
**Fibre-reinforced plastics — Thermosetting
moulding compounds and preregs —
Determination of flowability, maturation
and shelf life**

*Plastiques renforcés de fibres — Compositions de moulage
thermodurcissables et préimprégnés — Détermination de la fluidité,
de la maturation et de la durée de vie*



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Foreword

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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.

International Standard ISO 12115 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

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Introduction

The flowability of a moulding compound is a property which describes the ability of the compound to fill a mould cavity during the moulding operation.

The flowability varies with the age of the moulding compound due to the occurrence of a thickening process. This so-called maturation process is normally initiated by an additive, the aim being to prevent significant separation of the components of the moulding compound, and yet ensure sufficient flow for the moulding compound to be easy to handle and for it to spread out to fill the whole mould cavity when it is moulded.

When the flowability of a moulding compound has developed to a defined limit, the compound is said to have reached its matured state. This means it can be handled and moulded satisfactorily under given operating conditions.

The maturation and shelf life are determined from flowability measurements. The flowability is measured at several different points in time after production of the moulding compound, and the change in flowability plotted as a function of time. The shelf life of the moulding compound is determined by assessing the ease of handling and moulding behaviour as given by the flowability. Experience indicates that in some cases shelf life may also be dependent on the cure characteristics of the moulding compound (see ISO 12114).

Maturation and shelf life are not parameters in their own right. For a particular moulding compound, the shelf life, for instance, may even differ from one set of moulding conditions to another and from one application to the next.

Fibre-reinforced plastics — Thermosetting moulding compounds and preregs — Determination of flowability maturation and shelf life

1 Scope

This International Standard specifies two methods for the determination of the flowability of fibre-reinforced thermosetting moulding compounds and preregs. The methods apply to all fibre-reinforced thermosetting moulding compounds and differ in the test conditions and the apparatus required.

The methods may be used to assess the influence of individual components of the moulding compound on the moulding behaviour by determining the flowability of the compound. They are also suitable for quality control purposes, as well as the development of moulding compound formulations.

The major field of application is with moulding compounds based on unsaturated polyester (UP) resins.

Method I is a flowability test carried out at room temperature. Conducting the test at room temperature reduces the effect on the results of changes in the temperature of the moulding compound during the test.

Method II is a flowability test carried out under commonly used moulding conditions. In addition, the plate produced may be used for further testing.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 472:1988, *Plastics — Vocabulary*.

ISO 8605:1989, *Textile glass reinforced plastics — Sheet moulding compound (SMC) — Basis for a specification*.

ISO 8606:1990, *Plastics — Preregs — Bulk moulding compound (BMC) and dough moulding compound (DMC) — Basis for a specification*.

ISO 12114:1997, *Fibre-reinforced plastics — Thermosetting moulding compounds and preregs — Determination of curing behaviour*.

3 Definitions

For the purposes of this International Standard, the terms and definitions given in ISO 472, ISO 8605 and ISO 8606 apply, plus the following definitions:

3.1 flowability: The time-depending ability of a thermosetting moulding compound to flow and fill the cavity of a given mould under given conditions.

3.2 maturation: The process of thickening of thermosetting moulding compounds to a given level of flowability without significant separation of the components.

3.3 matured state: The level of thickening at which the flowability of a thermosetting compound is such that it can be handled and moulded satisfactorily under given operating conditions.

3.4 shelf life: The period after production of a thermosetting compound during which the flowability remains at a level at which the compound can be moulded without the need to make significant changes in the moulding conditions from those normally used.

3.5 elementary unit: The 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. Elementary units may be supplied in the form of rolls or packages, for instance.

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

4 General

4.1 Method I

This method calls for an unconstrained test specimen to be subjected to a constant load by a punch. The specimen is caused to flow to the side, and the change in height of the punch as it sinks is measured (see figure 1).

Fibre-reinforced moulding compounds exhibit a change in flowability during a flow process, due to their non-Newtonian behaviour. Two values are therefore needed to express the flowability of a moulding compound, one value expressing the instantaneous flowability and the other the change in flowability due to the non-Newtonian behaviour of the compound.

