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Carbon fibre — Determination of filament diameter and cross-sectional area

Fibres de carbone — Détermination du diamètre et de l'aire de la section transversale des filaments

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 11567:1995), which has been technically revised.

The main changes compared to the previous edition are as follows.

a) Method E, Determination of the diameter by scanning electron microscopy, has been added.

b) The number of the test specimens in method A, method B, method C and method D has been identified.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

This corrected version of ISO 11567:2018 incorporates the following corrections:

— Formula (1) in Clause 6 has been corrected.

Carbon fibre — Determination of filament diameter and cross-sectional area

1 Scope

This document specifies five test methods used for the determination of the diameter and cross-sectional area of single carbon fibre filaments.

The shape of the cross-section of the filaments from different suppliers can vary significantly. The term "diameter" used in this document applies to all cases, from a "true" diameter, where the filament is exactly circular in cross-section, to an "apparent" diameter where the filament is not circular.

The methods proposed are not necessarily directly applicable to all types of filament. The product specification determines the method to be used. If there is no specification, the selection of the appropriate method is a matter of judgement. The details given here are considered to be sufficiently precise to enable this choice to be made.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1888:2006, *Textile glass — Staple fibres or filaments — Determination of average diameter*

ISO 1889, *Reinforcement yarns — Determination of linear density*

ISO 10119, *Carbon fibre — Determination of density*

ISO 11566, *Carbon fibre — Determination of the tensile properties of single-filament specimens*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Five methods are proposed for the determination of the diameter and cross-sectional area of carbon fibre filaments:

— Method A:

Determination of the diameter by calculation.

— Method B:

Determination of the diameter by optical microscopy.

— Method C:

Determination of the diameter and cross-sectional area of transversely cut filaments by microscopy.

— Method D:

Determination of the diameter by laser diffractometry.

— Method E:

Determination of the diameter by scanning electron microscopy.

NOTE Method A gives only an average value of the diameter, which can be sufficient in certain cases, while methods B, C, D and E, which are experimental methods, provide actual values.

5 Test specimens

5.1 General

Because of the intrinsic variability in filament diameter between filaments and along the length of a filament, it is recommended that the diameter or cross-sectional area of 20 filaments in the yarn sample be measured and a statistical analysis of these results carried out.

Test specimens shall be taken from each yarn sample.

5.2 Method A

Yarns are used as test specimens, the amount of yarn taken are specified in ISO 10119 and ISO 1889.

Prepare at least 2 test specimens.

5.3 Methods B and D

Filaments taken from the yarns are used as test specimens, the length of the filaments are approximately 50 mm.

Prepare at least 20 test specimens.

5.4 Method C

Yarns are used as test specimens, the length of yarn taken are approximately 30 mm.

Prepare one test specimen, which consists of at least 20 filaments.

5.5 Method E

Filaments taken from the yarns are used as test specimens, the length of the filaments are determined according to the stage size of the scanning electron microscope.

Prepare at least 20 test specimens.

6 Method A: Determination of the diameter by calculation

An average filament diameter is determined from the linear density of the unsized yarn determined in accordance with ISO 1889, the density determined in accordance with ISO 10119 and the number of filaments in the yarn. The number of filaments in the yarn shall be given by the carbon fibre manufacturer.

Calculate the average filament diameter, d, expressed in micrometres, using Formula (1):

$$d = \sqrt{\frac{4t \times 10^3}{\pi \cdot \rho \cdot c}} \quad (1)$$

where

t is the linear density of the yarn, in tex;

ρ is the density of the yarn, in grams per cubic centimetre;

c is the number of filaments in the yarn.

7 Method B: Determination of filament diameter by optical microscopy

7.1 Principle

The apparent filament diameter is measured by optical microscopy, which gives the distance between the two edges of a filament when the filament is viewed from the side.

The accuracy of method B is limited by diffraction effects, and it is recommended that the method not be used when the filament diameter is less than 10 µm.

