

English version

Fibre reinforced plastics - Glass mat reinforced thermoplastics
(GMT) - Determination of flowability and solidification

Plastiques renforcés de fibres - Thermoplastiques
renforcés de mats de verre (GMT) - Détermination de la
fluidité et de la solidification

Faserverstärkte Kunststoffe - Glasmattenverstärkte
Thermoplaste (GMT) - Bestimmung der Fließfähigkeit und
des Erstarrens

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Foreword

This document (EN 14447:2005) has been prepared by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2005, and conflicting national standards shall be withdrawn at the latest by October 2005.

The description of flowability and solidification of glass fibre mat reinforced thermoplastic moulding compounds (GMT) is complex.

The test methods described in this document are for the determination of flowability and solidification. The flowability is the basic property of a moulding material which defines the ability to fill a given cavity.

The flowability of GMT has to be expressed by at least two parameters because of its non-Newtonian behaviour.

Parameters to express the behaviour of the compound as instantaneous values are:

- the pressure gradient – a measure of the resistance to the initiation of flow ;
- the pressure loss – a measure for the pressure change over the flow distance.

As overall or integral values are:

- the filling time – the duration of filling the cavity under given conditions ;
- the pressure integral – a measure of the resistance to overall flow ;
- the work during pressing – a measure of resistance to overall flow ;
- the partial work during pressing – a measure of resistance during a specific part of the flow.

These parameters are defined by this document plus other additional information.

The process of solidification must be determined separately and must not influence the results of the flowability test.

The complexity of the behaviour of GMT requires a large amount of testing for full understanding of the compound. This document specifies a method of test for an intensive analysis. Not all elements of the analysis may be required for every function for which this test method is applicable. The material (product) specification, or the person requesting the analysis should specify which elements of the analysis are required to be undertaken for a particular purpose e.g. product development, product qualification or quality control. To reduce the specific effort for the method and to open the applicability of the test equipment, the apparatus is similar to the one described in EN ISO 12115, *Fibre-reinforced plastics - Thermosetting moulding compounds and preregs - Determination of flowability, maturation and shelf life (ISO 12115:1997)*.

The geometry of the mould was chosen to enable the user to produce test plates for mechanical testing separate from the flowability test.

The effect of glass fibre reinforcement properties on the flowability was also assessed using a stepped plate mould which was supposed to produce more clearly distinguishing test results compared to a flat plate. This assumption was not verified in tests run up to now.

A second mould geometry therefore may be described later if necessary.

Orientation of the reinforcement fibres should be observed and tested by taking the samples either in the direction of the production machine or transverse to it.

Experience shows that the procedure for heating the material for moulding has a very strong influence on the results of the flowability and solidification test. To avoid unwanted effects an exact specification of heating is included.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This document defines a method for the determination of data suited to the assessment of the flowability and solidification of polypropylene based glass fibre mat reinforced thermoplastic moulding compounds (GMT) carried out with commonly applied moulding parameters.

The influence on moulding behaviour of both the individual components of the material and the moulding parameters may be determined by this method. It is suitable for quality control purposes as well as for the development of material formulations.

Depending on the objectives of the analysis, different elements of the procedure described in this document may be chosen.

This test method may be used for the production of a test plate suitable for the preparation of test specimens for the determination of materials properties.

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.

EN ISO 291, *Plastics — Standard atmospheres for conditioning and testing (ISO 291:1997)*

EN ISO 472:2001, *Plastics — Vocabulary (ISO 472:1999)*.

EN ISO 12115:1997, *Fibre-reinforced plastics — Thermosetting moulding compounds and preregs — Determination of flowability, maturation and shelf life (ISO 12115:1997)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN ISO 472:2001 and the following apply.

3.1

definition of GMT

glass mat reinforced thermoplastics

3.2

flowability

ability of a moulding material to flow and fill the cavity of a given mould under defined conditions

3.3

solidification

process of transition of the moulding material from a mouldable stage to a stage of sufficient rigidity for demoulding

3.4

elementary unit

smallest normally commercially available entity of a given product. The description (form, dimensions, mass, etc.) of the elementary unit will normally be defined in the product specification. This unit may be supplied in one of several forms :

- package ;
- pallet.

