INTERNATIONAL STANDARD

ISO 1268-4

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Fibre-reinforced plastics — Methods of producing test plates —

Part 4: Moulding of prepregs

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

Partie 4: Moulage des préimprégnés

Reference number
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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1268-4 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

This first edition cancels and replaces ISO 9353:1991, which has been technically revised. It considers input from prEN 2565, EN 2374 and ASTM D 5687.

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 8: Compression moulding of SMC and BMC*

— *Part 9: Moulding of GMT/STC*

— *Part 10: Injection moulding of BMC and other long-fibre moulding compounds — General principles and moulding of multipurpose test specimens*

— *Part 11: Injection moulding of BMC and other long-fibre moulding compounds — Small plates*

Fibre-reinforced plastics — Methods of producing test plates —

Part 4: Moulding of prepregs

1 Scope

This part of ISO 1268 describes the preparation of test plates from layers of preimpregnated unidirectional fibre or fabric (prepregs) under pressure and temperature in various types of equipment (for example, autoclave, bladder press, hydraulic press or vacuum bag equipment). It applies to all reinforcements and resins.

This method is applicable to reinforcements preimpregnated either with a partially cured thermosetting resin or with a thermoplastic resin. The test plate is formed by stacking layers of the preimpregnated material in the required sequence and orientation, followed by compaction and final consolidation under pressure/vacuum at a temperature above ambient. The prepared test plates are subsequently machined into the required test specimens.

Standard plates prepared in this manner may be used either for evaluating the components, i.e. the reinforcement, finish, resin, etc., or for verifying the overall quality of the finished product.

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.

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 1172, *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 of non-cellular plastics*

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

ISO 2818, *Plastics — Preparation of test specimens by machining*

ISO 7822, *Textile glass reinforced plastics — Determination of void content — Loss on ignition, mechanical disintegration and statistical counting methods*

3 Health and safety

See ISO 1268-1.

4 Principle

Standard plates, intended for the preparation of test specimens, are produced from fibre-reinforced prepregs by cutting the required number of layers to size and stacking them in the required sequence and orientation. The stack of prepregs is initially consolidated and trapped air removed using mechanical compaction and/or an applied vacuum. The assembled stack, normally sealed in an evacuated vacuum bag, is then finally consolidated using one of several possible combinations of heat and pressure depending on the equipment used and the material supplier's processing instructions. Suitable processing routes include using an autoclave, a pressclave, an applied vacuum only or a hydraulic press.

Plates with flat surfaces are prepared unless the effect of surface finish is being studied. They have to be of sufficient size to cover the maximum specimen size required in subsequent testing.

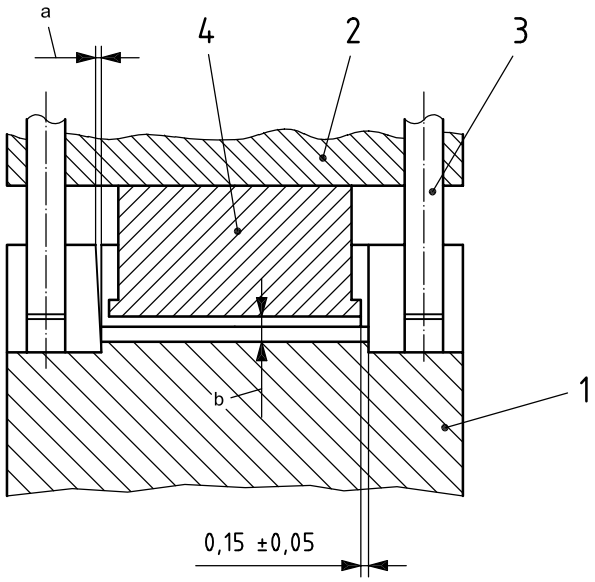
5 Apparatus

5.1 Processing equipment

5.1.1 **Press**, of any type, with the components specified in 5.1.1.1 to 5.1.1.4.

5.1.1.1 **The press itself** (see Figure 1), consisting of a frame, ram and base. The height of the frame shall be large enough to provide a moulding chamber where the mould containing the prepreg stack can be inserted in one operation. A gap between the ram and the frame of at least 0,20 mm shall be ensured by means of appropriately constructed guides.