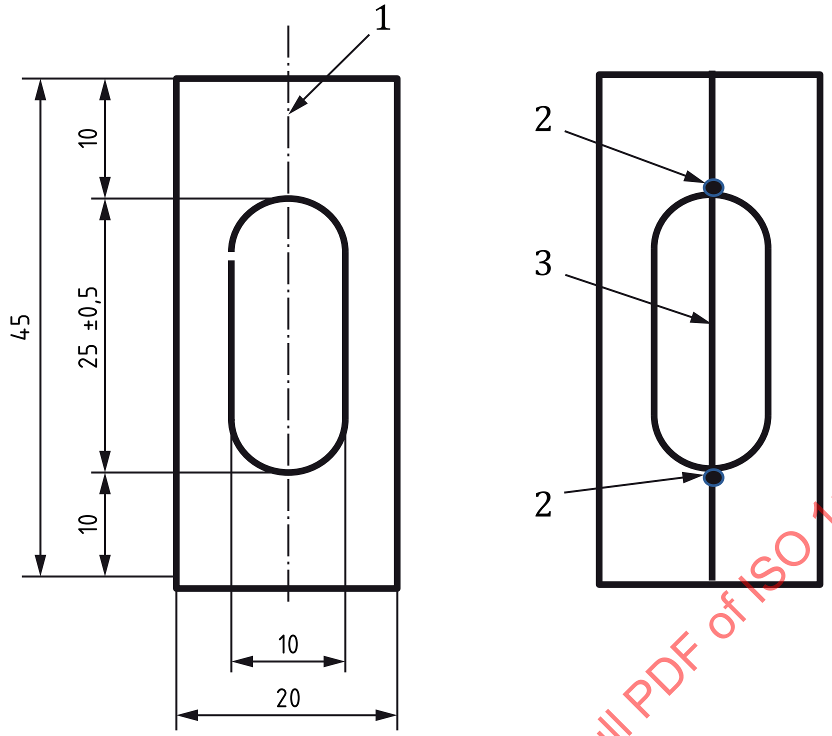
7.2 Apparatus

7.2.1 Microscope, fitted with a light source, a substage condenser, a stage, an objective and a special eyepiece (as described in 7.5). It shall be possible to move the stage in two directions perpendicular to each other in the horizontal plane and to rotate it about the vertical axis.

The objective and eyepiece shall provide magnifications of at least ×100 for looking for filaments and at least ×1 000 for the measurement of the filament diameter.

7.2.2 Specimen mount, with longitudinal slot, as shown in Figure 1.

Dimension in millimetres



Key

1 centreline

2 adhesive

3 single filament test specimen

Figure 1 — Specimen mount and test specimen mounted on the mount

7.3 Calibration of the microscope

Calibrate the microscope using a stage micrometer and a graduated eyepiece divided into hundredths of a millimetre.

7.4 Preparation of the test specimen

7.4.1 If the intention is also to determine the tensile properties of the specimen, proceed in accordance with ISO 11566. Otherwise, proceed as described in 7.4.2 or 7.4.3.

7.4.2 Place a single filament over the centre of the slot in the specimen mount. Attach temporarily one end of the filament to the mount with a piece of adhesive tape. Stretch the filament slightly and attach the other end to the other end of the mount with a second piece of adhesive tape.

Apply one drop of adhesive to the filament at each end of the slot to bond the filament securely in place.

7.4.3 Place specimens whose length does not exceed 25 mm on a glass slide. Place one drop of mounting fluid on the slide to wet the specimens and determine the diameter, as stipulated in ISO 1888:2006, method A.

7.5 Procedure

Because of the intrinsic variability in filament diameter between filaments and along the length of a filament, it is recommended that the diameter be measured three times at three different positions along the filament in order to average the diameter.

Mount the specimen between a slide and a cover slip. Then fill with mounting fluid, if necessary. The mounting fluid shall be chosen to have a refractive index in the range 1,43 to 1,53 at 20 °C, and shall not be hygroscopic and not affect the diameter of the fibre. Cedar oil and paraffin are examples of suitable fluids.

Move the stage of the microscope to position the light beam on the zone of the filament to be examined. Focus on the reticle by means of the eyepiece.

The moving reticle has two fixed wires mounted at right angles to each other, and a double wire running parallel to one of the fixed wires. The reticle can be moved, without changing its orientation, by means of a micrometer screw controlled by a graduated drum which turns past a fixed reference point.

Rotate the microscope eyepiece or stage in order to set the double wire exactly parallel to the axis of the filament under examination. After focusing on the filament, bring the double wire to coincide successively with each side of the image of the filament. Read the number of graduations on the drum necessary to pass from one position to the other (N_r divisions).

7.6 Expression of results

If n is the calibration constant of the eyepiece, i.e. the number of drum divisions corresponding to 1 µm on the micrometer objective, the diameter d, expressed in micrometres, of the filament is given by Formula (2):

$$d = \frac{N_r}{n} \tag{2}$$

where

N_r is the micrometer scale on the drum necessary to pass from one position to other;

n is the calibration constant of the eyepiece.

8 Method C: Determination of the diameter and cross-sectional area of transversely cut filaments by microscopy

8.1 Principle

A carbon fibre yarn is embedded in a resin block. The block is polished on a face normal to the fibre axis and viewed under a microscope. It may also be photographed.

This method is applicable to bundles of parallel fibres. It is especially recommended when the cross-sectional shape of the filaments in the yarn is far from circular.

The accuracy of method C is limited by diffraction of the light or electron beam, optical microscopy is recommended for filaments of diameter greater than 10 µm and electron microscopy for filaments less than 10 µm in diameter.

8.2 Apparatus

8.2.1 Optical microscope and/or scanning electron microscope.

8.2.2 Photographic equipment.

8.2.3 Photographic paper, plastic-coated.

8.2.4 Planimeter.

8.2.5 Electronic image analyser.

8.2.6 Polishing machine, of the type used to prepare metal specimens for microscopic observation.

NOTE The photographic equipment and paper are not always necessary for filaments of circular cross-section, but are necessary for filaments which are non-circular in cross-section.

