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Plastics — Glass-fibre-reinforced products — Determination of fibre length

Plastiques — Produits renforcés de fibres de verre — Détermination de la longueur des fibres

Reference number
ISO 22314:2006(E)

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Foreword

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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.

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ISO 22314 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Introduction

There is global interest in the determination of the length of glass fibres in all types of composite, to predict their characteristics (essentially mechanical ones). For these determinations, three steps are necessary:

— separation of the fibres from the composite;

— dispersion of the fibres to obtain individual fibres;

— measurement of their length.

After considering all the existing methods for separating the fibres from the resin, it was decided to develop the proposed method only for short glass fibres from thermoplastic resins, extracted by calcination.

The principles of the method are probably suitable for other composites, but it would need more development to obtain a method suitable for other conditions (thermoset resins, long fibres in thermoplastic or thermoset resins, carbon fibres).

Plastics — Glass-fibre-reinforced products — Determination of fibre length

1 Scope

This International Standard specifies a method of determining the length of the fibres present in a fibre-reinforced product. The method is applicable to moulding materials and to moulded parts. The test conditions specified limit the application of this method to thermoplastics reinforced with short glass fibres (less than 1 mm long), i.e. fibres whose length is less than or equal to 7,5 mm prior to incorporation in the moulding material and moulding.

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 472, *Plastics — Vocabulary*

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

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 472 apply.

4 Principle

The fibres are separated from the reinforced polymer and their length measured from a magnified image on a screen.

5 Apparatus

5.1 Muffle furnace, capable of maintaining a temperature of 625 °C ± 25 °C.

5.2 Crystallizing dish, diameter 80 mm to 110 mm.

5.3 Ultrasonic device.

NOTE A basic ultrasonic device, like those used in laboratories for cleaning purposes, has been found suitable.

5.4 Microscope or **stereoscope** (with fixed or variable magnification factors), equipped with a video camera, giving at least × 50 magnification.

5.5 Image acquisition device, that enables the image to be displayed on a TV monitor.

5.6 Reference micrometer, to calibrate the images at the selected magnification.

5.7 Data-processing equipment, with suitable image analysis software.

5.8 Drying oven, capable of maintaining a temperature of 130 °C ± 5 °C.

5.9 Laboratory and observation accessories (crucibles, evaporation dishes, spatulas, glass microscope slides).

6 Procedure

6.1 Preparation of the test specimen

From the sample to be examined, take the quantity of material necessary to obtain a concentration of fibres such that each image displayed on the screen contains a hundred or so fibres. This quantity will depend on the fibre content of the material and on the test conditions, in particular on the diameter of the crystallizing dish (the water depth in the crystallizing dish does not have any influence on the concentration of the fibres deposited on the glass slide, as the fibres precipitate in a horizontal plane).

EXAMPLE For a 30 % glass-fibre-reinforced polyamide, around 0,006 g of material are required when a 90 mm diameter crystallizing dish is used.

NOTE This assumes that the fibre length is less than 1 mm.

Calcine the material at 625 °C in accordance with ISO 1172 for 1 h 30 min and allow to cool.

NOTE The ash obtained is mainly made up of glass fibres and possibly also mineral fillers. The fibres are very fragile and all subsequent handling must be conducted with extreme caution in order not to break them and therefore invalidate the results.

Place a previously degreased glass microscope slide in a crystallizing dish (5.2) and pour in a quantity of demineralized water (containing a small amount of surfactant) just sufficient to cover the slide. The quantity of water is limited in order to avoid the formation of convection currents during subsequent evaporation, which lead to a selection of the fibres on the basis of their mass and therefore of their size.

Pour the ash into the crystallizing dish prepared as described above (or transfer a sufficient quantity on the tip of a spatula).

Place the crystallizing dish containing the ash in the ultrasonic device in order to disperse the fibres without any mechanical action. The time required for this dispersion is in the region of a few seconds to 1 min.

Then place the crystallizing dish in an oven (5.8) preheated to 130 °C and leave it there for approximately 1 h, in order to eliminate the water. Allow to cool down. This procedure may be omitted if heating tends to cause the fibres to stick together.

Place the crystallizing dish beside the microscope or stereoscope (5.4). Take the slide covered with fibres and place it under the microscope or stereoscope lens. If necessary, wipe off any fibres present on the underside of the slide.

6.2 Calibration

Calibrate the system using a reference gauge; a 1 mm or 1,5 mm length micrometric glass slide has been found suitable.

Calibration can be carried out by measuring the length of the reference gauge, pointing to the two ends as would be done when measuring the lengths the fibres, and comparing the reading with the gauge length. The recommended tolerance is 0,01 mm.

Calibrate the microscope as frequently as necessary.

6.3 Examination and measurements

The fibres are examined directly, without microscope cover glass or mounting fluid, in reflected or transmitted light, in the light-field or dark-field (annular illumination) mode.

Adjust the overall magnification chain (optics and projection) in order to obtain on the screen an image magnified between 50 and 100 times. The magnification shall be such that the whole lengths of 100 ± 20 fibres appear on the screen.

Measure manually the lengths of all the complete fibres appearing on the screen by clicking with the mouse on the ends of each fibre. Measure 100 ± 20 fibres in this way from each of three images, for a total of 300 ± 60 fibres.

NOTE 1 This manual measurement method can be replaced by a semi-automatic method. However, it must be realized that such methods are biased towards the smaller fibres and that the results obtained will therefore be systematically lower than those obtained by manual measurement.

NOTE 2 While outside the scope of this method, if longer fibres up to 5 mm in length are measured, the magnification can be reduced to as little as 15, provided the quality of the optical system is good enough to permit satisfactory measurement.

6.4 Expression of results

The results to be reported are:

— L_{n}, the mean fibre length, expressed in micrometres, calculated from the following equation:

$$L_{\mathrm{n}} = \frac{\sum_{i=1}^{i=n} L_i}{n}$$

where

L_i is the length of the ith fibre,

n is the number of fibres measured;

— σ, the standard deviation of the individual values;

— a histogram plotted for individual ranges over the entire measurement range (e.g. 50 ranges of 40 µm each over a measurement range extending from 0 µm to 2 000 µm);

— L_{p}, a weighted mean length, expressed in micrometres, calculated from the following equation:

$$L_{\mathrm{p}} = \frac{\sum_{i} n_i L_i^2}{\sum_{i} n_i L_i}$$ where n_i is the number of fibres of length L_i;

— the ratio $L_{\mathrm{p}}/L_{\mathrm{n}}$.

7 Test report

The test report shall contain the following information:

a) a reference to this International Standard, i.e. ISO 22314:2006;

b) all details necessary for identification of the sample;

c) the quantity of material sampled, the diameter of the crystallizing dish and the quantity of water added;

d) the magnification used;

e) the results obtained as described in Clause 6, as well as the minimum and maximum lengths measured;

f) the date of the measurement;

g) all procedural details not specified in this method.

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