The vertical motion of the punch depends on the flowability of the moulding compound and on the applied load. A moulding compound is considered as being suitably tested if its flowability is such that, after a load application period of 45 s, the punch has sunk to a level between 30 % and 70 % of the original thickness of the specimen. The test load is therefore selected to meet this condition.

By carrying out the test at suitable intervals after production, the maturation thickening process can be analysed, in particular the point at which this process levels out and the point at which the compound ceases to be mouldable without significant changes in the moulding conditions, the difference between these two points being the shelf life.

NOTE — For unsaturated polyester resin, the dependence of this thickening on temperature is a non-linear one. Consequently, the results of tests at room temperature may not accurately reflect the performance of the material at the actual moulding conditions.

4.2 Method II

This method calls for a test specimen to be moulded in a heated mould in a press under the normally used production conditions. The method is designed to determine the force necessary for the specimen to spread out and fill the mould within a certain time.

A given mass of sheet moulding compound, cut to the required size, or bulk moulding compound is placed in the centre of the mould cavity. The moulding compound is then caused to flow and is cured at the required pressure and temperature. The mould closing force is chosen such that filling of the cavity takes $10 \text{ s} \pm 5 \text{ s}$.

It is important that the curing process has no effect on the flowability of the moulding compound during the time in which the compound is filling the mould. For fast-curing compounds, therefore, a filling time of 10 s may be too long, and a filling time of 5 s may be required.

During the moulding process, the mould closing force and the pressure in the moulding compound at the centre and at the rim of the cavity are measured with sensors and recorded as a function of time (see figure 2). An assessment of the flowability (i.e. whether it is acceptable or not) is then made from the curves thus produced.

Comparison of flowability assessments from different tests requires consistent moulding conditions. For results to be comparable, any change in moulding conditions shall be restricted to the mould closing force.

NOTE — The aim of this method is to gain the most extensive information possible about the flowability of a moulding compound during the filling of the mould cavity. However, it is also possible, by choosing to sacrifice some information, to reduce the number of sensors used.

The sensor outputs can be recorded on a chart recorder or in a computer.

5 Apparatus

5.1 Method I

5.1.1 Apparatus for applying a constant load to the test specimen (see figure 3).

The apparatus consists of a stand, a weightpiece and a lever system designed to transmit the force produced by the weightpiece to a punch acting on the test specimen. The apparatus shall be frictionless, and the punch shall be mounted to move vertically.

The diameter of the punch shall be 300 mm. A platen with a diameter of 30 mm and a height of 3 mm shall be located under the punch. The force produced by the punch alone (deadweight of punch) shall be 11,0 N.

The apparatus shall be designed so that loads of 390 N, 1 000 N and 2 000 N can be applied to the test specimen, and so that the speed at which the punch descends on to the specimen at the beginning of the test is $1,5 \text{ mm/s} \pm 0,2 \text{ mm/s}$.

5.1.2 Displacement indicator, with an accuracy of $\pm 0,1 \text{ mm}$ or better, mounted to indicate the distance between the punch and the platen when the test specimen is under load.

5.1.3 Stopwatch, or other suitable time measurement device.

5.1.4 Punch attachment, for preparing test specimens from a bulk moulding compound.

An attachment designed to enlarge the bottom surface of the punch can be fitted to the punch (see figure 4) to prepare bulk moulding compound test specimens. The moulding compound is covered by aluminium foil, placed

inside the shoe attachment and compressed and compacted to a certain thickness by the punch. If this attachment is used, the punch motion shall be accurately limited, preferably by means of a mechanical stop acting on the punch or the lever system.

5.2 Method II

5.2.1 Hydraulic moulding press, capable of producing a mould closing speed of 3,5 mm/s and a mould closing force of up to 310 kN. The mould closing force shall be measured with a sensor and the steady-state value shall be adjustable to an accuracy of $\pm 3\%$. The time to reach the steady-state value in a static test shall be $0,6 \text{ s} \pm 0,1 \text{ s}$.

5.2.2 Heated shear edge mould (see figures 5 and 6), with a width of 200 mm and a minimum length of 590 mm, fixed to the press. The mould shall contain three pressure sensors: one in the middle and one at each end of the mould cavity. The sensors shall be fitted flush with the inside surface of the mould.

A displacement sensor shall be mounted outside the mould to measure vertical movement of the top of the mould of up to 20 mm.