Dimensions in millimetres



Key

1 base
2 upper frame
3 columns
4 ram

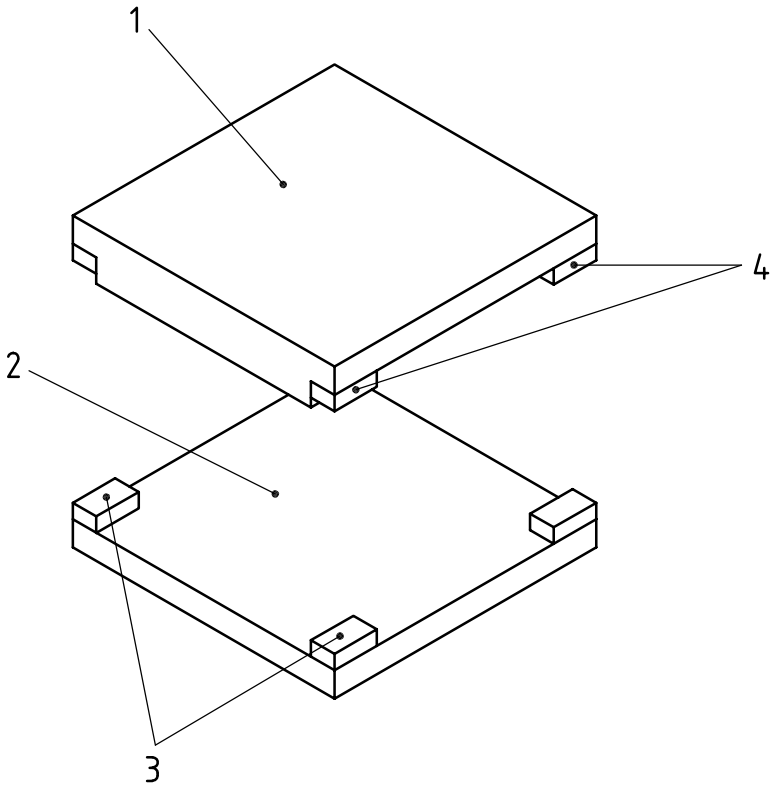
a 1° draft all round.
b All-around pinch-off.

Figure 1 — Moulding press

5.1.1.2 Open-sided mould (see Figure 2), consisting of two flat metal plates (a base plate and a cover plate) with spacers at the four corners controlling the thickness of the moulded plate. The dimensions of the plates shall be such that test specimens of the required size can be cut from the test plates produced using the mould. The surfaces of the mould plates facing the mould cavity shall be flat to within 0,05 mm and shall be polished or hard chrome plated. Suitable mould plate thicknesses are 5 mm for steel and 6 mm for aluminium alloy.

NOTE 1 A test plate of specific thickness can be obtained by placing suitably sized spacers between the mould plates at the corners of the mould.

NOTE 2 To aid recognition of the zero-degree direction of the test plate, an "arrow" can be engraved in the surface of the baseplate. Care is necessary, however, that the "arrow" moulded into each test plate does not affect the properties of the test specimens prepared from the plate. Alternatively, non-square test plates can be moulded (e.g. 350 mm long × 300 mm wide) with the zero-degree direction parallel to the longer side of the plate.



Key

1 cover plate
2 base plate
3 guidepieces
4 spacers of suitable thickness

Figure 2 — Mould

5.1.1.3 Means of applying the required moulding pressure, or executing the required force-time profile, with an accuracy of 5 %, over the required period of time.

5.1.1.4 Temperature measurement and control equipment, capable of ensuring a heat-up rate of at least 3 °C/min and maintaining the required curing temperature between the specified limits, or executing the required temperature-time profile.

5.1.2 Autoclave, of any dry-heat type, meeting the requirements of 5.1.1.3 and 5.1.1.4.

5.1.3 **Ventilated oven**, meeting the requirements of 5.1.1.4.

5.1.4 **Ruler**, for measuring the length and width of the test plate to the nearest 0,5 mm.

5.1.5 **Micrometer screw gauge**, for measuring the thickness of the test plate to the nearest 0,01 mm.

5.1.6 **Balance**, capable of weighing to the nearest 0,01 g.

5.1.7 **Cutting device**, such as a knife, with a sharp blade.

5.18 **Vacuum pump**, capable of producing a vacuum of 0,08 MPa or better.

5.1.9 **Supply of compressed air**, capable of applying a pressure of 0,7 MPa $\pm$ 2 %.

