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Carbon fibre — Determination of tensile properties of resin-impregnated yarn

Fibres de carbone — Détermination des propriétés en traction sur fils imprégnés de résine

Reference number
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Contents

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 10618 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 13, *Composites and reinforcement fibres*.

Annexes A to D of this International Standard are for information only.

Carbon fibre — Determination of tensile properties of resin-impregnated yarn

1 Scope

This International Standard specifies a method of test for the determination of the tensile strength, tensile modulus of elasticity and strain at maximum load of a resin-impregnated yarn specimen. The method is applicable to yarns (continuous and staple-fibre yarns) of carbon fibre for use as reinforcements in composite materials.

The tensile modulus of elasticity may be calculated by one of two methods, A and B. The result obtained will not necessarily be the same in each case.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 527-1:1993, *Plastics — Determination of tensile properties — Part 1: General principles.*

ISO 1886:1990, *Reinforcement fibres — Sampling plans applicable to received batches.*

ISO 1889:1997, *Reinforcement yarns — Determination of linear density.*

ISO 10119:—[1)], *Carbon fibre — Determination of density.*

ISO 10548:1994, *Carbon fibre — Determination of size content.*

3 Terms and definitions

For the purposes of this International Standard, the terms and definitions given in ISO 527-1 apply, with the following addition:

3.1
cross-sectional area of a yarn
A_f
the linear density of a yarn divided by the density of the material in the yarn

NOTE It is expressed in square metres.

1) To be published. (Revision of ISO 10119:1992)

4 Symbols

The symbols used in this International Standard are as follows:

σ_f tensile strength, in megapascals;

F_f maximum tensile force, in newtons;

A_f cross-sectional area of yarn, in square metres;

ρ_f density of yarn, in kilograms per cubic metre;

T_{tf} linear density of yarn, in tex;

T_{ti} linear density of impregnated yarn, in tex;

E_f tensile modulus of elasticity, in gigapascals;

L_0 extensometer gauge length, in millimetres;

L_u extensometer reading, in millimetres, at maximum load;

ΔL variation in the length, in millimetres, corresponding to the variation in the force, in newtons;

ΔF variation in the force, in newtons, corresponding to the variation in the length, in millimetres.

5 Principle

A sample of yarn is uniformly impregnated with resin, then cured to provide thin composite rod test specimens. The specimens are loaded in tension at a constant speed by a suitable mechanical-testing machine until failure in order to determine the load-extension curve.

The tensile strength, the tensile modulus of elasticity and the strain at maximum load are calculated from the force-extension relationship.

The tensile modulus is determined by dividing the variation in the stress by the corresponding variation in the strain between two points. For carbon-fibre yarns, the relation between stress and strain is not linear, hence a chord modulus must be defined. In method A, the modulus is defined between two stress levels, whereas in method B it is defined between two strain levels. For the purposes of this International Standard, method B is the reference method. The linear density and the size content have to be determined independently.

NOTE The precision of the values obtained is the same for method A and for method B. Method A, or other methods, may be used for purchase-specification or quality-control purposes by agreement between customer and supplier.

6 Apparatus and materials

6.1 Impregnating resin, compatible with the yarn and its size. The viscosity of the resin or resin solution shall be such that sufficient resin pick-up is achieved to ensure uniform impregnation. The strain at failure of the cured resin shall be at least twice that of the fibre, preferably three times. In this respect, heat-curable epoxy-resin systems with a viscosity during impregnation of preferably less than 1 000 mPa·s are suitable (see annex A for example), as is any formulation capable of giving test specimens that fulfil the requirements of this International Standard. The resin formulation, however, shall be specified in detail and shall be agreed upon between the fibre manufacturer and the user.

6.2 Impregnation apparatus.

Test specimens can be prepared by any method which produces a uniformly impregnated, smooth specimen.

These methods include both single- and multiple-specimen preparation techniques. A multiple-specimen impregnation apparatus may consist of the following:

6.2.1 Holder for the yarn-feed bobbin, with yarn-tensioning devices.

6.2.2 Impregnation bath, with temperature-control devices and impregnation rollers or yarn-tensioning bars.

6.2.3 Unit to remove excess resin from the impregnated yarn by passing it over rollers covered with fabric, paper or felt and/or through a die.

6.2.4 Frame, to wind up the impregnated yarn, preferably made of wood or metal coated with rubber.

6.3 Curing oven, with temperature control. A fan circulation oven is preferred to ensure uniform curing of the resin.

The temperature of the curing oven shall be controlled at ± 3 °C.

6.4 Tensile-testing machine, with a constant crosshead speed, equipped with force- and extension-recording devices. The accuracy of the force indication shall be better than 1 % of the recorded value. The specimen-gripping system shall ensure that the test specimen is aligned with the axis of the test machine.

6.5 Extensometer linked to a continuous-recording device, which automatically records the extension within the gauge length of the extensometer as a function of the force acting on the test specimen. The extensometer shall be sufficiently light to induce only negligible stresses in the test specimen.

The extensometer gauge length shall be at least 50 mm, but preferably 100 mm. The tolerance on the gauge length shall be ±1 %.

The extensometer shall have a measurement tolerance of not more than 0,1 % over the required extension-measurement range.

