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SIST ENV 1007-3:2000

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Advanced technical ceramics - Ceramic composites - Methods of test for reinforcements - Part 3: Determination of filament diameter

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

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Céramiques techniques avancées - Céramiques composites - Méthodes d'essai pour renforts - Partie 3: Détermination du diametre des filaments

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CEN

European Committee for Standardization
Comité Européen de Normalisation
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Foreword

This European pre-Standard has been prepared by CEN/TC 184 "Advanced technical ceramics" the secretariat of which is held by BSI.

ENV 1007 "Advanced technical ceramics - Ceramic composites - Method of test for reinforcements" has four Parts:

Part 1 : Determination of size content
Part 2 : Determination of linear density
Part 3 : Determination of filament diameter
Part 4 : Determination of tensile properties of filament at ambient temperature

CEN/TC184 approved this European Prestandard by resolution 4/1992 during its fifth meeting, held in Brussels in 1992-03-31.

In accordance with the CEN/CENELEC Internal Regulations, the following countries are bound to announce this European Prestandard.

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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Advanced technical ceramics – Ceramic composites – Methods of test for reinforcements – Part 3: Determination of filament diameter

1 Scope

This Part of ENV 1007 specifies three methods for the determination of the diameter and cross-sectional area of ceramic single filaments, as used in fibre reinforcement of ceramic composites.

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

2 Principle

The diameter and cross-sectional 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 filament which has a circular cross-section, and is the only method suitable for short fibres.

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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 filament which has a non-circular cross-section.

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c) The Laser method (Method C) which determines the apparent filament diameter by laser interferometry. This is used for filament which has 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 micrometer device.

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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 1000 times for measurement of filament diameter, and also of about 100 times for filament identification.

The mounting 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.

4.3 Calibration

The microscope shall be calibrated by using a 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 micrometer.

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4.4 Procedure

Mount several small lengths of ceramic fibre, cut from a yarn sample, between a microscope slide and a cover slip.

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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 image. Read the number of graduations of the drum which results from passing from one of these positions to the other.

4.5 Expression of results

Calculate the diameter of the filament from the following expression:

$$d = \frac{N_R}{2n}$$

where d is the filament diameter in micrometres
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 fibre, cut from a yarn sample, 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.

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5.2 Apparatus

5.2.1 Optical microscope, transmission type, with associated photographic equipment.

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 fibre and the matrix, without shrinkage.

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

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

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5.4 Procedure

Select a magnification of between 1000 times and 1500 times. If possible, polarized 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 required magnification is calculated by photographing a stage micrometer (see 4.3) under the same conditions.

Measure, by either the planimeter or image analyzer, the surface area of each filament image. Divide this value by the square of the magnification to give the cross-sectional area of the filament. Calculate the filament diameter from the following expression:

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

where d_e is the equivalent filament diameter
S is the cross-sectional area of the filament

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

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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-polarized light, of wavelength 632,8 mm 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).

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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.

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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.

6.4 Procedure

Measure the interfringe distance to 0,5 mm using a graduated rule, as shown in figure 4. Measure the distance between the specimen and the screen, which shall be greater than 500 mm. Calculate the filament diameter from the expression:

$$d = \frac{\lambda . D}{i}$$

where

d is the filament diameter in micrometres

i is the interfringe distance in millimetres

λ is the wavelength of the laser beam in micrometres

D is the distance between the specimen and the screen in millimetres