The displacement measurements shall indicate the thickness of the moulding compound in the mould cavity. The shear edge clearance shall be adjusted to approximately 0,05 mm.

For pressure measurement, piezoelectric sensors are normally used.

This mould may also be used for assessing the behaviour of thermosetting moulding compounds in accordance with method II of ISO 12114. In this case, a temperature sensor will have to be mounted near the centre of the mould and the middle pressure sensor relocated, as specified in ISO 12114.

5.2.3 Sensor output recording equipment, to record

- the mould closing force;
- the pressure at the centre and at both ends of the mould cavity;
- the movement of the top of the mould.

NOTE — The mould closing force should preferably be determined by measuring the hydraulic pressure.

The sensors and recording equipment shall be capable of operating over the following ranges:

- hydraulic pressure (mould closing force): 0 to 280 bar;
- mould cavity pressure: 0 to 150 bar;
- displacement of top of mould: 0 to 20 mm.

6 Sampling

For a sheet moulding compound, take a laboratory sample over the full width of the roll. Reduce the width by 5 cm on each side to avoid edge effects.

For a bulk moulding compound, take a laboratory sample from the centre of an elementary unit. When taking the laboratory sample, do not remove any protective sheet, and place the sample immediately after taking it, in a suitable bag to avoid loss of volatile matter or absorption of moisture.

The laboratory sample shall be of a size sufficient to enable the required number of test specimens to be prepared (see 8.1).

7 Conditioning

Condition the laboratory sample for 1 h at a temperature of $23\text{ °C} \pm 1\text{ °C}$.

8 Test specimens

8.1 General

For both methods, take three specimens from the conditioned laboratory sample.

NOTE — The product specification or the person ordering the analyses may require that the determination be performed on additional specimens or at specific places in the elementary unit or laboratory sample.

If test conditions are specified in the material specification, these conditions shall be used whenever possible.

8.2 Method I

8.2.1 Sheet moulding compound specimens

For each test specimen, cut one layer of the moulding compound laboratory sample to a minimum size of 45 mm in diameter or 50 mm square. Remove the cover sheet, and immediately cover the specimen with aluminium foil.

Take one specimen from the middle, one from the right and one from the left of the laboratory sample, to take account of the various machine areas.

8.2.2 Bulk moulding compound specimens

Place approximately 20 g of moulding compound between two sheets of aluminium foil and distribute it as evenly as possible over a circular area approximately 40 mm in diameter. Then compact the specimen evenly to a thickness of 3 mm. The surface of the specimen after compression shall be 45 mm in diameter or larger. The protective aluminium foil shall not be thicker than 20 µm.

8.3 Method II

8.3.1 Sheet moulding compound specimens

Remove the protective cover sheet and stack complete layers on top of one another. Cut the stack to a width of 200 mm and a length of either $140\text{ mm} \pm 10\text{ mm}$ or $280\text{ mm} \pm 10\text{ mm}$, using the tolerance of $\pm 10\text{ mm}$ to ensure that the specimens are of constant mass. The length and thickness of each test specimen shall be such that, after moulding, the thickness of the plate produced shall be approximately 4 mm. Select the length (140 mm or 280 mm) and the number of layers accordingly.

8.3.2 Bulk moulding compound specimens

Shape a suitable quantity of moulding compound by hand to give a test specimen measuring $200\text{ mm} \times 140\text{ mm}$ or $200\text{ mm} \times 180\text{ mm}$. Weigh each test specimen to produce a thickness of the plate of approximately 4 mm.

9 Procedure

9.1 Method I

9.1.1 Test temperature

Carry out the test at $23\text{ °C} \pm 2\text{ °C}$. Ensure that the apparatus is stable at this temperature by measuring the temperature in the test room to confirm that it is within the required tolerance limits.

9.1.2 Preparation of apparatus

Adjust the apparatus to give the load selected (see 5.1.1) and set the displacement-measuring sensor as specified in 5.1.2.

9.1.3 Test

Place a test specimen centrally under the punch. Apply the load and start the stopwatch, at the same time recording the punch height H_0 as indicated by the displacement indicator (see figure 1). The height H_0 defines the position of the punch at the beginning of loading and corresponds to the thickness of the specimen. It is the reference value for the determination of the percentage distance travelled by the punch as it descends. Record the punch height after 15 s (H_{15}) and after 45 s (H_{45}), or after longer periods if required.