NOTE For a given product, the dimensions, mass or volume of the elementary unit may change, as fabrication techniques evolve, without necessarily causing any modification in the properties or the way in which these properties vary throughout the elementary unit.

4 Principle of the test method

This method is based on a moulding procedure using a normal commercial press, a plate mould and the usual conditions for moulding polypropylene GMT.

Usual moulding conditions for polypropylene GMT include:

- mould temperature c.a. 60 °C ;
- material temperature c.a. 190 °C – 230 °C ;
- closing speed of the press c.a. 15 mm/s ;
- thickness of the moulding c.a. 3 mm.

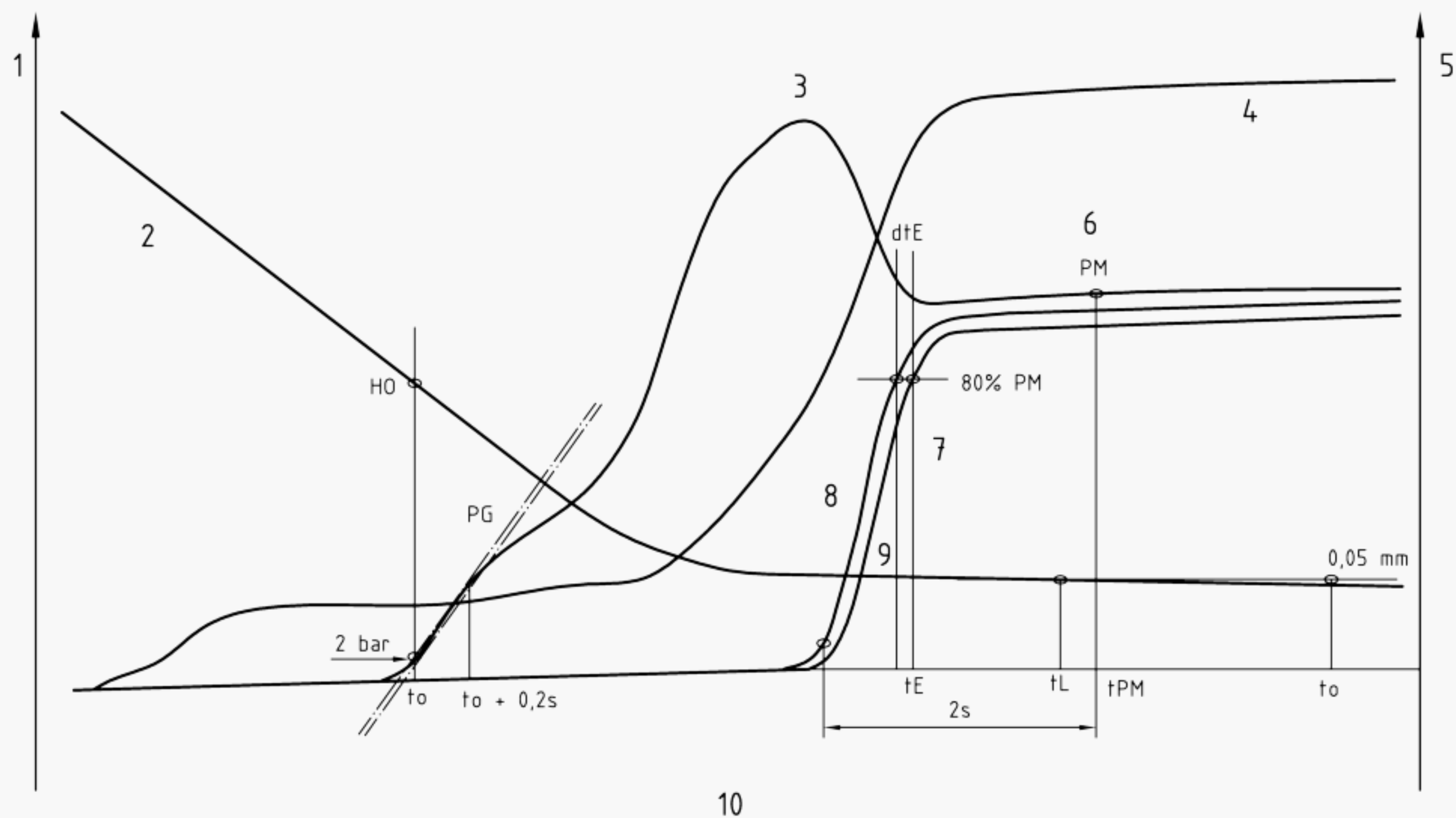
The GMT, cut to the required size and heated to moulding temperature is inserted in the centre of the cavity. Then the material is allowed to flow and solidify under pressure.

