SLOVENSKI STANDARD
SIST EN ISO 12114:1999

01-maj-1999

Z vlakni ojačeni polimerni materiali – Duromerne mase za oblikovanje in predimpregniranja – Določevanje poteka zamreženja (ISO 12114:1997)

Fibre-reinforced plastics - Thermosetting moulding compounds and prepregs - Determination of cure characteristics (ISO 12114:1997)

Faserverstärkte Kunststoffe - Härtbare Formmassen und Prepregs - Bestimmung des Härtungsverhaltens (ISO 12114:1997)

Plastiques renforcés de fibres - Compositions de moulage et préimprégnés - Détermination des caractéristiques de durcissement (ISO 12114:1997)

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Fibre-reinforced plastics - Thermosetting moulding compounds and prepregs - Determination of cure characteristics (ISO 12114:1997)

Plastiques renforcés de fibres - Compositions de moulage et préimprégnés - Détermination des caractéristiques de durcissement (ISO 12114:1997)

Faserverstärkte Kunststoffe - Härtbare Formmassen und Prepregs - Bestimmung des Härtungsverhaltens (ISO 12114:1997)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

Foreword

The text of the International Standard ISO 12114:1997 has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with 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of an identical text or by endorsement, at the latest by November 1997, and conflicting national standards shall be withdrawn at the latest by November 1997.

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

Endorsement notice

The text of the International Standard ISO 12114:1997 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 12114

First edition
1997-05-15

Fibre-reinforced plastics — Thermosetting moulding compounds and prepregs — Determination of cure characteristics

Plastiques renforcés de fibres — Compositions de moulage thermodurcissables et préimprégnés — Détermination des caractéristiques de durcissement

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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.

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 12114 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres.*

Introduction

The description of the cure characteristics of thermosetting moulding compounds is rather complex. Various parameters and properties like reactivity, temperature, pressure and shrinkage are of interest to the end user. Some of these parameters influence each other. For this reason, several test methods are necessary for quality control and to determine the effect of varying the proportion of any particular component in the formulation. The characteristics determined are selected to enable different moulding compounds and prepregs to be compared and their shelf life assessed.

One important characteristic of thermosetting compounds is their reactivity, i.e. their ability to polymerize under the influence of a catalyst and/or heat. This reactivity can be evaluated by various methods. This International Standard gives two such methods:

— the first of which is a simple procedure which is limited to the determination of reactivity and shelf life by measuring the variation of temperature as a function of time under the conditions usually used in production;

— the second method is more sophisticated and gives information on the physical behaviour of the compound under simulated moulding conditions.

The description of the curing behaviour of a thermosetting compound is not possible by a single value. It is dependent on several parameters, like pressure, temperature and shrinkage, which influence each other. This interdependence must also be described. Such a description can be given by mathematical functions or expressed in terms of a set of curves for the various parameters, as in this International Standard. The curves for a particular compound are all shown in the same figure to enable the interdependence of the parameters concerned to be seen at a glance.

Fibre-reinforced plastics — Thermosetting moulding compounds and prepregs — Determination of cure characteristics

1 Scope

This International Standard specifies two methods for the determination of the cure characteristics of fibre-reinforced thermosetting moulding compounds and prepregs.

The characteristics measured are

— the heat generated by the exothermic reaction;

— the thermal expansion of the compound accompanying the rise in temperature;

— the shrinkage due to the curing reaction.

The methods specified are applicable to all compounds comprising thermosetting matrices reinforced with fibres. The major field of application is compounds based on unsaturated polyester resins.

Method I describes a test method to determine the reactivity of a thermosetting compound by a simple procedure. This method examines only the reactive behaviour of the matrix and its state of stabilization (one factor affecting the shelf life of the moulding compound).

Method II describes a test method which needs more sophisticated equipment, takes more time but provides additional detailed information about the physical behaviour of the compound under simulated practical moulding conditions. It produces a plate which can be used for further testing to determine properties of the moulded article.

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 — Prepregs — Bulk moulding compound (BMC) and dough moulding compound (DMC) — Basis for a specification.*

3 Definitions

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

3.1 reactivity: The reactivity of a thermosetting material is taken, by convention, as the maximum gradient, in degrees Celsius per second, of a plot of the temperature of the thermosetting material as a function of time during curing.

3.2 curing behaviour: The behaviour of a thermosetting material when moulded under commonly used moulding conditions, as described by the following set of parameters:

— cure time;

— thermal expansion;

— shrinkage due to the curing reaction;

— net shrinkage (reaction shrinkage less thermal expansion).

The values of these parameters will depend on the actual moulding conditions used.

3.3 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 Apparatus

4.1 Method I

4.1.1 Cylindrical metal mould, with an inside diameter of 20 mm, equipped with a heater and a temperature control system, and with a heated steel punch which fits inside the cylindrical mould (see figure 1).

4.1.2 Device for applying a minimum force of 15 daN to the punch (see figure 2).

4.1.3 Insulated thermocouple, diameter 1 mm, located at the centre of the bottom of the mould and rising 5 mm into the mould cavity. The thermocouple shall be insulated from the heated steel block by insulation having a thickness of > 2 mm.

4.1.4 Suitable recording system, for plotting a curve of temperature versus time.

4.1.5 Device for preparing moulding-compound test specimens (see figure 3).

4.1.6 Syringe, for measuring out paste specimens.

4.1.7 Balance, accurate to the nearest 0,1 g.

4.1.8 Brass bristle brush, to clean the mould.

4.2 Method II

4.2.1 Suitable press, capable of applying a moulding pressure of 100 bar ± 3 %.

4.2.2 Shear edge mould, mounted in the press, with a cavity of surface area not less than 200 cm^2, heatable to 200 °C with an accuracy of 1 % (see figure 4). The mould shall be equipped with a pressure sensor and temperature sensor, mounted in the centre of the mould. For pressure measurements, piezoelectric sensors are normally used.

The temperature sensor shall be insulated from the mould to measure changes in temperature at the surface of the moulding compound with an accuracy of ±1 %.

Both sensors shall be flush with the surface of the mould cavity. A displacement sensor shall be fitted to the mould to measure mould lid travel of about 20 mm with a resolution of 0,01 mm.

NOTE — For practical reasons, it is recommended that the sensors be mounted at a maximum distance apart of 30 mm and equidistant from the axis of symmetry.

4.2.3 Recording equipment, connected to the sensors in the mould, to plot the variation with time of

— pressure (from 0 to 150 bar);

— displacement (from 0 to 20 mm);

— temperature (from 0 to 200 °C).

4.2.4 Calipers, for measuring the thickness of the moulded test specimen.

5 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 7.1).

6 Conditioning and test atmospheres

6.1 Conditioning

Condition the laboratory sample for a time sufficient to establish temperature equilibrium. The temperature of the laboratory sample immediately before the test specimens are taken shall not be less than 18 °C.

6.2 Test atmosphere

The atmosphere for testing shall be the same as that used for conditioning.