UDC 678.5/.8.067 – 419.8 : 620.11

Ref. No. : ISO/R 1268-1970 (E)

Revised

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1268

PLASTICS

PREPARATION OF GLASS FIBRE REINFORCED, RESIN BONDED, LOW-PRESSURE LAMINATED PLATES OR PANELS FOR TEST PURPOSES

1st EDITION

April 1970

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 1268, *Plastics – Preparation of glass fibre reinforced, resin bonded, low-pressure laminated plates or panels for test purposes,* was drawn up by Technical Committee ISO/TC 61, *Plastics,* the Secretariat of which is held by the American National Standards Institute (ANSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1268 which was circulated to all the ISO Member Bodies for enquiry in May 1967. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	India	Spain
Austria	Iran	Sweden
Belgium	Israel	Switzerland
Bulgaria	Japan	Turkey
Canada	Korea, Dem. P. Rep. of	U.A.R.
Czechoslovakia	Korea, Rep. of	United Kingdom
France	Netherlands	U.S.A.
Germany	Poland	Yugoslavia
Greece	Romania	
Hungary	South Africa, Rep. of	

The following Member Body opposed the approval of the Draft :

Italy

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1970, to accept it as an ISO RECOMMENDATION.

ISO Recommendation | R 1268 | April 1970

PLASTICS

PREPARATION OF GLASS FIBRE REINFORCED, RESIN BONDED, LOW-PRESSURE LAMINATED PLATES OR PANELS FOR TEST PURPOSES

1. SCOPE

This ISO Recommendation describes the preparation of test plates or panels by bonding glass cloth or mats with low-pressure thermosetting resins. In this ISO Recommendation the term "low-pressure thermosetting resins" relates to resins which cure at or above room temperature either without loss of reaction products or which are not significantly altered by loss of reaction products during cure and which can be processed with a pressure up to 3.5 MN/m^2. The purpose of this ISO Recommendation is to standardize the preparation of the plates or panels from which test specimens subsequently are machined. Standard specimens prepared in this manner may be used either for evaluating the components; i.e. the glass reinforcement, finishes, resins, catalysts, curing agents, etc., or for verifying the overall quality of the finished "comprehensive" products.

2. PRINCIPLE

Preparation of the plates or panels by one of the following methods :

2.1 Method A

The glass reinforcement impregnated with liquid resin containing a suitable catalyst or curing agent is moulded in a press under conditions of temperature and pressure appropriate for the resin and curing system.

2.2 Method B

The glass reinforcement impregnated with resin partially cured to the B stage (prepreg), but capable of further curing, is moulded in a press at a mould temperature higher than room temperature.

2.3 Method C

The glass reinforcement is manually laminated or laid up with simultaneous impregnation with a resin containing a suitable catalyst or curing agent. The curing of the resin proceeds at room temperature or at elevated temperature. No press is required for preparation of the laminate.

3. EQUIPMENT FOR THE MAKING OF PLATES OR PANELS BY METHODS A AND B

3.1 Press

For moulding the plates or panels, use may be made of any hydraulic or mechanical press which permits

(*a*) clamping the mould as specified in clause 3.2;

(*b*) maintaining the moulding force with an accuracy of ± 5 % over the period of time required for the curing of the resin;

(*c*) maintaining the closing speed of the mould in accordance with the requirements of clause 4.1.4. Generally the closing speed should be low and adjustable.

3.2 Mould

3.2.1 One of two types of moulds is chosen, depending on

(*a*) the nature of the glass reinforcement;

(*b*) the type of resin and curing system;

(*c*) the purpose of testing.

3.2.2 The mould, type 1 (see Fig. 1) or type 2 (see Fig. 2), should comply with the following conditions, but in other respects the design is optional :

(*a*) the working surface of the mould should be a square, the dimensions of which are within the limits specified for types 1 and 2 (between 150 mm × 150 mm and 400 mm × 400 mm);

(*b*) the active surfaces should be hardened, ground and hard-chromium plated; the active surface of the lower die should bear a mark which, when impressed on the moulded plate or panel, will identify the surface formed by the lower die. Care should be taken to ensure that such marks do not influence the results of subsequent testing (types 1 and 2, see Note below);

(*c*) the type 1 mould is a two-part (open type) mould consisting of two plane parallel plates separated at constant distance during the moulding operation by means of spacers. This type of mould ordinarily is used for specification purposes;

(*d*) the type 2 mould is a three-part positive type consisting of a removable chase (frame, for example F, in Fig. 2) and an upper and lower die. The cavity should have such dimensions that the material required for the prescribed plate thickness can be introduced in a single charge. The tool should be provided with suitable guiding (for example K in Fig. 2) so that a clearance, b, of 0.15 ± 0.05 mm can be maintained around the perimeter of the punch. It is recommended that the inner walls of the frame be tapered as shown in Figure 2, where $c = 6$ mm and $\alpha = 1°$;

(*e*) the temperature of any spot of the active surface of the mould should differ by not more than ± 2 °C from the value indicated by the temperature check sensitive element.

