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Fibre-reinforced plastics — Methods of producing test plates —

Part 3:

Wet compression moulding

Plastiques renforcés de fibres — Méthodes de fabrication des plaques d'essai —

Partie 3: Moulage par compression voie humide



Reference number ISO 1268-3:2000(E)

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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 3.

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 part of ISO 1268 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 1268-3 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Together with the other parts (see below), this part of ISO 1268 cancels and replaces ISO 1268:1974, which has been technically revised.

ISO 1268 consists of the following parts, under the general title *Fibre-reinforced plastics* — *Methods of producing test plates*:

- Part 1: General conditions
- Part 2: Contact and spray-up moulding
- Part 3: Wet compression moulding
- Part 4: Moulding of prepregs
- Part 5: Filament winding
- Part 6: Pultrusion moulding
- Part 7: Resin transfer moulding
- Part 9: Moulding of glass mat thermoplastics (GMT)

Part 8: Compression moulding of SMC, BMC and DMC, is in preparation.

Fibre-reinforced plastics — Methods of producing test plates —

Part 3:

Wet compression moulding

1 Scope

This part of ISO 1268 specifies a method of making test plates by wet compression moulding. Using this method, test plates can be made in a reproducible manner, making it possible to compare the results of tests carried out at different times and/or in different places.

Test specimens cut from plates made by wet compression moulding are useful in characterizing the reinforcement used. The reinforcement can be either mats or fabrics. The following properties of these materials are of particular interest:

- water absorption (ISO 62);
- flexural strength and flexural modulus (ISO 178);
- impact strength (Charpy) (ISO 179);
- tensile strength, tensile modulus and elongation at break (ISO 527-4).

This part of ISO 1268 is intended to be used in conjunction with ISO 1268-1.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 1268. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 1268 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1172:1996, Textile-glass-reinforced plastics — Prepregs, moulding compounds and laminates — Determination of the textile-glass and mineral-filler content — Calcination methods.

ISO 1183 (all parts), Plastics — Methods for determining the density and relative density of non-cellular plastics.

ISO 1268-1, Fibre-reinforced plastics — Methods of producing test plates — Part 1: General conditions.

ISO 2555, Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method.

3 Health and safety

See ISO 1268-1.

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4 Principle

To make plates by wet compression moulding, a press is used with two flat mould plates. The lower plate is fixed and the upper plate can be pressed on to it. Reinforcement materials like mats or fabrics are placed on the lower plate and a sufficient amount of a suitable resin system is poured on top of the reinforcement. The upper plate is then pressed on to the lower plate. As a result, the resin system flows through the reinforcement. The distance between the plates can be adjusted by means of spaces, thus enabling the relative amounts of reinforcement and resin system to be varied. The plates can be either at ambient temperature or at a higher temperature, the temperature determining the time for the resin system to cure.

5 Materials

5.1 Reinforcement

The reinforcements shall be in the form of flat sheets that can be trimmed to the required size. In wet compression moulding, textile glass is usually used as the reinforcement.

It is very important that the layers are sufficiently strong to withstand the resin flow during compression. This means that the reinforcement must be relatively insoluble in the resin system.

5.2 Resin

An unsaturated polyester (UP) resin is normally used. It is essential that the viscosity is sufficiently high for compression moulding. Generally, a viscosity higher than 1 000 mPa·s is suitable (measured at 23 °C by the Brookfield method as described in ISO 2555). This can be achieved by adding filler to the UP resin, commonly 50 parts or more of filler to every 100 parts of UP resin. If necessary, a colorant can also be added. Furthermore, a suitable mould release system should preferably be added to the resin system, or otherwise a release agent should be applied to the mould.

An accelerator should preferably be added to the mixture of resin and filler, followed by an initiator. The curing system comprising the accelerator and initiator should be chosen in such a way that the resulting resin system has a sufficiently long pot-life. When the plates are at ambient temperature, the pot-life should be long enough to allow the resin system to be poured on to the reinforcement and to flow through the reinforcement. After producing several test plates, the temperature in the mould will stabilize at a higher temperature (normally between 30 $^{\circ}$ C and 60 $^{\circ}$ C) due to the exothermic reaction. This enables the pot-life to be lengthened since the geltime during compression is shortened by the higher temperature.

6 Plate dimensions

The moulded plates shall be either circular or square. Recommended dimensions are a diameter of 300 mm for circular plates and $300 \text{ mm} \times 300 \text{ mm}$ for square plates. In both cases, the recommended thickness of the moulded plate is 4 mm. These dimensions allow the measurement (in one direction) of tensile properties, flexural properties, impact strength and water absorption.

Other dimensions may also be used. However, it shall be borne in mind that the minimum thickness is determined by the reinforcement layers. Plates should preferably include several reinforcement layers so that compensation is made for any lack of homogeneity in any individual reinforcement layer.

7 Reinforcement content

The preferred reinforcement content depends on the reinforcement type (mat or fabric). Furthermore, the value of the reinforcement content expressed as a percentage of the total mass will depend, in addition, on the amount of filler in the resin (more filler will increase the density of the resin).

For reinforcements that consist only of mats, the reinforcement content should preferably lie in the range 20 % to 40 % by mass. For reinforcements consisting of fabrics or other multiaxial reinforcements, the target range is 40 % to 60 %. For other reinforcement types, the range of reinforcement contents will greatly depend on the structure of the reinforcement.

