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Plastics — Compression moulding of test specimens of thermoplastic materials

Plastiques — Moulage par compression des éprouvettes en matières thermoplastiques

ISO/CEN PARALLEL PROCESSING

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC *61*, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 293:2004), which has been technically revised.

The main changes are as follows:

— the definition for "cooling rate" has been revised (see 3.6);

— requirements regarding the biggest clamping force and the highest platens temperature have been changed (see 4.1);

— the description of common specifications of positive mould has been given (see 4.2.3.3);

— the conditions for the use of vacuum oven while material drying have been added (see 5.1.1);

— the methods of cooling rate have been revised (see Table 1);

— a bibliography has been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

For reproducible test results, specimens with a specified 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.

The cooling rate in the crystallization stage has a great influence on the properties of semi-crystalline or crystalline polymer (such as PB, PE, PP, etc.), so it is necessary to control the cooling rate more strictly at the cooling stage.

Plastics — Compression moulding of test specimens of thermoplastic materials

1 Scope

This document 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 can be machined or stamped.

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

This document is not applicable to reinforced thermoplastics.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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

Note 1 to entry: The moulding temperature is usually expressed in degrees Celsius.

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 1 to entry: The demoulding temperature is usually expressed in degrees Celsius.

3.3
preheating time
time required to heat the material in the mould up to the *moulding temperature* (3.1) while maintaining the contact pressure

Note 1 to entry: The preheating time is usually expressed in minute.

3.4
moulding time
time during which full pressure is applied while maintaining the *moulding temperature* (3.1)

Note 1 to entry: The moulding time is usually expressed in minute.

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 temperature* (3.1) and *demoulding temperature* (3.2) by the time required to cool the mould to the *demoulding temperature* (3.2)

Note 1 to entry: The average cooling rate is usually expressed in degrees Celsius per minute.

3.6
cooling rate
controlled cooling rate
<linear>constant rate of cooling in a specified temperature range obtained by controlling the flow of the cooling fluid in such a way that over each specified time interval the deviation from this specified cooling rate does not exceed the specified tolerance

Note 1 to entry: The cooling rate is usually expressed in degrees Celsius per minute.

Note 2 to entry: The specified time interval-controlled cooling rate is based on the system design of moulding press. Usually the shorter time interval used, the more accurate controlled cooling rate can be gained.

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 12 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 320 °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 mould (picture frame mould)” (see Figure 1) and “positive mould” (see Figure 2), are used for compression moulding test specimens of thermoplastics.

Figure 1 — Schematic diagram of flash mould (picture frame mould)

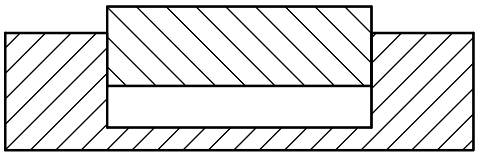

Figure 2 — Schematic diagram of positive mould

Flash mould permits 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 0,16 Ra, see ISO 21920-2). 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.

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 mould (picture frame mould)

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 4 mm to 5 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 the main test area of the specimens can be cut or machined without using the outer 20 mm perimeter of the sheet.

4.2.3.3 Positive mould

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.

NOTE 1 For positive moulds, if needed, holes are normally drilled in the mould for measuring the temperatures.

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.

NOTE 2 For the majority of specimen types and dimensions specified in ISO 20753, a square cavity can be used such as 170 mm × 170 mm for obtaining enough specimens for one test by each moulding and it also can make the mould weight lighter.

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, or for 16 h ± 1 h at 80 °C ± 2 °C in a vacuum 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

