plastic sheet. Handle the sheet, chase, and adhering aluminum foil as a unit from this point.

7.7 If a second pressing is desired (see 7.1), proceed as follows: after molding as in 7.6, remove the assembly from the press (take off the aluminum foil) and knock the molded sheet out of the chase. Discard the flash from this sheet. Cut the sheet into at least four pieces of approximately equal size. Replace the pieces within the chase and between aluminum foil parting sheets. The pieces must not be spread over the open volume of the mold but should be stacked so the press can squeeze out any air. Add enough milled crepe to make up for the flash lost in the first molding. Put smooth backing plates behind the aluminum parting sheets and repeat the molding procedure specified in 7.6.

For Procedure C:

7.8 Compression molding is performed using parting sheets of uncoated polyester film or aluminum foil. Any wrinkles in the parting sheets should be smoothed out before use. While foil is usually not suitable for re-use, it may be used again if undamaged.

7.9 Place the polyethylene plastic in the chase between polyester film or aluminum foil backed by smooth backing plates. With the platens at the correct temperature insert the assembly between the platens. Allow a heating period of 5 min with the platens closed and no pressure applied, but with the platens in contact with the mold assembly. Then bring to full pressure as quickly as possible. After full pressure has been applied, continue the heating from 3 to 5 min. At the end of this heating period, turn off the heat and cool the press platens at a rate of 15 ± 2°C/min until a platen temperature of 76°C is reached for Types I and II polyethylene plastics and 95°C for Types III and IV. Continue to cool the platens until they are warm to the touch. Then remove the assembly from the press (Note 8 and Note 9).

NOTE 9—The platen cooling rate can be varied by controlling the flow of water through the platens. The flow of water can be controlled by the

[6] Available from Superintendent of Documents, U.S. Government Printing Office, North Capital and "H" Sts., NW Washington, DC 20401.