NOTE. – The two main surfaces of the moulded plate or panel are not exactly equivalent with regard to all of their properties. During the period between filling and closing of the mould the lower surface of the test plate is heated longer and to a higher temperature than the upper surface. It may therefore be advisable in preparing test methods, specifications, property schedules, etc. to state the particular surface to which specifications or properties should be referred.

4. WORKING PROCEDURES

4.1 Method A

4.1.1 The glass reinforcement should be shorn, cut or chopped to rectangles, the width and length of which are 3 to 6 mm less than the dimensions of the cavity of the tool.

4.1.2 The glass reinforcement thus prepared should be dried at 80 °C for a period of 1 hour, except in the case of sized or finished glass reinforcements requiring lower temperature. After drying, it is cooled for 1 hour in a desiccator over a drying agent and then impregnated.

4.1.3 The laminate is prepared at a relative humidity of 65 % unless otherwise agreed. The impregnation of the glass reinforcement with a resin containing the required catalyst or curing agent may be conducted outside the mould on a suitable foil or film (placed on a heated support pad, if necessary) by freely spreading the resin with a spatula on individual layers of the glass reinforcement, or the impregnation can be carried out in the mould cavity. An excess of at least 20 % of resin as compared with the resin content of the cured product (in the finished plate) is consumed in making the plate. After lamination of the required number of layers, the impregnated glass reinforcement is placed in the cavity of the mould and heated to curing temperature.

Unless otherwise specified, not more than 50 % of the gel time of the resin at the temperature of the impregnation may elapse before the charge is inserted in the mould. Unless otherwise specified, mould release agents facilitating the removal of the moulded plates may be used only if those agents are not soluble in the resin and if such substances have been proved to exert no influence on the properties of either the plate or the test specimens to be made from it. The observance of this point is particularly important if the specimens are to be tested for electrical properties, flame resistance, or freedom from taste and odor. Polytetrafluorethylene and silicones can normally be used.

Using the type 1 mould, it is recommended that the impregnated glass reinforcement be covered with a second layer of foil or film before it is placed between the plates of the mould. The impregnated glass reinforcement thus is pressed between the two layers of foil or film, both of which should be at least 30 % larger than the dimensions of the glass reinforcement. These films or foils are stripped from the plate after it has been moulded and cooled.

4.1.4 Moulding procedure depends considerably on the properties of the resin. Unless otherwise specified, the mould, heated to the required temperature, should be closed and the moulding pressure should be attained before the gelification of the resin at the given moulding temperature starts. The time between the closing of the mould and attainment of the required moulding force should be specified (this time is reckoned from the first deflection of the pressure measuring instrument to the attainment of the value required) with as uniform a rise of force as possible. The moulding force should be kept constant (see clause 3.1) for the prescribed period of time.

4.1.5 When the type 2 mould is used, the glass content of the moulded plate and its thickness are functions of temperature, pressure, and other factors depending upon the properties of the glass reinforcement and of the resin. For this reason it will be necessary, before making the number of plates or panels required for preparation of the test specimens, to determine by experiment the number of layers of glass reinforcement and the moulding pressure necessary to obtain plates or panels of the required thickness. When the type 1 mould (see Fig. 1) is used, the number of layers can be calculated (see clause 4.3.3) and the thickness of the plate can be governed by the use of appropriate spacers. The magnitude and tolerances of the thickness, within and between plates, should be in agreement with the requirements of the testing method. Where the glass content is not subject to separate agreement, the laminate produced should contain 60 ± 2 % (m/m) of glass in the case of fabrics and 40 ± 4 % (m/m) in the case of mats. Unless otherwise specified, the principal directions of all layers of the reinforcement should be the same. In other cases the orientation of every individual layer of the reinforcement should be stated using schematic drawing.

4.1.6 The temperature and time of curing is stipulated by agreement, depending on the type of resin, catalyst, or curing agent. The temperature stipulated should be so maintained during the curing cycle that the temperature values indicated by the temperature check sensitive elements will remain within the ranges shown in the diagram in Figure 3; line 1 represents temperature fluctuations during curing times up to 30 minutes; line 2 over 30 minutes.

4.1.7 After the lapse of the moulding process the test plate has to be taken from the mould and cooled in such a way that any deformation, damage, etc. is avoided.

4.1.8 Unless otherwise stipulated by agreement, the plates are used without further treatment for making test specimens by machining. If not specified in the relevant ISO Recommendations on testing, the types, sizes and orientations of the specimens with respect to the orientation of the glass reinforcement should be stipulated by separate agreement. The margins of the plate up to at least 15 mm from its edges should be discarded.

4.2 Method B

4.2.1 The glass reinforcement impregnated with resin partially cured to the B stage and no longer sticky is shorn, cut, or chopped to rectangles, the width and length of which are 3 to 6 mm less than the dimensions of the cavity of the tool.

4.2.2 The test pieces, prior to moulding, may be, if necessary, stored in a desiccator or at reduced temperature.

4.2.3 The required number of layers of impregnated glass reinforcement are freely laid on top of each other and then placed in the cavity of the mould.

