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Plastics — Compression moulding of test specimens of thermoplastic materials

Plastiques — Moulage par compression des éprouvettes en matières thermoplastiques

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 293 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This third edition cancels and replaces the second edition (ISO 293:1986), of which it constitutes a minor revision intended, above all, to update the normative references (Clause 2).

Introduction

For reproducible test results, specimens with a defined state are required. In contrast to injection moulding, the aim of compression moulding is to produce test specimens and sheets for machining or stamping of test specimens that are homogeneous and isotropic.

In the process of compression moulding, mixing of material takes place on a negligible scale. Granules and powders fuse only at their surfaces and preforms (milled sheets) are only partially softened.

Isotropic and homogeneous specimens can, therefore, only be obtained when the moulding material is itself homogeneous and isotropic. This has to be considered when processing multiphase materials, such as ABS, which retain their internal structure.

Plastics — Compression moulding of test specimens of thermoplastic materials

1 Scope

This International Standard specifies the general principles and the procedures to be followed with thermoplastics in the preparation of compression-moulded test specimens, and sheets from which test specimens may be machined or stamped.

In order to obtain mouldings in a reproducible state, the main steps of the procedure, including four different cooling methods, are standardized. For each material, the required moulding temperature and cooling methods are as specified in the appropriate International Standard for the material or as agreed between the interested parties.

The procedure is not recommended for reinforced thermoplastics.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 286-1, *Geometrical product specifications (GPS) — ISO coding system for tolerances of linear sizes — Part 1: Bases of tolerances and fits*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
moulding temperature
temperature of the mould or the press during the preheating and moulding time, measured in the nearest vicinity to the moulded material

3.2
demoulding temperature
temperature of the mould or the press platens at the end of the cooling time, measured in the nearest vicinity to the moulded material

NOTE For positive moulds, holes are normally drilled in the mould for measuring the temperatures defined in 3.1 and 3.2.

3.3
preheating time
time required to heat the material in the mould up to the moulding temperature while maintaining the contact pressure

3.4
moulding time
time during which full pressure is applied while maintaining the moulding temperature

3.5
average cooling rate (non-linear)
rate of cooling by a constant flow of the cooling fluid, calculated by dividing the difference between moulding and demoulding temperatures by the time required to cool the mould to the demoulding temperature

NOTE The average cooling rate is usually expressed in degrees Celsius per minute.

3.6
cooling rate
constant rate of cooling in a defined temperature range obtained by controlling the flow of the cooling fluid in such a way that over each 10 min interval the deviation from this specified cooling rate does not exceed the specified tolerance

NOTE The cooling rate is usually expressed in degrees Celsius per hour.

4 Apparatus

4.1 Moulding press

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 mould cavity) of at least 10 MPa.

The pressure shall be maintained to within 10 % of the specified pressure during the moulding cycle.

The platens shall be capable of

a) being heated to at least 240 °C;

b) being cooled at a rate given in Table 1.

The difference between the temperatures of any points of the mould surfaces shall not vary by more than ± 2 °C during heating and ± 4 °C during cooling.

When the heating and cooling system is incorporated in the mould, it shall comply with the same conditions.

The platens or mould 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 mould are cooled by a heat-conducting fluid (usually cold water) in a channel system.

For quench cooling (see method C in Table 1), two presses shall be used, one for heating during moulding and the other for cooling.

For a specified cooling method, the flow rate of the heat-conducting fluid shall be predetermined in a test without any material in the mould.

The temperature may be continuously controlled in the centre between the upper and lower platen of the press.

4.2 Moulds

4.2.1 General

The characteristics of the test specimens prepared by using different types of moulds are not the same. In particular, the mechanical properties depend on the pressure applied to the material during cooling.

In general, two types of moulds, “flash moulds” (see Figure 1) and “positive moulds” (see Figure 2), are used for compression moulding test specimens of thermoplastics.