5.2 Auxiliary apparatus and materials, as required

5.2.1 **Rubber seals**, of a shape and size such that they can be placed round the test plate in the mould, and resistant to a temperature at least 20 °C higher than the curing temperature.

5.2.2 **Release film**, resistant to a temperature at least 20 °C higher than the curing temperature, made of a material such as poly(vinyl fluoride) (PVF), polytetrafluoroethylene (PTFE) or PTFE-coated fabric.

5.2.3 **Perforated release film**, resistant to a temperature at least 20 °C higher than the curing temperature, made of a material such as PVF, PTFE or PTFE-coated fabric.

5.2.4 **Flexible pressure blanket**, resistant to polymerization products and resistant to a temperature at least 20 °C higher than the curing temperature, made of a material such as PVF, PTFE or PTFE-coated fabric.

5.2.5 **Breather material**, such as aluminium gauze or glass-fibre fabric.

5.2.6 **Absorbent material**, for absorption of excess resin, e.g. woven glass-fibre fabric.

NOTE Woven glass-fibre fabrics having a mass per unit area of 100 g/m^2 and 300 g/m^2 are capable of absorbing approximately 60 g and 115 g of resin, respectively, per square metre. Polyamide-fibre fabric having a mass per unit area of 60 g/m^2 is capable of absorbing approximately 40 g of resin per square metre.

5.2.7 **Metal edge strips**, of a suitable length and a width of 15 mm, for placing round the test plate in the mould. The thickness of the strips will depend on the thickness of the test plate to be produced.

5.2.8 **Sealing tape**, resistant to a temperature at least 20 °C higher than the curing temperature.

6 Procedure

6.1 Condition the materials to be used for preparing the test plates, including sufficient prepreg material, for at least 2 h in one of the standard atmospheres specified in ISO 291. Carry out subsequent preparation of the laminate stack (see 6.4) in the same atmosphere.

6.2 If the material has been stored at a temperature lower than the conditioning temperature, keep it in an airtight bag to prevent moisture pick-up until it reaches the conditioning temperature.

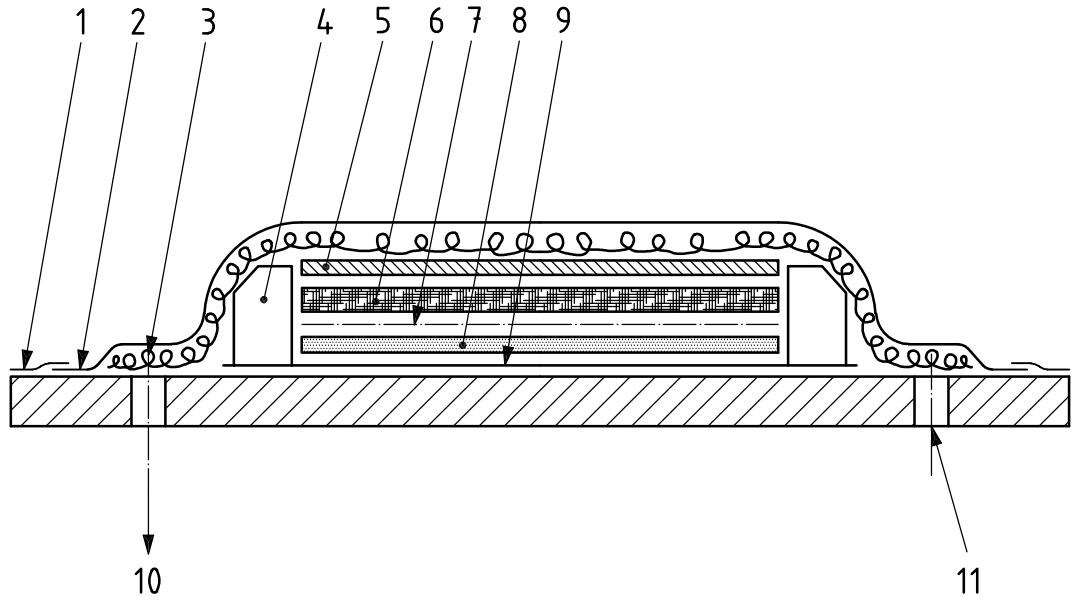
6.3 After conditioning, cure thermoset-based material within 6 h, unless otherwise specified.