8.3 Preparation of test specimen

Select a yarn specimen 30 mm in length, embed it in an uncured resin such as an unsaturated-polyester resin, and then cure the resin to give a block. Polish a face of the block perpendicular to the yarn axis by means of the polishing machine.

Carry out the polishing in stages, using abrasive paper and alumina powder or diamond paste. Check the final surface finish by means of an optical microscope.

Details of a suggested preparation procedure for test specimens are given in Annex A.

8.4 Procedure

8.4.1 General

Rotate the microscope eyepiece or stage in order to set the double wire exactly parallel to the axis of the filament under examination. After focusing on the filament, bring the double wire to coincide successively with each side of the image of the filament. Read the number of graduations on the drum necessary to pass from one position to the other (N_r, divisions).

8.4.2 Optical microscopic examination

A reflection microscope, as used in metallography, is suitable, as the face of the specimen is placed on the stage and is thus exactly normal to the optical axis.

In view of the diameters of currently available carbon filaments, use magnifications in the range ×1 000 to ×1 500. If possible, use polarized light, with a crossed polarizer and analyser, and a half-wavelength plate to improve the definition and contrast of the image.

If the filament diameter is to be determined by examination of a photograph, select and photograph a zone typical of the filament population.

Make a positive print, with a magnification of unity or, preferably, enlarged, on plastic-coated photographic paper whose dimensions do not change during development and subsequent processing treatments.

Determine the actual magnification by photographing a micrometer objective under the same conditions.

8.4.3 Scanning electron microscopy

Carry out scanning electron microscopic examinations and photography in accordance with the instructions provided by the manufacturer of the microscope.

8.5 Measurement of the diameter

8.5.1 Filaments of circular cross-section

8.5.1.1 Visual examination

Measure and read off the diameter of each filament selected. Calculate the cross-sectional area from the diameter.

8.5.1.2 Photography

Measure the diameter, on the photographic print, of each filament selected. Calculate the actual diameter by dividing by the magnification. Calculate the cross-sectional area from the diameter.

8.5.2 Filaments of non-circular cross-section

8.5.2.1 Visual examination

Not applicable.

8.5.2.2 Photography

Using a planimeter, measure the cross-sectional area, on the photographic print, of each filament selected. Divide this value by the square of the magnification to give the cross-sectional area S of the filament in square micrometres.

Calculate the "apparent" diameter, d, in micrometres, using Formula (3):

$$d = 2\sqrt{\frac{S}{\pi}} \tag{3}$$

where S is the cross-sectional area.

Measurements of the cross-sectional area and diameter may also be carried out by means of an image analyser. If so, use the image analyser in accordance with the manufacturer's instructions.

9 Method D: Determination of the diameter by laser diffractometry

9.1 Principle

When a filament is irradiated with coherent monochromatic light such as a laser beam, the distance between two diffraction images on a screen is a function of the diameter of the filament. The diameter can be calculated from the distance between the images, wavelength of the light and the focal length of the system.

NOTE Method D is suitable for the determination of the diameter of filaments of circular cross-section. For filaments which are not circular in cross-section, but oval or kidney-shaped, for instance, this method gives an "apparent" diameter.

9.2 Apparatus

9.2.1 He-Ne laser transmitter, of 2 mW power, or any other type of transmitter.

9.2.2 Specimen holder, designed as a goniometer with an attachment to hold a specimen mount (7.2.2).

9.2.3 Screen, made of white cardboard.

9.2.4 Rule, graduated in millimetres.

9.3 Preparation of test specimen

The test specimen is the same as that for method B (see 7.4.2).

9.4 Procedure

Because of the intrinsic variability in filament diameter between filaments and along the length of a filament, it is recommended that the diameter be measured three times at three different positions along the filament in order to average diameter.

Place the specimen mount in the specimen holder so that the filament is in the laser beam path (the diameter of the beam is about 0,5 mm, so it is not difficult to place the filament in the right position).

Measure, with the rule, the distance between the two dark zones nearest to the centre of the diffraction pattern on the screen. (The diffraction pattern on the screen has a width of about 0,5 mm, and measuring this distance is not difficult.)

Rotate the specimen about its axis by 15°, using the goniometer to determine the angle, and repeat the measurement. Repeat this procedure at 15° intervals up to an angle of 165° in order to obtain the average diameter.

9.5 Expression of results

For each measurement, calculate the filament diameter, d, expressed in micrometres, using Formula (4):

$$d = \frac{\lambda D}{l} \tag{4}$$

where

λ is the wavelength, in micrometres, of the laser light (in the case of an He-Ne laser, A = 0,632 µm);

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

l is half of the distance, expressed in millimetres, between the two dark zones nearest to the centre of the diffraction pattern.

10 Method E: Determination of filament diameter by scanning electron microscopy

10.1 Principle

The apparent filament diameter is measured by scanning electron microscope, which gives the distance between the two edges of a filament when the filament is viewed from the side.

The accuracy of method E is limited by diffraction of the electron beam, electron microscopy is recommended for filaments of diameter less than 10 µm in diameter.

10.2 Apparatus

10.2.1 Scanning electron microscope.