4.2.4 The rest of the working procedure is identical with that described in clause 4.1.

4.3 Method C

4.3.1 The glass reinforcement is shorn, cut or chopped to dimensions not less than 400 mm × 400 mm and dried as indicated in clause 4.1.2.

4.3.2 The impregnation of the glass reinforcement with liquid resin is carried out on a smooth rigid support pad, heated if necessary, and provided with a suitable release agent (see clause 4.1.3). The support pad is covered with a thin layer of catalyzed resin, then the first layer of the glass reinforcement is laid out and covered again with a layer of resin, and so forth. Each layer or each second layer (depending on the thickness of the glass reinforcement) before being covered with resin is rolled with a special roller (see Note below) so as to remove all visible air bubbles and to impregnate the glass reinforcement thoroughly with resin over the whole area. The rolling at the same time removes excess resin. An excess of at least 20 % of resin as compared with the resin content in the cured product should be used for a lamination of the required thickness. Not more than 50 % of the gel time of the resin at the temperature of the impregnation may elapse before the end of the lamination. The remaining resin is poured over the last layer of glass reinforcement which is then covered with a plane glass or similar plate. This plate is provided with a release agent. It is pressed down by hand without enclosure of bubbles until it contacts the three spacers determining the thickness of the plate and it is left in that position until the resin is cured. Where rigid glass plates cannot ensure a smooth surface free from bubbles, flexible plates or foils may be used, in which case care should be exercised to keep the thickness tolerance within the prescribed limits. The laminate is prepared at a relative humidity of 65 % unless otherwise agreed.

NOTE. – Well-suited for this purpose are soft-faced rollers; for example, rollers covered with mohair tissue. A rod or paddle covered with polytetrafluorethylene may be used instead. Air bubbles in light-coloured resins can be seen more easily if the support pad for the lamination is dark-coloured or translucent and lighted from below.

4.3.3 The number of layers of glass reinforcement should be such that the glass content of the cured plate is 50 ± 2 % (m/m) in the case of glass fabrics and 30 ± 4 % (m/m) in the case of mats. By agreement the plates may also contain other proportions of glass. For one lot, however, the glass content should be constant within the range which should be specified. The magnitude and tolerances of the thickness, within and between plates, should be in agreement with the requirements of the testing method. For the determination of the required number of layers of glass reinforcement it will be necessary to make a preliminary experiment or to calculate this number by the use of the formula

$$n = \frac{e\,\rho_g\,\rho_n\,b}{g\,[b\rho_n + \rho_g(1-b)\,]}$$

where

n is the number of layers;

e is the thickness of the plate, in centimetres;

ρ_g is the density of the glass in grammes per cubic centimetre;

ρ_n is the density of the cured resin in grammes per cubic centimetre;

b is the glass content, by mass, expressed as a fraction of one;

g is the mass of glass reinforcement per unit area in grammes per square centimetre.

4.3.4 Unless otherwise specified, the laminated plate or panel prepared as described in clause 4.3.2 is either left for 48 hours at room temperature or is placed together with its support pad in an oven or heated air bath. Curing at room temperature may be accomplished at temperatures ranging from 20 to 25 °C. In the case of curing in an oven or heated air bath the temperature should be checked by means of two sensitive elements situated not more than 50 mm above opposite corners of the plate. Temperatures indicated by these elements should not vary more than shown in the graph, Figure 3.

4.3.5 In the case of curing at room temperature, the plate may be taken off the support pad after 48 hours but, unless otherwise specified, it is then to be left freely exposed for 21 days at room temperature in a standard atmosphere, according to ISO Recommendation R 291, *Standard atmospheres for conditioning and testing,* on a plane surface. If less than 21 days elapse between curing of the plate at room temperature and the making of test specimens, the time elapsing between the making of the test specimens and their testing should be exactly specified. The margins of the plate up to at least 20 mm from the edge may not be used for making test specimens and the working portions of the specimens are not to be taken any closer than 40 mm from the edge.

4.3.6 In the case of curing at elevated temperatures, the plate or panel, together with its support pad, is taken out of the air bath at the end of the curing time and is left to cool freely for at least 60 minutes. After being removed from the support pad, the plate may be used for making test specimens with the exclusion of margins as specified in clause 4.3.5.

5. REPORT OF PREPARATION OF TEST PLATE OR PANEL

The report of preparation of the test plate should include the following data :

(*a*) place and date of production of test plate;

(*b*) description of materials used for preparation of plate (nature and type of resin, catalyst, curing agent or other additives including the amounts used, nature and type of glass reinforcement, nature of finish, etc.);

(*c*) description of production equipment (type of press, mould, method of checking temperatures and pressures, etc.);

(*d*) working procedure (designation of method as given in this ISO Recommendation, moulding pressure (or force), temperature, curing time, release agent used, post-curing, etc.);

(*e*) date indicating when test pieces are to be made;

(*f*) dimensions of plate, mass of plate and its glass content;

(*g*) special notes.