The moulding conditions are defined by a given press closing speed, force and mould temperature. Complete filling of the cavity is a precondition.

During the moulding process, the closing force and the material pressure in the centre and at the corners of the cavity are measured and recorded as a function of time (see Figure 1).

From the measured sensor signals the results are evaluated and the flowability and solidification is determined and judged in relation to the specific mould filling requirements. This document offers several elements for the analysis of this measured data. Depending on the purpose of the test some of the elements of the analysis may be omitted.

The determination of the flowability and solidification of different tests requires consistent moulding parameters.



Key

- | | |
|------------------------|---------------------|
| 1 Pressure | 6 Material pressure |
| 2 Closing displacement | 7 Pressure edges |
| 3 Pressure centre | 8 Left |
| 4 Closing force | 9 Right |
| 5 Displacement | 10 Time |

Figure 1 — Pressure and Displacement response during the test

5 Sampling

Three specimens shall be selected from the elementary unit or from the laboratory sample.

NOTE 1 GMT is normally delivered in stacks of blanks, not in the full width of sheets as manufactured. Their size and shape may influence how the sample is taken.

If the blank is of sufficient size, take all three specimens from the one blank, one from the left, one from the middle and one from the right.

If the blank is not large enough to take all three specimens from the one blank then take only one specimen per blank, selecting three blanks successively from the stack.

NOTE 2 If the blank is not large enough to take a single complete specimen, arrangements should be made with the manufacturer to supply sufficient number of larger blanks from the same production lots to enable this test to be undertaken.

To define the influence of possible orientation, documentation of the production direction relative to the sampling direction is required. The product specification or the person ordering the analysis may require that such determination is to be performed on more specimens or at specific places of the elementary unit or laboratory sample.

6 Conditioning

Specific thermoplastic matrices may require conditioning. Details of the conditioning process shall be defined with the person ordering the test in accordance with EN ISO 291.

7 Apparatus

7.1 Hydraulic moulding press

A hydraulic moulding press with a closing speed of 15 mm/s and capable of a pressure of 14 MPa loaded onto the moulding material. For the purpose of this method the respective press shall be used purely as force controlled and must not be speed controlled.

The closing force must be measured with a sensor and be adjustable to an accuracy within 3 % of the stationary value. The pressure build up time (tested by closing the press without material in the mould) to reach 80 % of the nominal closing force must be $(0,60 \pm 0,05)$ s. A stationary force value of 100 % of the nominal closing force shall be reached after 1,0 s.

7.2 Temperature controlled plate mould

A temperature controlled plate mould (see Figure 2) with a minimum width of 200 mm and a length of 590 mm, fixed onto the press described above.

This shear edge mould contains three pressure sensors; on the middle, and one on each of the cavity's corners. The sensors shall be built in secure and level with the surface. Outside the mould, a displacement sensor shall be installed, capable of detecting mould movement up to 25 mm. The displacement measurement shall indicate the thickness of the material in the cavity (see also EN ISO 12115). The shear edge shall be adjusted to c.a. 0,03 mm.

NOTE For pressure measurements in the cavity piezoelectric sensors are recommended.