6.4 From the conditioned prepreg material, cut the number of layers needed to produce a cured test plate of the required length, width and thickness, cutting each layer at the orientation required by the lay-up sequence given in the specification or test method (see Annex A). Stack the cut layers of prepreg on the base plate of the mould in the required sequence.

Insert a thermocouple into the edge of the stack for temperature control of the moulding process. The disposition of the laminate stack and auxiliary materials typically used for autoclave processing is shown in Figure 3 for the preferred test plate with flat faces. If the effect of surface finish is being studied, replace the upper layer of perforated release film by the material relevant to the effect under study. Figure 4 shows the disposition of the laminate stack and auxiliary materials used in various types of bladder press.

NOTE 1 The number of layers of absorbent material (5.2.5) used to absorb excess resin will depend on the resin content required for the cured test plate. The plate thickness and resin content are also a function of pressure, temperature and other factors depending on the properties of the fibre/resin system used (see Note 2).

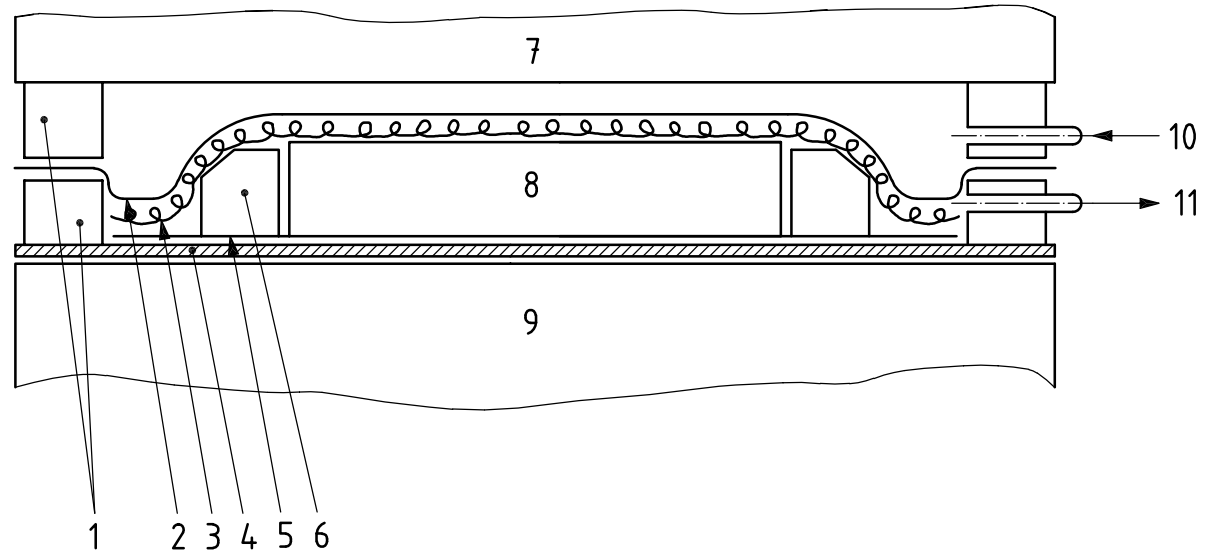
NOTE 2 It may be necessary to carry out preliminary experiments to determine the number of layers of prepreg material and the number of layers of absorbent material required, at a given pressure, to obtain cured plates of the required thickness and fibre content. For low-bleed systems, the nominal ply thickness can be used to determine the number of layers of prepreg material needed.



