INTERNATIONAL STANDARD

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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

Reference number
ISO 11567:1995(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 11567 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Annex A of this International Standard is for information only.

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

1 Scope

This International Standard specifies four test methods which may be used for the determination of the diameter and cross-sectional area of single carbon fibre filaments.

It is important to note that the shape of the cross-section of the filaments from different suppliers may vary significantly. The term "diameter" used in this standard 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 may not be directly applicable to all types of filament. The product specification should specify which method should 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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 10119:1992, *Carbon fibre — Determination of density.*

ISO 10120:1991, *Carbon fibre — Determination of linear density.*

ISO 11566:—[1], *Carbon fibre — Determination of the tensile properties of single-filament specimens.*

3 Principle

Four 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

NOTE 1 Method A gives only an average value of the diameter, which may be sufficient in certain cases, while methods B, C and D, which are experimental methods, provide actual values.

4 Test specimens

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.

1) To be published.

4.1 Method A

Yarns are used as test specimens, the amount of yarn taken being as specified in ISO 10119 and ISO 10120.

4.2 Methods B and D

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

4.3 Method C

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

5 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 10120, 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 that given by the carbon fibre manufacturer.

Calculate the average filament diameter d, expressed in micrometres, from the following equation:

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

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.

6 Method B: Determination of filament diameter by optical microscopy

6.1 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.

NOTE 2 The accuracy of method B is limited by diffraction effects, and it is recommended that the method is not used when the filament diameter is less than 10 µm.

6.2 Apparatus

6.2.1 Microscope, fitted with a light source, a substage condenser, a stage, an objective and a special eyepiece (as described in 6.5). 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.

6.2.2 Specimen mount, with longitudinal slot, as shown in figure 1.

Dimensions in millimetres

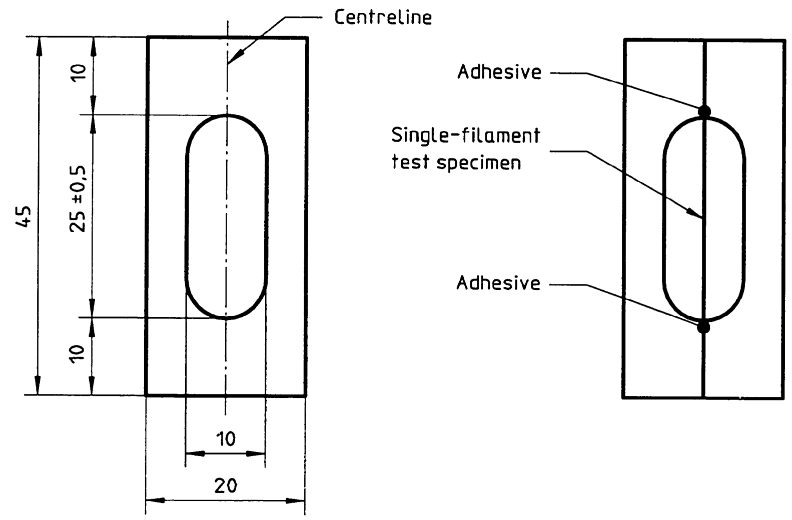

Figure 1 — Specimen mount and test specimen mounted on the mount

6.3 Calibration of the microscope

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

6.4 Preparation of the test specimen

6.4.1 If the intention is also to determine the tensile properties of the specimen, proceed in accordance with ISO 11566. Otherwise, proceed as described in 6.4.2.

6.4.2 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.

6.5 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.

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 focussing 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).

6.6 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 the following equation:

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

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

7.1 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, but can also be used without modification to examine the distribution of the fibres in a unidirectional composite, as well as for the measurement of the fibre content by volume. It is specially recommended when the cross-sectional shape of the filaments in the yarn is far from circular.

NOTE 3 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.

7.2 Apparatus

7.2.1 Optical microscope and/or **scanning electron microscope.**

7.2.2 Photographic equipment.

7.2.3 Photographic paper, plastic-coated.

7.2.4 Planimeter.

7.2.5 Electronic image analyser.

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

NOTE 4 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.

7.3 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 is given in annex A.

7.4 Procedure

Rotate the microscope eyepiece or stage in order to set the double wire exactly parallel to the axis of the filament under examination. After focussing 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).

7.4.1 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.