Figure 1 — Types of flash (“picture frame”) moulds

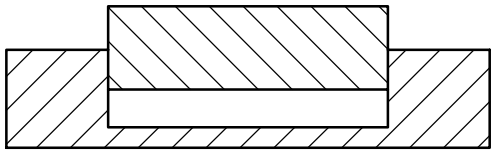

Figure 2 — Positive-type mould

Flash moulds permit excess moulding material to be squeezed out and do not exert moulding pressure on the moulding material during cooling. They are particularly convenient for preparing test specimens or panels of similar thickness or comparable levels of low internal stress.

With positive moulds, the full moulding pressure, neglecting friction, is exerted on the material during cooling. The thickness, stress and density of the resulting mouldings depend on mould construction, size of material charge and the moulding and cooling conditions. This type of mould produces consolidated test specimens with moulded surfaces and is therefore particularly suitable for obtaining flat surfaces or suppressing the formation of voids within test specimens.

4.2.2 Fabrication

The moulds shall be made of materials capable of withstanding the moulding temperature and pressure. The surfaces in contact with the material shall be polished to obtain good surface conditions on the specimens (recommended surface roughness 0,16Ra, see ISO 4287). Specimen removal can be made easier by chromium plating these surfaces. For specimens of small dimensions, a 2° taper is strongly recommended.

Blind holes may be drilled in the mould so that temperature can be measured in the vicinity of the moulded material by using thermocouples or mercury thermometers.

Depending on the performance of the press (see 4.1), the moulds may have built-in heating and/or cooling devices similar to those described for the press platens.

An alloy steel, resistant to mechanical shock and heat-treated to provide a tensile strength of 2 200 MPa, will generally be satisfactory for the moulds. However, in the special case of PVC moulding materials, the use of martensite stainless steel treated to provide a tensile strength of 1 050 MPa is recommended.

4.2.3 Types

4.2.3.1 General

The type of mould used shall be capable of producing test specimens of the types and states specified in the appropriate International Standard for the material or shall be agreed upon between the interested parties.

4.2.3.2 Flash (“picture frame”) moulds

With this type of mould, the excess material is squeezed out and the moulding pressure during cooling is only exerted on the frame and not on the material. The thickness in the centre of the mouldings is slightly less than at the edges due to the shrinkage during cooling. Directly moulded test bars may also have sink marks or voids if the shrinkage is hindered by sticking of the plastic material to the mould.

To overcome these disadvantages, stamping or machining of test specimens from the central part of compression-moulded sheets is preferred.

For moulding sheets, simple and economical flash moulds can be used, consisting of a frame covered with two plates (see Figure 1). The lower and upper plates, having a thickness of about 1 mm to 2 mm, can be made from polished steel or chromium-plated brass to aid release. To avoid the plastic material sticking to the plates, they can be covered by a flexible foil, for example of aluminium or polyester.

Use of a release agent is not allowed.

The thickness of the chase shall be appropriate to the moulded sheet thickness.

The size of the moulding frame shall be such that specimens can be cut or machined without using the outer 20 mm perimeter of the sheet.

4.2.3.3 Positive moulds

These moulds (see Figure 2) are fitted with one or two male pistons and a female part. They allow known pressure, neglecting friction, to be applied to the material, and to be maintained during the moulding and cooling times.

The thickness of the moulding will depend on the quantity of material, its thermal expansion, and the loss of material due to clearances in the moulds. The losses will be a function of the flow of the material at the chosen moulding temperature, the applied pressure, the time over which the pressure is applied, mould construction, etc.

Correct guidance of the male part in the female part is facilitated by use of a round cavity. A fit between these parts of H7g6 (see ISO 286-1) is recommended, i.e. between 15 μm and 90 μm for a round cavity of diameter 200 mm. The mould may be fitted with one or several ejection pins to make part removal easier.

Shims maybe used in positive moulds to aid in controlling thickness. These are removed at the start of the cooling phase.

5 Procedure

5.1 Preparation of moulding material

5.1.1 Drying of granular material

Dry the granular material as specified in the relevant International Standard or in accordance with the material supplier’s instructions. If no instructions are given, dry for 24 h ± 1 h at 70 °C ± 2 °C in an oven.