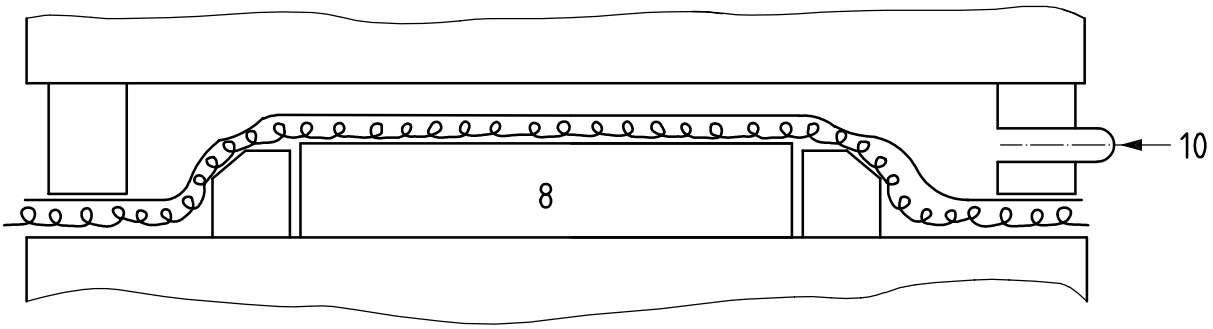
Key

1 sealing tape (5.2.7)
2 pressure blanket (5.2.4)
3 breather material (5.2.5)
4 metal edge strip (5.2.6)
5 mould cover plate
6 absorbent material (5.2.5)
7 perforated release film (5.2.3)
8 laminate lay-up
9 release film (5.2.2)
10 ventilation to atmosphere outside autoclave
11 port for connections to e.g. temperature and/or pressure sensors

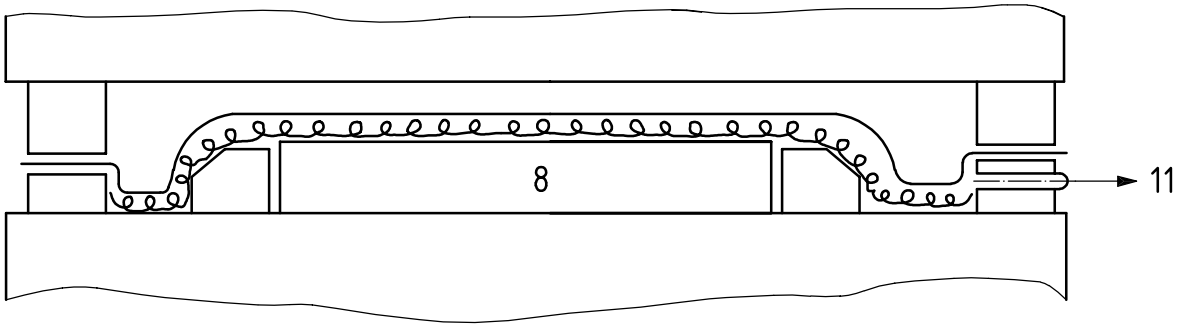
Figure 3 — Typical test plate assembly for moulding in an autoclave