9.2 Method II

Choose and record the test conditions. Adjust the controls on the moulding press accordingly. For normal testing, the temperature of the inside wall of the mould shall be $140\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$. In certain circumstances, however, other working temperatures may be required. If so, they shall be agreed between the interested parties and stated in the test report.

The closing force shall be chosen from the series of values given by the expression

$$30\text{ kN} + n\text{ }40\text{ kN}$$

where $n = 1, 2, 3, \dots$

so that the mould filling time is as close to 10 s as possible, and in any case within the range $10\text{ s} \pm 5\text{ s}$ (see 4.2).

Start the recording system.

Insert the specimen exactly into the centre of the mould cavity and immediately close the mould. The top of the mould shall come into contact with the test specimen within 5 s after insertion. If only flowability measurements are required, stop the recording system after about 20 s. Wait for the curing process to complete and open the mould.

10 Expression of results

10.1 Method I

Determine, from the curve recorded (see figure 5), the following parameters:

- The percentage decrease in specimen thickness (Q_t) for each punch height measurement, expressed as a percentage of the initial specimen thickness, calculated from the equation

$$Q_t = \frac{(H_0 - H_t)}{H_0} \times 100$$

where

H_0 is the initial specimen thickness;

H_t is the thickness at time t ($t = 15\text{ s}, 45\text{ s}$ or longer times if measured).

A value of Q_{45} less than 30 % or greater than 70 % shall be discarded and the test repeated using another load as appropriate (see 4.1).

- b) The non-Newtonian contribution DQ, due to the non-Newtonian behaviour of the moulding compound, which is calculated as a percentage from the equation

$$DQ = Q_{45} - Q_{15}$$

where

Q_{45} is the percentage decrease in specimen thickness at 45 s;

Q_{15} is the percentage decrease in thickness at 15 s.

- c) The mean value and standard deviation, calculated for each set of results obtained at the same load and same value of t .

10.2 Method II

Determine, from the curves recorded (see figure 2), the following parameters:

- a) The time of onset of flow t_0 , which corresponds to the time at which the pressure at the centre of the mould cavity begins to rise.
- b) The end of the mould filling time t_E , which is the time at which the pressure at the rim of the mould has reached 95 % of its final (stationary) value.
- c) The moulding-compound pressure P_M , which is the pressure at the centre of the mould cavity at time t_E .
- d) The initial specimen thickness H_0 , which corresponds to the reading of the displacement indicator in the mould closed position at time t_0 .
- e) The final specimen thickness H , which is the thickness of the moulded test specimen at time t_E .
- f) The pressure gradient, which is the difference between the pressure at time t_0 and the maximum pressure at the centre of the mould cavity, divided by the duration of the increase, and is calculated from the equation

$$PG = \frac{p_{\max} - p_{t_0}}{t_{\max} - t_0}$$

where

$p_{\max}$ is the maximum pressure at the centre of the mould cavity;

p_{t_0} is the pressure at the centre at time t_0 ;

$t_{\max}$ is the time of the pressure maximum;

t_0 is the time of onset of flow.

- g) The mould filling time t_F , calculated from the equation

$$t_F = t_E - t_0$$

- h) The pressure integral P , calculated for the pressure at the centre of the mould cavity over the interval between t_0 and t_E .

11 Precision

The precision of this method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added at the following revision.

12 Test report

The test report shall include at least the following information:

- a) a full description and identification of the moulding compound tested;
- b) a full description of the procedures used for sampling and for the preparation of the test specimens (in particular, for method II, the number of layers used for sheet moulding compound specimens and the length of such specimens, or the mass of bulk moulding compound specimens);
- c) the test conditions used;
- d) each individual result, their average value and the standard deviation:
 - 1) for method I
 - (i) the percentage decrease in specimen thickness,
 - (ii) the non-Newtonian contribution, in percent;
 - 2) for method II
 - (i) the mould filling time,
 - (ii) the moulding-compound pressure,
 - (iii) the initial specimen thickness and the final specimen thickness,
 - (iv) the pressure gradient,
 - (v) the pressure integral;
- e) any procedural details not mentioned in the standard that may have affected the results.