7.4.2 Scanning electron microscopy

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

7.5 Measurement of the diameter

7.5.1 Filaments of circular cross-section

7.5.1.1 Visual examination

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

7.5.1.2 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.

7.5.2 Filaments of non-circular cross-section

7.5.2.1 Visual examination

Not applicable.

7.5.2.2 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, from the following equation:

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

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.

8 Method D: Determination of the diameter by laser diffractometry

8.1 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 5 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.

8.2 Apparatus

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

8.2.2 Specimen holder, designed as a goniometer with an attachment to hold a specimen mount (6.2.2).

8.2.3 Screen, made of white cardboard.

8.2.4 Rule, graduated in millimetres.

8.3 Preparation of test specimen

The test specimen is the same as that for method B (see 6.4).

8.4 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.

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.

8.5 Expression of results

For each measurement, calculate the filament diameter d, expressed in micrometres, from the following equation:

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

where

λ is the wavelength, in micrometres, of the laser light (in the case of an He-Ne laser, λ = 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.

9 Precision

The precision of this method is not known because interlaboratory data are not available. Interlaboratory data are being obtained and a precision statement will be added at a subsequent revision.

10 Test report

The test report shall include the following information:

a) a reference to this International Standard and the method used (A, B, C or D);

b) all details necessary for identification of the fibre tested;

c) the filament diameters, in micrometres, and/or cross-sectional areas, in square micrometres, for the specimens tested;

d) details of any additional measures taken to facilitate the measurement procedure.

Annex A
(informative)

Suggested method for the preparation of test specimens for method C

A.1 Apparatus and materials

A.1.1 Embedding resin, for example an unsaturated-polyester resin, an epoxy resin or a mixture of unsaturated-polyester resin and acrylic resin, room-temperature cure type.

A.1.2 Disposable plastic beaker, capacity 100 ml to 200 ml.

A.1.3 Glass or plastic tube, diameter approximately 30 mm, length approximately 20 mm.

A.1.4 Glass plate, with a flat, smooth surface, measuring 300 mm × 300 mm.

A.1.5 Double-sided adhesive tape.

A.1.6 Rotary polishing machine, with a water spray, as commonly used to prepare metal specimens for microscopic examination.

A.1.7 Abrasive paper, water-resistant quality, of various abrasive-grain sizes (grades 100 to 800).

A.1.8 Polishing cloth, made of viscose satin or shammy leather.

A.1.9 Abrasive powder (alumina powder or diamond paste), as commonly used to prepare metal specimens for microscopic examination.

A.2 Procedure

Attach a piece of adhesive tape (A.1.5) measuring approximately 40 mm × 40 mm to one side of the glass plate (A.1.4).

Apply a release agent such as silicone grease to the inside of the glass or plastic tube (A.1.3) to facilitate subsequent removal of the block of resin from the tube. Press one end of the tube against the piece of adhesive tape on the glass plate so that the tube adheres to the glass plate.

With the glass plate on a horizontal surface so that the tube is vertical, suspend a yarn specimen so that it hangs down into the tube. It is recommended that this be done by attaching the yarn to a length of metal wire which is then placed across the top of the tube. Several specimens can be hung in the same tube.

Prepare the embedding resin (A.1.1) by mixing the resin, hardener and catalyst in a plastic beaker (A.1.2).

Pour the embedding resin into the tube, and keep it at room temperature until the resin cures to a solid. Further curing in an oven at elevated temperatures is allowable if the resin does not cure sufficiently for the rest of the procedure to be carried out.

Remove the block of resin containing the embedded specimen from the tube.

Polish one end of the block of resin on the polishing machine (A.1.6), using grade 100 to 150 abrasive paper (see A.1.7) and the water spray.

Change the abrasive paper to a finer grain size (up to grade 800) and continue polishing.

Finally, polish on the polishing machine (A.1.6) with a suspension of alumina powder or with diamond paste (A.1.9), using the polishing cloth (A.1.8). Continue polishing until no scratches can be detected on the polished surface when examined under an optical microscope at a magnification of ×1 500.

Typical examples of the conditions used for this stage are as follows:

— turntable speed: 200 rpm;

— load on the block of resin: 1 N to 2 N;

— concentration of abrasive in the suspension: 2 g/l to 5 g/l;

— rate of flow of suspension: 20 to 40 drops per minute;

— time: 5 h.

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