a) Overpressure/vacuum method



b) Overpressure method



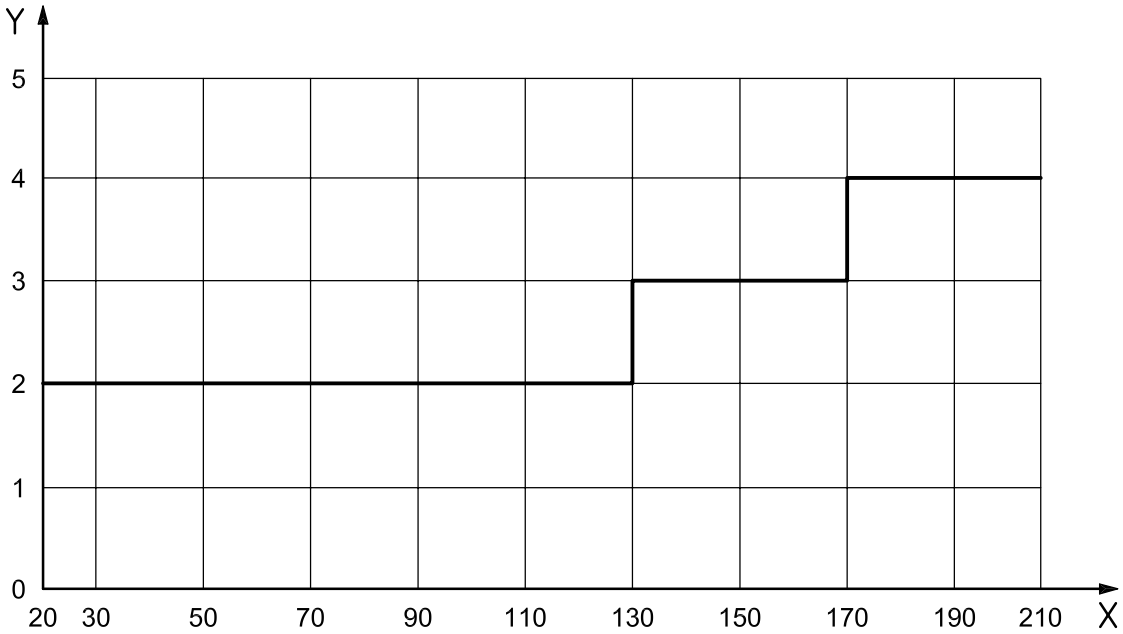
c) Vacuum method

Key

1	metal edge strips (5.2.6)	7	mould cover plate
2	pressure blanket (5.2.4)	8	lay-up as in Figure 3
3	breather material (5.2.5)	9	lower platen of press
4	mould base plate	10	compressed-air supply
5	release film (5.2.2)	11	to vacuum
6	rubber seal (5.2.1)		

Figure 4 — Schematic examples of moulding in a bladder press

6.5 The temperature, pressure and time of curing shall be as specified in the material data sheet or determined by agreement between the interested parties, and will depend on the type of resin and curing agent. The temperature stipulated shall be maintained during the curing cycle, i.e. the temperature indicated by the temperature measurement equipment shall remain within the range required for the resin system used (see Figure 5). The temperature at any point on the surface of the test plate during moulding shall not differ by more than ± 2 °C from the value indicated by the temperature measurement equipment.



Key

X curing temperature, °C

Y permitted variation in temperature, °C

Figure 5 — Examples of permissible variations in curing temperature

6.6 After completion of the consolidation process, remove the test plate from the press or autoclave and cool it, if necessary, in such a way that deformation, damage, etc., is avoided.

6.7 The fibre orientation(s) relative to the length direction of the plate shall be indicated on a pressure-sensitive paper tag applied to the plate or by some other suitable means agreed between the interested parties (see Note 2 to 5.1.1.2).

6.8 Trim off and discard at least 10 mm from the edges of the test plate.

6.9 Where no other treatment is specified, the plates may be used, in this condition, to produce test specimens. Some guidance on machining may be obtained from the general standard on plastics machining, ISO 2818.

If not specified in the relevant test method standard, the type of specimen cut from the test plate, the size of the specimens and their orientation with respect to the orientation of the reinforcement within the plate shall be stipulated by a separate agreement.

7 Determination of the quality of the test plates

7.1 Weigh the trimmed plate to the nearest 0,1 g.

7.2 Using a ruler (5.1.4), measure the length and width of the trimmed plate across each end and along each side to an accuracy of 0,5 mm. Calculate the arithmetic mean of each pair of measurements and round to the nearest millimetre.

7.3 Using a micrometer screw gauge (5.1.5), measure the thickness at each of the four corners (but not closer than 25 mm to the edges) and in the centre to an accuracy of 0,05 mm. Calculate the arithmetic mean of the five measurements and round to the nearest 0,1 mm.

7.4 Determine the uniformity of the porosity and of other defects in the plate by a non-destructive method (e.g. C-scan ultrasonic inspection), using a procedure agreed on between the interested parties (if required).

7.5 Use only uniform and otherwise acceptable areas for cutting out test specimens.

7.6 If required to measure the fibre content, void content and density, take a test specimen with the dimensions (20 × 10) mm × the thickness of the plate from each of two diagonally opposite corners of the plate.

a) Determine the density of these specimens by one of the methods specified in the various parts of ISO 1183.

b) For both test specimens, determine the fibre content by volume and by mass in accordance with ISO 1172 for plastics reinforced with glass fibre or other inert fibres. For carbon-fibre-reinforced plastics, use a method suitable for this reinforcement as agreed between the interested parties.[1)]

c) Determine the void content in accordance with ISO 7822.

8 Precision

See Annex B.

9 Test plate preparation report

The report of preparation of the test plate shall include the following:

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 the orientation of the layers;

d) a description of the materials used, including the batch and roll number of the reinforcement, the nature and type of resin, the nature and type of fibre and the nature of the finish;

e) a description of the apparatus used (type of press, type of autoclave, type of mould, method of checking temperatures and pressures, etc.);

f) details of the working procedure (the moulding pressure or force-time profile, the temperature-time profile, the post-cure time and temperature, etc.);

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1) For carbon-fibre composites, an International Standard is in preparation (ISO 14127, *Carbon-fibre-reinforced composites — Determination of the resin, fibre and void contents*).

g) the mass, in grams, of the plate;

h) the length, width and thickness of the plate (individual measurements and average value, in millimetres);

i) the fibre content of the plate (when measured) (individual measurements and average value, in percent by volume and percent by mass);

j) the void content of the plate (when measured) (individual measurements and average value, in percent by volume);

k) the uniformity of the plate (when measured), as shown by a non-destructive test method, noting the method used;

l) any deviations from this part of ISO 1268.

