BRITISH STANDARD

BS EN 1007-3:2002

Advanced technical ceramics — Ceramic composites — Methods of test for reinforcement —

Part 3: Determination of filament diameter and cross-section area

The European Standard EN 1007-3:2002 has the status of a British Standard

ICS 81.060.30

National foreword

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The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics, which has the responsibility to:

— aid enquirers to understand the text;

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Advanced technical ceramics - Ceramic composites - Methods of test for reinforcement - Part 3: Determination of filament diameter and cross-section area

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Hochleistungskeramik - Keramische Verbundwerkstoffe - Verfahren zur Prüfung der Faserverstärkungen - Teil 3: Bestimmung des Faserdurchmessers und -querschnitts

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Contents

Foreword

This document (EN 1007-3:2002) 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of an identical text or by endorsement, at the latest by March 2003, and conflicting national standards shall be withdrawn at the latest by March 2003.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 1007-3:1993.

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 the European Standard specifies the conditions for the determination of the diameter and cross-section area of ceramic single filament, as used in fibre reinforcement of ceramic composites for three methods.

NOTE The cross-sectional area of filaments from different suppliers will vary significantly. The term “diameter” applies both to circular cross-section (“true diameter”) and non-circular cross-sections (“apparent diameter”).

2 Principle

The diameter and cross-section area of the ceramic filament may be measured by three methods, as follows:

a) the longitudinal profile method (Method A), which determines the filament diameter by optical microscopy. This is used for filaments which have a circular cross-section;

b) the transverse section method (Method B), which determines the filament diameter and the cross-sectional area by optical microscopy. This is mainly used for filaments which have a non-circular cross-section;

c) the laser method (Method C) which determines the apparent filament diameter by laser interferometry. This is used for filaments which have a circular cross-section.

3 Sampling

Take 25 ceramic filaments at random from each sample unit, for measurement by one of the three methods. If any rules of sampling are used, these shall be reported (see clause 7).

4 Longitudinal profile method (Method A)

4.1 Introduction

The diameter of the filament is measured by optical microscopy, by measuring the distance which separates the two edges of a single filament examined in a longitudinal direction.

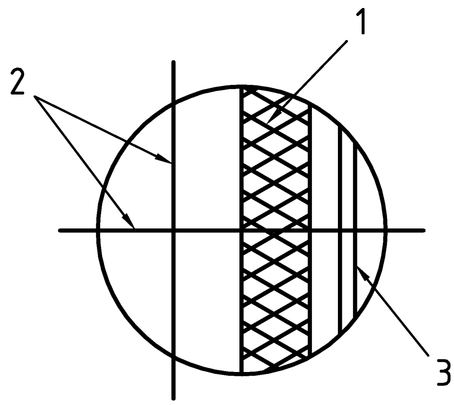
4.2 Apparatus

The microscope shall be fitted with a light source, a sub-stage condenser, a stage, an objective and a micrometer eyepiece. This eyepiece is either fitted with two fixed orthogonal wires and a double wire parallel to one of the two directions (see Figure 1), or may be of the double curtain type which uses semi-opaque blinds which are moved by a micrometre device.

The stage shall move in two horizontal planes perpendicular to each other and may be rotated about a vertical axis. The objective and the selected eyepiece shall have a magnification of at least 1 000 times for measurement of filament diameter, and also of about 100 times for filament identification.

The immersion medium for the filament samples shall have a refractive index of between 1,43 and 1,53 when measured at 10 °C. The medium shall not be hygroscopic or react with the filament.

NOTE Cedar oil and paraffin are suitable.



Key

1 Single fixed

2 Single filament

3 Double wire

Figure 1 – The principle of the micrometer eyepiece (Method A)

4.3 Calibration

The microscope shall be calibrated by using a traceably calibrated stage micrometer and a micrometer eyepiece. This operation consists of determining the calibration constant, which is the number of graduations of the drum which correspond to a movement of one micrometre on the stage micrometre.

4.4 Procedure

Mount several small lengths of ceramic fibre cut from a multifilament tow sample ona microscope slide, suitably spread out, add a few drops of the immersion medium, and cover with a microscope cover slip.

Select one of these fibre specimens and scan it by moving the stage to position the light beam on it. Focus the graticule by means of the eyepiece.

Rotate the eyepiece or the stage in order to observe the double wire (see 4.2) exactly parallel to the axis of the filament being measured. Focus on this filament and then bring the double wire to coincide successively with the two sides of the filament image. Read the number of graduations of the drum which results from passing from one of these positions to the other.

4.5 Calculation of results

Calculate the diameter of the filament from the following expression:

$$d = \frac{N_R}{n}$$

where

d is the filament diameter, expressed in micrometers (µm);

N_R is the number of graduations of the drum (see 4.4);

n is the calibration constant (see 4.3).