To acquire data as viscosity etc., essential for simulation software, isothermal tests may be required. In this particular case, the mould must be capable of operating at temperatures above 200 °C.

Dimensions are in millimetres

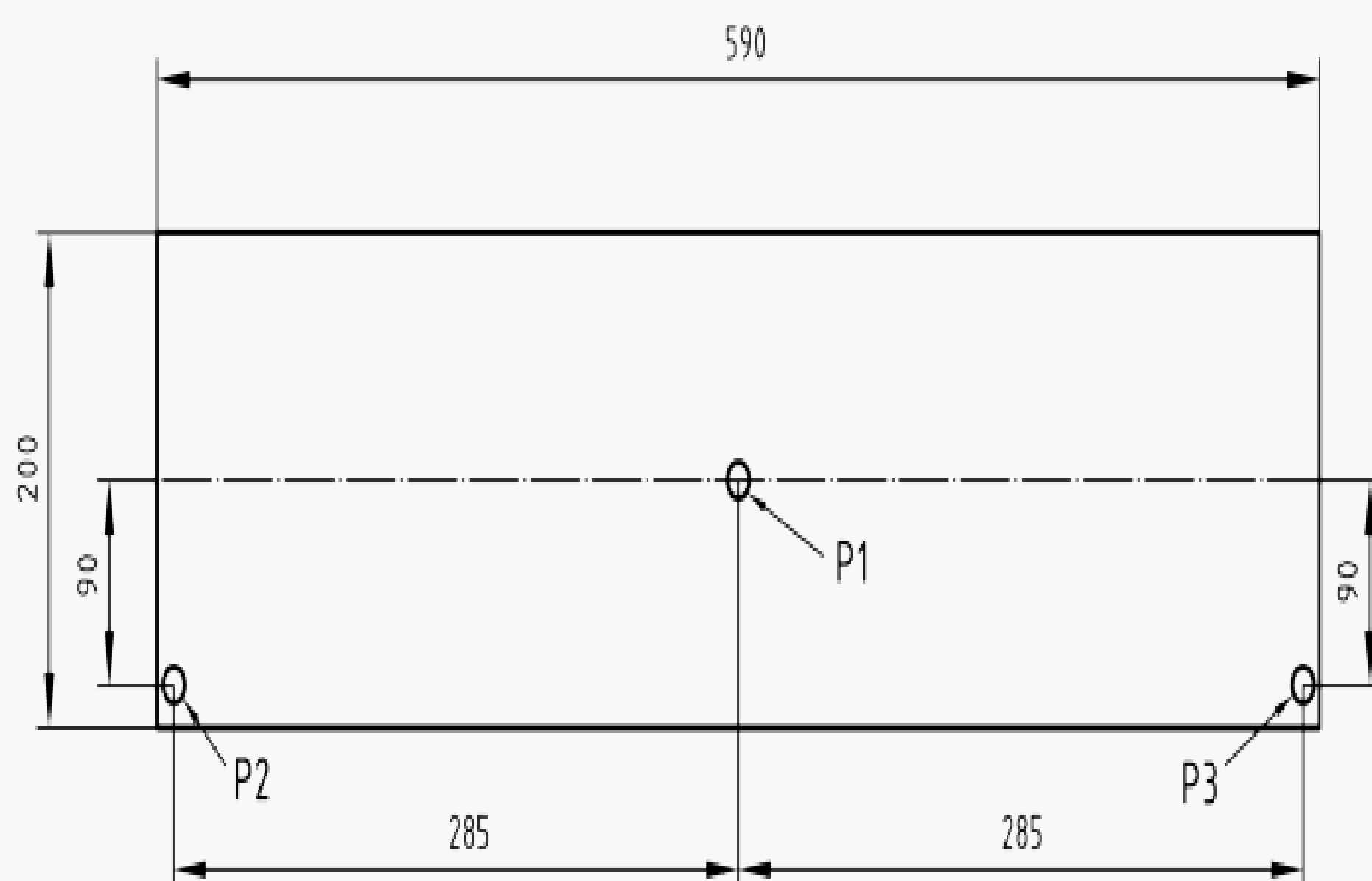


Figure 2 — Press mould 200 x 590

7.3 Heating oven

The GMT material shall be heated in a plate contact oven.