Annex A
(normative)

Stacking designation system

A.1 Introduction

The purpose of a laminate stacking designation system is to provide a method of describing the lay-up of a laminate based on unidirectional (UD) or fabric-reinforced layers. Use of the designation will avoid confusion, and also avoid the manufacture and testing of incorrect test plates.

A.2 Reference plane and direction

A reference plane and direction are selected before preparing the designation, as follows:

— The reference plane is normally designated as the bottom layer of the laminate. The top layer can also be used, but if so this shall be indicated in the designation.

— The reference direction, sometimes known as the zero-degree (0°) direction, is usually the principle fibre direction in the reference plane or the principle fibre direction in the laminate as a whole. This direction is designated 0°. Positive angles are measured anticlockwise when viewed looking towards the reference plane (i.e. from above for a bottom reference plane).

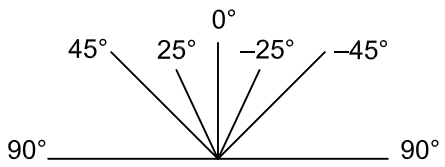

Figure A.1 — Angles to be used in describing ply orientation in the stack

A.3 Designation parameters

The designation has the following general form:

$$X\text{:}\left[\left(\theta_1 m_1 b_1 / \theta_2 m_2 b_2\right)_n\right]_{s\,L}$$

where

X (optional)	indicates the total number of plies (see Clause A.4).
Subscripts 1, 2, etc.,	are used to indicate to which particular ply (or set of plies) an orientation θ, material type b and number of plies m applies.
θ	is the ply orientation, in degrees, with respect to the reference direction. It is expressed as an angle between $\pm$ 0° and $\pm$ 90° (see Figure A.1). For UD tapes, the ply orientation is the direction of the fibre. For fabrics, the ply orientation is the warp direction of the fabric. For special (e.g. multiaxial) fabrics, the ply orientation shall be defined in the note explaining the designation indicating the types of material used (see "b" below). For fabrics, a negative sign shall be used when the absence of a sign would lead to the incorrect assumption that the angle is positive.

m	is the number of plies of a particular orientation stacked together.
b	is the designation of the type of material (e.g. b_x, b_{x+1}, etc.) where different materials are used (different fibre types and/or different fibre formats). An explanation is given in a note.
n	is a number designating the number of plies, or sets of plies, stacked sequentially with the same sequence of orientations. Such sets are enclosed in parentheses.
s	is used as a subscript to indicate geometrical symmetry about the mid-plane (i.e. when the plies in one half of the laminate are repeated as a mirror image in the other half).
f	is used as a subscript to indicate a fabric, or this can be indicated by a note (see "b" above). Unidirectional fibre plies do not have this subscript.
L	indicates further instructions not related to the ply orientation but to ply disposition, such as "flip-flop", "staggered", "wrap face", "nested", etc. These shall be fully described in attached notes.

A.4 Compilation of designation

The stacking designation system is compiled as follows:

— the orientations of the plies are listed from left to right in the designation, starting at the reference plane and proceeding to the opposite side of the laminate;

— a forward slash is inserted between each ply (e.g. 0/90/0);

— parentheses are used to enclose a set of plies of a given type stacked together, followed by the number of plies of this type (m) or, for a repeating sequence of plies, by the number of times this sequence is repeated (n);

— square brackets are used to enclose the full designation;

— the subscript "s" is added after the second square bracket if the designated lay-up is repeated in the reverse order after the mid-plane (i.e. if the laminate is symmetrical about the mid-plane);

— a single central ply not repeated in an otherwise symmetrical lay-up is indicated by over-scoring the symbol representing that layer or by a backslash (\) following the symbol representing the layer.

Optionally, the total number of layers (X) (unidirectional plies and fabric layers each counting as one unit) can be given at the beginning of the designation, followed by a colon.

A.5 Computer codes

For computer codes, where subscripts cannot be used, subscripts shall be preceded by colons.