5 Transverse section method (Method B)

5.1 Introduction

The sample of ceramic multifilament tow is embedded in a resin block, the block is polished on a face normal to the axis of the filament and the cross-sectional area determined by optical microscopy.

5.2 Apparatus

5.2.1 Reflected light optical microscope.

5.2.2 Planimeter or image analyzer.

5.2.3 Polishing machine.

5.2.4 Resin for sample preparation and means for its storage in a mould.

5.3 Sample preparation

Choose a resin which permits good adhesion between the ceramic filament and the matrix, with minimal shrinkage.

Position the sample so that the multifilament tow is perpendicular to the face of the resin block which is to be polished and examined. Pour the resin into the mould around the multifilament tow and polymerize the resin according to a known temperature-time cycle.

Cool and then polish the selected surface until no scratches are visible on the surface of the resin block.

5.4 Procedure

Select a magnification of between 1 000 times and 1 500 times. If possible, polarised light with crossed polarizer and analyzer and a half-wavelength plate should be used to improve the definition and contrast of the image. When an inverted microscope is used, the face of the specimen may be placed on the stage and is thus normal to the optical axis.

Measure the cross-sectional area of each filament by planimetry, using either a photograph of known magnification or an image analyzer. The actual magnification is calculated by photographing a stage micrometer (see 4.3) under the same conditions.

Measure, by either the planimeter or image analyzer, the cross-sectional area of each filament image. Divide this value by the square of the magnification to give the cross-sectional area of the filament.

5.5 Calculation of results

Calculate the filament diameter from the following expression:

$$d_e = 2\sqrt{\frac{S}{\pi}}$$

where

d_e is the equivalent filament diameter, expressed in micrometers (µm);

S is the cross-sectional area of the filament, expressed in square micrometers (µm^2).

For filaments with circular cross-section, the filament diameter can be calculated directly.

6 Laser interferometry method (Method C)

6.1 Introduction

The diffraction image obtained when a filament is illuminated by a laser beam is observed on a screen and the distance between diffraction fringes is measured.

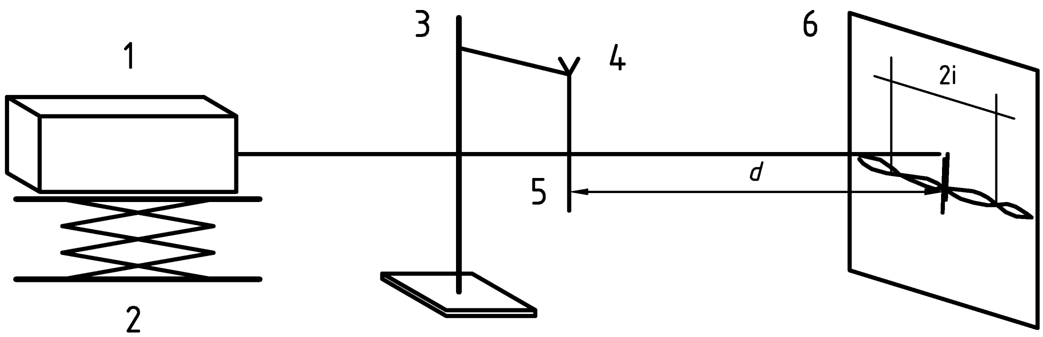
6.2 Apparatus

6.2.1 Low-powered laser

NOTE A suitable type is a helium-neon laser with monochromatic non-polarised light, of wavelength 632,8 nm and power 0,5 mW.

6.2.2 Vertical support, mounted on a platform able to be displaced in two perpendicular directions, vertical and horizontal.

6.2.3 Projection screen, the plane of which is perpendicular to the laser beam (see Figure 2).



Key

1 Laser
2 Elevator support
3 Support
4 Grip
5 Test specimen
6 Screen

Figure 2 – Arrangement for laser interferometry (Method C)