8 Apparatus

8.1 Mould

A pair of mould plates that form two parallel planes shall be used. Because this process does not require very high pressures, a mould of relatively light construction can be used. Generally, pressures in the range 1 bar to 10 bar are sufficient. To produce test plates of a specific thickness, the mould needs to be provided with spacers. The rigidity of the two mould plates shall be high enough to ensure that the faces of the test plates do not deviate from parallel by more than \pm 0.3 mm.

8.2 Press

The mould plates shall be bolted to a press. One plate (usually the lower) shall be fixed to the press frame; the other (upper) plate shall be fixed to the piston of the press so that it can be moved vertically. The piston should preferably have a stroke of at least 500 mm and a speed which is adjustable between at least two levels: "rapid", which should lie in the range 25 mm/s to 250 mm/s, and "compression", which should lie in the range 0,2 mm/s to 2 mm/s.

The press shall be capable of applying a high enough compression force. A pressure range of 1 bar to 10 bar will usually be sufficient to apply the force needed for the recommended test plate dimensions.

9 Procedure

Cut the reinforcement layers to size. The number of layers of reinforcement shall be such that the desired reinforcement content is achieved for a given test plate thickness. The number of layers required, n, can be calculated using the following equation:

$$n = \frac{e\rho_f \rho_m b}{g \left[b\rho_m + \rho_f \left(\mathbf{1} - b \right) \right]}$$

where

e is the thickness of the plate, in centimetres;

 ρ_f is the density of the reinforcement, in grams per cubic centimetre;

 ho_m is the density of the resin system, in grams per cubic centimetre;

b is the reinforcement content by mass, expressed as a fraction;

g is the mass per unit surface area of the reinforcement, in grams per square centimetre.

Densities should preferably be obtained from the manufacturer. However, if this is not possible, determine them by one of the methods described in ISO 1183.

Next prepare the resin system. Calculate the amount of resin system required from the desired reinforcement content of the plate, allowing an excess of 0 % to 10 % for the resin which will flow out of the reinforcement. The following equation may be used:

$$m = \left[\left(1 - b \right) / b \right] ngA \left(1 + x \right)$$

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where

- m is the mass of resin system required, in grams;
- b is the reinforcement content by mass, expressed as a fraction;
- n is the number of layers of reinforcement;
- g is the mass per unit surface area of the reinforcement, in grams per square centimetre;
- A is the surface area of a layer of reinforcement, in square centimetres;
- *x* is the excess of resin system used, expressed as a fraction.

Open the press and place the reinforcement layers on the lower plate. Pour the resin system on to the middle of the reinforcement and close the press. The first stage in closing the press shall be as quick as possible. During the last millimetres, however, the closing speed shall be reduced significantly. The quality of the test plate will depend partly on the final closing speed and the length of this part of the stroke. Therefore, this part of the closing operation may need optimization in order to obtain a good plate. The excess of resin x may also need to be varied to optimize plate quality.

When the mould is closed, the time to cure the resin in the mould (which may or may not be pre-heated), will be a matter for optimization. Curing at ambient temperature will require longer times, and the pot-life of the resin system will have to be adjusted accordingly.

Because this technique does not give test plates with very well-defined edges, they should preferably be trimmed using a diamond saw. Trimming should be done in such a way that plate edges are removed in those places in which it is not certain that all the reinforcement layers are present. Trimming should result in a plate with a uniform reinforcement distribution.

10 Verification of the characteristics of the plate obtained

10.1 Fibre content

Because a filler is generally present in the resin system, the reinforcement content by mass of the plate should preferably be determined by a calcination method. For glass-fibre reinforcement, use the procedure described in ISO 1172.

10.2 Appearance and impregnation

After moulding, the visual appearance and quality of impregnation of the plate shall be investigated to confirm that the laminate is of suitable quality.

10.3 Dimensions of the plate

Because the width and diameter of the plate are not well defined with this technique (the plate is trimmed after moulding), it is not necessary to measure these dimensions. On the other hand, the thickness of the plate shall be measured. Making measurements at different locations gives an indication of the thickness uniformity of the plate and of the mean thickness produced by the spacers used.

11 Test plate preparation report

The test plate preparation report shall include the following information:

- a) a reference to this part of ISO 1268;
- b) the place and date of production of the test plate;
- c) details of the number of layers (plies), the stacking sequence and orientation of the layers;
- d) a description of the materials used (including type of reinforcement, type of resin, type of filler, if applicable, catalyst curing system, etc.);
- e) a description of the equipment used (mould, etc.);
- f) the operating conditions (moulding pressure, moulding temperature, closing speed, etc.);
- g) the thickness of the test plate produced;
- h) the fibre content and filler content, if applicable;
- i) the quality of the plate (appearance, impregnation);
- j) any other information needed to reproduce the specimens exactly;
- k) any deviations from this part of ISO 1268.

Bibliography

- [1] ISO 62, Plastics Determination of water absorption.
- [2] ISO 178, Plastics Determination of flexural properties.
- [3] ISO 179 (all parts), Plastics Determination of Charpy impact properties.
- [4] ISO 527-4, Plastics Determination of tensile properties Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites.

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