A typical designation in computer code is given below:

Standard	Reference plane	No. of plies	Stacking sequence	Note(s), if any
ISO 1268-4,	bottom plane,	7:	[45:2b1/(90/0\):b2]:s	b1 = glass woven fabric b2 = UD glass tape

which corresponds to the full description [45_f/45_f/90/0/90/45_f/45_f].

A.6 Examples

Top	0	Reference plane — bottom Designation = $[0/90]_S$ Full description = [0/90/90/0] Computer code = [0/90]:s
	90	
S-	90	
Bottom	0	
Top	0	Reference plane — bottom Designation = $[0/\overline{90}]_S$ Full description = [0/90/0] Computer code = [0/90\]:s
S-	90	
Bottom	0	
Top	0	Reference plane — bottom Designation = $[-45_f/0/90/0]$ Full description = $[-45_f/0/90/0]$ Computer code = [–45:f/0/90/0]
	90	
	0	
Bottom	–45 fabric	
Top	+45	Reference plane — bottom Designation = 9: $[(\pm 45)_2/\overline{0}]_S$ Full description = [45/–45/45/–45/0/–45/45/–45/45] Computer code = 9: [(45/–45):2/0\]:s
	–45	
	+45	
	–45	
S-	0	
	–45	
	+45	
	–45	
Bottom	+45	
Top	45 fabric	Reference plane — bottom Designation = $[45_{f6}]_L$ (L= Flip-flop disposition) Full description = $[45_f/45_f/45_f/45_f/45_f/]_L$ (L= Flip-flop disposition) Computer code = [45:f6]:L
	45 fabric	
	45 fabric	
	45 fabric	
	45 fabric	
Bottom	45 fabric	
Top	45	Reference plane — bottom Designation = $[(45/0/-45/90)_2]_s$ or $[45/0/-45/90]_{2s}$ Full description = [45/0/–45/90/45/0/–45/90/90/–45/0/45/90/–45/0/45] Computer code = [(45/0/–45/90):2]:s or [45/0/–45/90]:2s
	0	
	–45	
	90	
	45	
	0	
	–45	
S-	90	
	90	
	–45	
	0	
	45	
	–45	
	90	
	0	
Bottom	45	

Annex B
(informative)

Precision

The precision data in Table B.1 are for a single batch of low-bleed carbon-fibre/epoxy prepreg, delivered direct to eight sites. Each site manufactured test plates in accordance with prEN 2565 (in preparation) at three thicknesses using autoclaves under the time/pressure conditions specified by the material manufacturer. Specimens were machined and tested using a range of mainly QA test methods at a single site. [4]

Table B.1 — Precision data for test specimens taken from plates manufactured using autoclaves at eight different sites

Property	Panel thickness	Mean value	Repeatability r	Standard deviation of r/mean %	Reproducibility R	Standard deviation of R/mean %
Fibre mass fraction	1 mm	69,3 %	2,44 %	1,4	8,44 %	4,4
	2 mm	67,1 %	5,43 %	2,5	6,54 %	3,5
	5 mm	69,2 %	3,41 %	1,5	4,35 %	2,2
ILSS (ISO 14130)	2 mm	104 MPa	10,0 MPa	3,4	22,4 MPa	7,6
Flexural testing (ISO 14125)	2 mm					
E_{11}		122 GPa	6,81 GPa	2,0	31,3 GPa	9,1
E_{22}		7,96 GPa	0,58 GPa	2,6	1,89 GPa	8,5
s_{11}		1 780 MPa	246 MPa	4,9	321 MPa	6,4
s_{22}		151 MPa	39,9 MPa	9,4	56,0 MPa	13,2

Bibliography

[1] EN 2374, *Aerospace series — Glass fibre reinforced mouldings and sandwich composites — Production of test panels*

[2] prEN 2565, *Preparation of carbon fibre reinforced resin panels for test purposes*

[3] ASTM D 5687, *Standard Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation*

[4] ISO 14125, *Fibre-reinforced plastic composites — Determination of flexural properties*

[5] ISO 14130, *Fibre-reinforced plastic composites — Determination of apparent interlaminar shear strength by short-beam method*

[6] SIMS, G.D., *Validation results from VAMAS and ISO round-robin exercises*, 10th International Conference on Composite Materials, Canada, 1995

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