The plates shall maintain contact with the specimen and keep the GMT compacted while heating. The temperature of the heating platens must be controlled to an accuracy of ± 2 °C. The heating time must be measured with an accuracy of ± 1 s. The end of heating time must be defined by an optical or acoustic signal.

NOTE The method of heating may affect the temperature distribution and the material consistency and even cause degradation of the polymer. Such effects may also cause variability of the test results. To prevent unwanted effects upon the material, heating by contact with heating plates, covered by a mould release foil, is recommended.

The heating time shall be determined by measuring the temperature of the material in a reference heating test. The heating time depends upon the material and its thickness. Usually the heating time ranges between 2 min and 5 min for a thickness of c.a. 4 mm. For heating of more than one layer the heating time must be increased.

7.4 Sensor reading equipment

Equipment to record the closing force and the material pressure in the centre and on both corners of the cavity.

Equipment to record the mould movement.

Equipment to record the closing force, pressure and displacement, connected to the respective sensors. The closing force measured must not contain any influence or effects of friction or inertia of mould or press.

Calibration range shall be:

— hydraulic pressure (closing force)	0 bar to 280 bar / 0 kN to 2 000 kN ;
— cavity pressure	0 bar to 250 bar ;
— displacement	0 mm to 25 mm.

8 Preparation of test specimen

The test specimen used to charge the mould is prepared using equally sized blanks of GMT. After heating the blanks must be stacked on top of one another.

The width of the blanks is smaller than the cavity width.

NOTE Some GMT materials exhibit an increase of blank size while heating and melting. The width of the cut specimen is chosen such that the stacked blanks fit the width of the mould without causing problems during loading the mould but also not flowing in the width direction during the test.

The length can be chosen to achieve a thickness of the moulded plate of 3 mm. The allowed range of length of the blanks is 25 % or 33 % of the mould length.

The thickness of the plate shall be 3 mm for a maximum result resolution. In agreement between the interested parties the thickness of the test plate can be adjusted to be equal to a specific production moulding.

9 Procedure

- Place specimens into the heating oven.

NOTE 1 The temperature has a decisive effect on flowability and solidification. The oven temperature and the heating time should be agreed upon by the interested parties ordering or running the test.

- The preferred mould temperature is (60 ± 2) °C.

- The closing force shall be chosen, such that the cavity pressure (material pressure) is 7 MPa. If other pressures are required the pressure of 3,5 MPa or 14 MPa shall be chosen.
- Start the recording system.
- Take specimens from the oven immediately after end of heating time and stack the individual blanks into one package. The stacking of the blanks and their orientation shall be noted.
- Insert specimens exactly into the centre of the cavity and immediately close the mould. The mould stamp must come into contact with the material within (35 ± 5) s after end of heating time including a time no longer than 4 s after placing the charge into the mould.

NOTE 2 The handling of the heated blanks in air after taking out of the oven may cause loss of temperature. To reduce this effect to a minimum possible, the handling time is restricted. The most severe loss of temperature to the unflowing GMT is caused by the mould surface. Therefore an additional requirement is defined restricting the resting time in the mould prior to flow.

- Stop the recording system after c.a. 20 s for flowability measurement or after c.a. 90 s for additional solidification measurement. For moulding thickness larger than 3 mm increase the required times.
- Wait for sufficient solidification and open the mould.

10 Expression of the results

10.1 General

The results shall be evaluated from the recorded curves (see Figure 1).

10.2 Start of flow t_0

The time of start of flow is the beginning of the pressure rise up to 2 bar in the centre of the cavity.

10.3 End of filling t_E

The time end of filling is defined by the pressure rise at the corners of the cavity to 80 % of the material pressure. The time of filling is defined by the later of the two pressures measured at the corners. The time of the two pressures in the corner of the cavity shall be compared to state symmetry of filling.

