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Advanced technical ceramics - Ceramic composites - Methods of test for reinforcement - Part 1: Determination of size content

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Hochleistungskeramik - Keramische Verbundwerstoffe - Verfahren zur Prüfung der Faserverstärkungen - Teil 1: Bestimmung des Schlichtegehaltes

Céramiques techniques avancées - Céramiques composites - Méthodes d'essai pour renforcements - Partie 1: Détermination du taux d'ensimage

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This European Standard was approved by CEN on 6 July 2002.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 1007-1:2002) has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by March 2003, and conflicting national standards shall be withdrawn at the latest by March 2003.

This document supersedes ENV 1007-1:1993.

EN 1007 has six parts:

— EN 1007-1 *Part 1: Determination of size content.*

— EN 1007-2 *Part 2: Determination of linear density.*

— EN 1007-3 *Part 3: Determination of filament diameter and cross-section area.*

— prEN 1007-4 *Part 4: Determination of tensile properties of filament at ambient temperature.*

— prEN 1007-5 *Part 5: Determination of distribution of tensile strength and of tensile strain to failure of filaments within a multifilament tow at ambient temperature.*

— ENV 1007-6 *Part 6: Determination of tensile properties of filament at high temperature.*

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, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This part of EN 1007 specifies the conditions for determination of the size content of ceramic fibres, including among others silicon carbide, silicon nitride, silicon carbonitride, alumino-silicate, alumina and silicon oxide fibres.

NOTE Carbon fibres are not covered by this European Standard. EN ISO 10548 should be used for carbon fibres.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 291, *Plastics - Standard atmospheres for conditioning and testing (ISO 291:1997).*

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
size
materials applied to the ceramic fibres to facilitate the handling and use of the fibre

3.2
size content
the mass of the size expressed as a percentage of the original mass of the sized ceramic fibre in the conditioned state

3.3
elementary unit
the smallest commercially available unit of a given product

NOTE For fibre, usually this is a spool.

4 Principle

Weighing of test specimens before and after removal of size, according to conditions and specified method of solvent extraction.

The solvent chosen is dependent on size type and is usually indicated by the manufacturer of the fibre.

5 Apparatus and chemicals

5.1 Balance, accurate to ± 0,1 mg.

5.2 Oven, for drying specimens, capable of being controlled at the chosen temperature ± 5 °C.

5.3 Desiccator, containing a suitable desiccant (for example, silica gel, calcium chloride, phosphorus pentoxide).

5.4 Thimble, typically 25 mm in diameter and 65 mm in height.

5.5 Rubber gloves or tweezers.

5.6 Cutting blade.

5.7 Reflux extractor with condenser.

5.8 Heating device, such as a water bath, hot plate or isomantle, controllable to the required temperature.

5.9 Boiling flask.

5.10 Organic solvent, such as 2-butanone (methylethylketone), tetrahydrofuran, dimethylformamide, dichloromethane (methylene chloride), acetone, dichloroethane or distilled water, as a function of the size type.

WARNING: Extraction and handling of organic solvents must be done under fume extraction.

6 Test specimen

At least three test specimens shall be taken at random from each elementary unit. The minimum mass of the test specimen is 2 g. Care shall be taken to handle the specimen only with gloved hands or tweezers.

7 Conditioning of test specimens prior to weighing

Storage of the test specimens shall be in one of the atmospheres specified in EN ISO 291. Any drying procedure of the sized fibre prior to weighing shall be agreed between supplier and customer.

NOTE For example, (105 ± 5) °C, 1 h.

8 Procedure

8.1 Dry the thimble for 1 h at (105 ± 5) °C. Weigh the dried thimble, M_2 and the test specimen of conditioned fibre, M_1. Put the test specimen in the thimble. Fill the boiling flask with a suitable solvent. Adjust the volume of solvent to ensure there is sufficient to fill the reflux system.

8.2 Put the thimble, containing the test specimen, into extractor with a condensor and fix the whole to the boiling flask. Adjust the rate of refluxing to 5 cycles/h and the number of hours of reflux to ensure that complete extraction of the size is achieved. Remove the thimble immediately after the last refluxing.

NOTE To ensure that complete extraction is achieved, usually preliminary trial runs with different extracting periods are performed.

8.3 Dry the thimble with the desized test specimen during one hour in the oven set at a temperature of (110 ± 5) °C. Put the thimble and the test specimen in a desiccator and allow to cool down, then weigh the whole to within 0,1 mg.

If the solvent used has a boiling point above 100 °C, set the temperature of the oven at a value 10 °C higher than the boiling point of the solvent.

8.4 Run again operations described in 8.3 until the difference in weight of two successive weighings is lower than 0,2 mg. M_3 is the final weight.

9 Calculation of results

Calculate the size content for each test specimen in accordance with the following equation:

$$SC_i = \frac{M_1 - (M_3 - M_2)}{M_1} \times 100$$

where

SC_i is the size content expressed in percent (%), and i = 1, 2, 3;

M_1 is the weight of the dried test specimen, expressed in gram (g);

M_2 is the weight of the dried thimble, expressed in gram (g);

M_3 is the weight of the dried thimble plus the desized test specimen, expressed in gram (g).

The size content SC expressed in percent is the arithmetic mean of the above three results.

10 Test report

The test report shall contain at least the following information:

a) the name and address of the testing establishment;

b) the date of the test, unique identification of report and of each page, signatory, customer name and address;

c) a reference to this European Standard, i.e. "Determined in accordance with EN 1007-1";

d) the description of the test material: type of fibre, batch number, date of receipt, type of sizing if it is known;

e) conditions of extraction: type of solvent, temperature, duration;

f) the size content (SC);

g) comments about the test or test results.