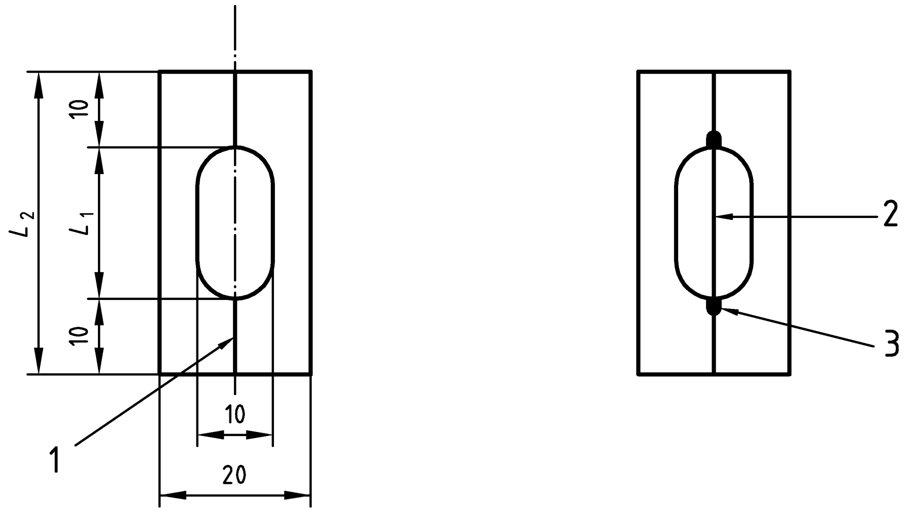
6.3 Sample preparation

Use a mounting tab for preparation of the filament specimen as shown in Figure 3. Centre the specimen over the tab slot with one end taped to the tab and lightly stretch the filament on the mounting tab axis. Tape the opposite end of the filament to the tab.

Carefully place a small amount of adhesive on the filament at each edge of the slot (see Figure 3) and bond the filament to the mounting tab. The length of the filament shall be between 10 mm and 50 mm.

Place the filament specimen in the mounting tab between the laser and the screen.

Dimensions in millimetres



Key

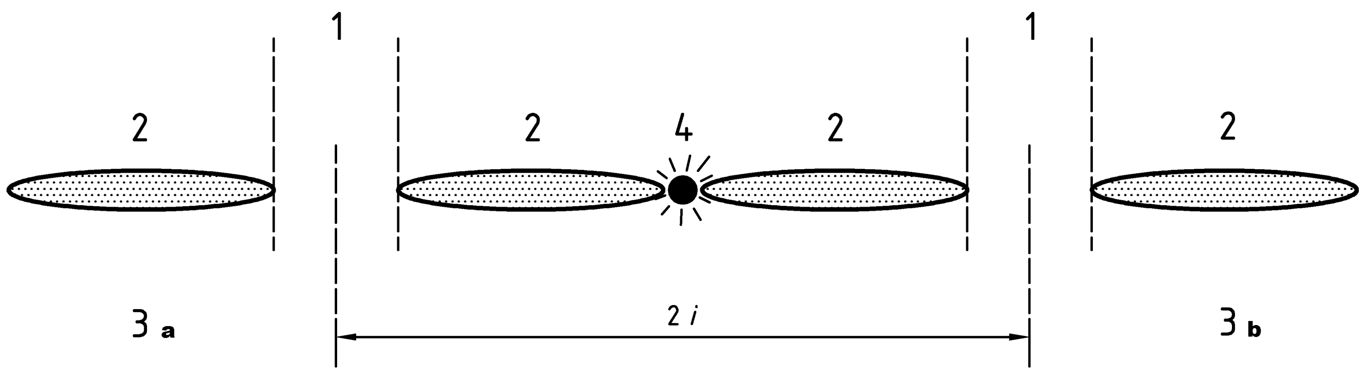
1 Centre line

2 Single filament test specimen

3 Adhesive

Figure 3 – Mounting tab with filament (Method C)

6.4 Procedure



Measure the interfringe distance, 2*l*, to 0,5 mm using a graduated rule, as shown in Figure 4.

Key

1 Dark zone

2 Light zone

3a Medium of the dark zone

3b Medium of the light zone

4 Impact of direct beam

Figure 4 – Definition of laser interference fringe (Method C)

Measure the distance between the specimen and the screen, which shall be greater than 500 mm.

6.5 Calculation of results

Calculate the filament diameter from the expression:

$$d = \frac{\lambda \, . \, D}{i} . 10^{-3}$$

where

d is the filament diameter, expressed in micrometre (µm);

i is the interfringe distance, expressed in millimetre (mm);

λ is the wavelength of the laser beam, expressed in nanometre (nm);

D is the distance between the specimen and the screen, expressed in millimetre (mm).

7 Test report

The test report shall contain at least the following information:

a) the name of the testing establishment;

b) date of the test, report identification and number, operator, signatory;

c) a reference to this European Standard, i.e. “Determined in accordance with EN 1007-3”;

d) a description of the test material: type of filament, batch number, date of receipt;

e) the method of measurement selected, Method A, Method B or Method C (see clause 2);

f) the type of diameter measured (see clause 1);

g) the rules of sampling or sampling plans used, if any (see clause 3);

h) the number of filaments measured (see clause 3);

i) the mean filament diameter or cross-section area and the range of measurements;

j) where required, the mean filament cross-sectional area and the range of measurements;

k) comments about the test or the test results.

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