If the time difference between the two pressures at 80 % of the material pressure determined to define filling time exceeds 3 % of the filling time the respective test shall be neglected and be repeated.

10.4 Filling-time t_F

$$t_F = t_E - t_0 \quad (1)$$

10.5 Starting Height H_0

The closing position at time t_0 is the starting height H_0 .

10.6 Thickness D

The material thickness at the time t_{PM} as defined by the material pressure PM (see 10.7) is the thickness D .

10.7 Material Pressure PM

The pressure reached in the centre of the cavity at the time 2 s after the first rising of the pressures in the corners of the cavity (which defines the time t_{PM}) is the material pressure PM .

10.8 Pressure Gradient PG

The gradient PG is defined as the difference of the pressures in the centre of the mould between the zero-point t_0 and at $t_0 + 0,2$ s divided by the duration of the increase (0,2 s).

$$PG = \frac{P_1(t_0 + 0,2 \text{ s}) - P_1(t_0)}{0,2 \text{ s}} \quad (2)$$

10.9 Pressure Drop PD

The pressure in the centre of the cavity when the later of the pressures at the corners reaches 2 bar divided by the distance of the pressure transducers.

10.10 Pressure Integral PI

The integral of the material pressure in the centre of the cavity shall be determined between the t_0 and t_E .

10.11 Work during pressing PL

The integral of the closing force (applied onto the material) between positions reached at t_0 and t_E .

10.12 Partial work during pressing PPL

Pressing work between other positions as defined in the material specification.

10.13 Thickness Shrinkage Sz

Thickness shrinkage shall be determined between t_E and t_Q . The time t_Q is the time of equilibrium. The time of equilibrium is determined by the displacement curve reaching the horizontal. The displacement value at t_Q is $d(t_Q)$.

Thickness shrinkage Sz is calculated by:

$$Sz = \frac{d(t_E) - d(t_Q)}{d(t_Q)} \times 100 (\%) \quad (3)$$

10.14 Cooling Time t_c

The end of cooling is defined by determining the intersect of a parallel line to the displacement curve at t_Q with a position of $d(t_Q) + 0,05$ mm and the displacement curve.

This intersect defines the time t_L .

Cooling time t_c is calculated by:

$$t_c = t_L - t_E \quad (4)$$

11 Test report

The test report shall include:

- a) full description and identification of the tested moulding material.
- b) exact description of sampling.
- c) test conditions.
- d) number, stacking and the geometry of the GMT – blanks used.
- e) starting height H_0 and the thickness D .
- f) material pressure and the closing force.
- g) individual test results as required in the product specification.
- h) each individual value of the expression of the results required in the product specification.
- i) three and more individual values, the average value and standard deviation.
- j) operating details not mentioned in this document that may affect the results.

Annex A (informative)

Other type of mould use

The mould may also be used for testing and flowability, maturation and shelf life and the curing characteristics of thermosetting moulding materials (according to Method 2 of EN ISO 12114 "*Fibre-reinforced plastics — Thermosetting moulding compounds and preregs — Determination of cure characteristics (ISO 12114:1997)*") respectively Method 2 of EN ISO 12115:1997 "*Fibre-reinforced plastics — Thermosetting moulding compounds and preregs — Determination of flowability, maturation and shelf life (ISO 12115:1997)*"). An additional temperature sensor may be placed in the centre of the mould according to 7.2 of Method 2 of EN ISO 12114:1997.

Annex B (informative)

Preparation of test specimens for mechanical testing

In case of using the system described here for the production of test plates for mechanical testing the provisions of Clause 8 (Preparation of test specimen) shall be followed with the following changes.

The thickness for mechanical testing shall be in accordance with the relevant test method.

The closing force shall be chosen to result in 14 MPa material pressure.

The charge coverage of the mould shall be in the range of 25 % - 30 %.

Bibliography

EN ISO 12114:1997, *Fibre-reinforced plastics — Thermosetting moulding compounds and preregs — Determination of cure characteristics (ISO 12114:1997)*.