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International Standard



293

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Plastics — Compression moulding 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 293 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 293-1974), of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Plastics — Compression moulding test specimens of thermoplastic materials

0 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.

1 Scope and field of application

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 shall be as specified in the appropriate International Standard for the material or as agreed between the interested parties.

NOTE — The procedure is not recommended for reinforced thermoplastics.

2 References

ISO/R 286, *ISO system of limits and fits — Part 1: General, tolerances and deviations*.

ISO 468, *Surface roughness — Parameters, their values and general rules for specifying requirements*.

3 Definitions

For the purpose of this International Standard, the following definitions apply.

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

3.2 demoulding temperature: The 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: The time required to heat the material in the mould up to the moulding temperature while maintaining the contact pressure.

3.4 moulding time: The 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.

The average cooling rate is usually expressed in kelvins 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 shall not exceed the specified tolerance.

The cooling rate is usually expressed in kelvins 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.

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The platens shall be capable of

- a) being heated to at least 240 °C;
- b) being cooled at a rate given in the table.

The difference between the temperatures of any points of the mould surfaces shall not vary by more than ± 2 K during heating and ± 4 K 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 the table), two presses shall be used, one for heating during moulding and the other for cooling.

NOTES

- 1 For a specified cooling method, the flow rate of the heat-conducting fluid should be predetermined in a test without any material in the mould.
- 2 The temperature may be constantly controlled in the centre between each 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 mould 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.

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 a good surface

condition on the specimens (recommended surface roughness $0,16 R_a$, see ISO 468). 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.

NOTES

- 1 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.
- 2 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

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.

4.2.3.1 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 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.

4.2.3.2 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/R 286) is recommended, i.e. between 15 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 may be 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 ± 1 h at 70 ± 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.

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

5.2 Moulding

Adjust the mould temperature to within ± 5 K 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 flexible foils (see 4.2.3.1) 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)

NOTES

1 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 has to be adjusted accordingly.

2 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 the table.

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 the table.

Table — Cooling methods

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

In the case of quench cooling (see method C in the table), 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 ≤ 40 °C if no other instructions are given.

NOTES

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

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

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6 Inspection of the moulded specimens or sheets

After cooling, check the moulded specimens or sheets for appearance (i.e. for sink marks, shrink holes, discolorations) 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 cross-linking, using the method specified in the relevant International Standard or as agreed between the interested parties.

7 Test report

The test report shall contain the following information:

- a) reference to this International Standard;
- b) dimension of the specimen and its intended use;
- c) complete identification of moulding material (type, designation, etc.);
- d) preparation of moulding material:
 - 1) drying conditions for granulates and powder,
 - 2) processing conditions used in the preparation of preforms and their average thickness;
- e) type of mould and foil used;
- f) moulding conditions:
 - 1) preheating time,
 - 2) moulding temperature, pressure and time,
 - 3) cooling method used,
 - 4) demoulding temperature;
- g) state of specimen, if applicable;
- h) any other observations.



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Figure 1 — Types of flash ("picture frame") moulds

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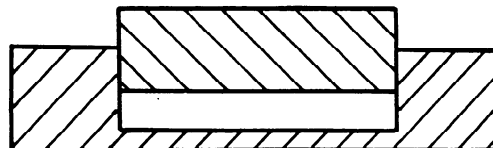


Figure 2 — Positive-type mould