5.1.2 Preparation of preforms

Direct moulding of sheet from granules shall be the standard procedure, provided that a sufficiently homogeneous sheet is obtained. Normally, this means that the sheet is free from surface irregularities and internal imperfections. Direct moulding from powder or granules may sometimes require melt homogenization using a hot melt milling or mixing procedure to achieve a satisfactory final sheet. Conditions shall be used that do not degrade the polymer. This can usually be achieved by not milling or mixing for more than 5 min after

melting. The preform sheet obtained shall be thicker than the test sheet to be moulded and of sufficient size to enable the test sheet to be moulded.

Storing the preforms in a dry, airtight container is recommended.

5.2 Moulding

Adjust the mould temperature to within $\pm 5\,^{\circ}C$ of the moulding temperature specified in the relevant International Standard or as agreed between the interested parties.

Place a weighed quantity of the material (granules or preforms) in the preheated mould. If granular material is used, make sure that it is evenly spread over the mould surface. The mass of the material shall be sufficient to fill the cavity volume when it is melted and allow about a 10 % loss for a flash mould and about a 3 % loss for a positive mould. With flash moulds, cover the mould with a flexible foil (see 4.2.3.2) and then place the mould in the preheated press.

Close the press and preheat the material charge by applying a contact pressure for 5 min. Then apply full pressure for 2 min (moulding time, see 3.4) and then cool down (see 5.3).

A preheating time of 5 min is the standardized time for evenly spread material charges sufficient for sheets up to 2 mm thickness. For thicker mouldings, this time shall be adjusted accordingly.

NOTE At contact pressure, the press is just closed with a pressure low enough to avoid flow of the material. Full pressure means a pressure sufficient to shape the material and squeeze out the excess material.

5.3 Cooling

5.3.1 General

With some thermoplastics, the cooling rate affects the ultimate physical properties. For this reason, the cooling methods are specified in Table 1.

The method of cooling shall always be stated together with the final physical properties. The appropriate cooling method is normally given in the relevant International Standard for the material. If no method is indicated, method B shall be used.

5.3.2 Cooling methods

The appropriate cooling method shall be selected from Table 1.

Table 1 — Cooling methods

Cooling method	Average cooling rate (see 3.5) $^{\circ}C \cdot min^{-1}$	Cooling rate (see 3.6) $^{\circ}C \cdot h^{-1}$	Remarks
A	10 ± 5		
B	15 ± 5		
C	60 ± 30		Quench cooling
D		$5 \pm 0{,}5$	Slow cooling

In the case of quench cooling (see method C in Table 1), transfer the mould assembly from the heating press to the cooling press as quickly as possible by suitable means, for example using a pair of tongs.

The demoulding temperature shall be $\leqslant 40\,^{\circ}C$ if no other instructions are given.

The use of two presses is required for method C (see 4.1).

Method D is recommended for producing test specimens free of any internal stress or for slow cooling after annealing of previously prepared sheets.

6 Inspection of the moulded specimens or sheets

After cooling, check the appearance of the moulded specimens or sheets (for sink marks, shrink holes, discoloration) and for conformance to specified dimensions. If any moulding defects are found, the test specimens or sheets shall be discarded.

Make sure that there is no degradation or unwanted crosslinking, using the method specified in the relevant International Standard or as agreed between the interested parties.

7 Specimen preparation report

The test report shall contain the following information:

a) a reference to this International Standard;

b) the dimensions of the specimens and their intended use;

c) all details necessary for complete identification of the moulding material (type, designation, etc.);

d) details of the preparation of the moulding material:

   1) drying conditions for granulates and powder,

   2) processing conditions used in the preparation of preforms and their average thickness;

e) the type of mould and foil used;

f) the moulding conditions:

   1) preheating time,

   2) moulding temperature, pressure and time,

   3) cooling method used,

   4) demoulding temperature;

g) the state of the specimens, if applicable;

h) the date of preparation of the specimens;

i) any other observations.

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