Examples of suitable extensometers are given in annex D. Other strain-measuring equipment, such as optical or laser instruments, may be used, if suitable.

6.6 Balance, accurate to 0,1 mg, to weigh the test specimens in order to determine the linear density of the impregnated yarn.

6.7 Graduated rule, at least 500 mm long and accurate to ± 1 mm, or **other suitable measuring device.**

7 Test specimens

7.1 Number of test specimens

Prepare sufficient test specimens to enable five valid determinations to be made. If any of the specimens fails within the grips or at the tabs, or because of damage caused by the extensometer, discard the result and carry out a repeat on a fresh test specimen.

7.2 Length of test specimens

For test specimens with tabs, the length of the test specimen between the tabs shall be either 150 mm ± 5 mm or 200 mm ± 5 mm. For test specimens without tabs, the total length of the test specimen shall be 250 mm ± 5 mm or 300 mm ± 5 mm (at least the extensometer gauge length plus twice the grip length).

In cases of dispute, for test specimens with tabs the length between the tabs shall be 150 mm ± 5 mm and for test specimens without tabs the length of the test specimen shall be 250 mm ± 5 mm.

7.3 Preparation of test specimens

The procedure using the impregnation apparatus described in 6.2 is as follows:

Place the yarn bobbin on the holder.

Pour the impregnating-resin mixture into the resin bath and adjust the temperature and viscosity to the desired values.

Draw the yarn through the resin bath and through the system designed to remove the excess resin while ensuring adequate resin impregnation.

Adjust the unwinding tension to approximately 3 mN/tex.

Wind the impregnated yarn on to the frame.

Place the frame in the oven.

Cure the resin in accordance with the resin manufacturer's instructions.

When the resin has been cured, remove the frame from the oven. After removal of the impregnated yarn from the frame, cut off a sufficient number of specimens.

Select the test specimens according to the criteria given in 7.5.

7.4 Determination of other fibre properties

7.4.1 General

In order to be able to calculate the tensile strength and tensile modulus as described in clause 10, a number of other fibre properties must first be determined.

7.4.2 Linear density of yarn

Determine the linear density of the yarn by the method given in ISO 1889.

7.4.3 Size content of yarn

Determine the size content of the yarn by the method given in ISO 10548.

7.4.4 Density of the carbon fibre

Determine the density of the carbon fibre by one of the methods given in ISO 10119.

7.4.5 Linear density of impregnated-yarn test specimen

Using the rule or other suitable device, measure the length of a test specimen after it has been cut to length and prior to tabbing. Weigh the test specimen on the balance. Calculate the linear density by dividing the mass of the test specimen by its length, expressing the result in grams per kilometre (tex).

7.5 Criteria for selection of test specimens

7.5.1 Each test specimen shall be confirmed as straight when checked using a suitable jig. It shall be uniform in appearance and without any of the following defects:

— broken filaments;

— resin droplets;

— fibre misalignment.

7.5.2 The resin content shall be at least 35 % by mass. The resin content of the specimens can be calculated, in mass percent, from the linear density of the test specimen T_{ti}, in tex, and the linear density of the unimpregnated yarn T_{tf}, in tex, from the equation

$$\text{Resin content} = \frac{T_{ti} - T_{tf}}{T_{ti}} \times 100$$

7.5.3 The yarn shall be uniformly impregnated.

7.6 Tabbing of test specimens

If a test specimen fails within the grips of the tensile-testing machine, the result is not valid. Fixing tabs to the test specimen helps reduce the frequency of such failures. The tabs also help assure correct alignment of the test specimen in the grips.

Specimens can nevertheless be used with or without tabs.

If tabs are necessary, the equipment required depends on the type of tab chosen. In all cases where tabs are used, the gripped length shall be at least 30 mm. See annex C for examples.

8 Atmosphere for conditioning and testing

The atmosphere used for conditioning and testing shall be selected from those defined in ISO 291.

9 Procedure

9.1 Set the crosshead speed of the tensile-testing machine to cause specimen failure in not less than 3 min (for quality-assurance purposes only, a crosshead speed of up to 50 mm/min may be used).

9.2 For test specimens with tabs, install grips which fit the type of tab used. Adjust the distance between the grips to the prescribed specimen length (see 7.2).

For test specimens without tabs, install grips which are equipped with flat faces made of sheet materials of moderate elasticity and high coefficient of friction such as hard rubber sheet. The sheet can be bonded onto the metal faces of the grips with a suitable adhesive. If test specimens are found to slip within the grips during the test, it has been found useful to insert abrasive paper between the test specimen and the faces of the grips.

Because the test specimens are so fragile, it is recommended that the grip system is actuated by compressed air.

9.3 Clamp a test specimen between the grips of the test machine.

9.4 Fix the extensometer carefully to the test specimen.

9.5 Start the recording device and load the test specimen to failure.

9.6 If the specimen fails within the grips or at the tabs, or because of damage caused by the extensometer, discard the result and carry out a repeat on a fresh test specimen.

10 Expression of results

10.1 Tensile strength

10.1.1 For each test specimen, calculate the tensile strength of the yarn σ_f, in megapascals, from the following equation:

$$\sigma_f = \frac{F_f}{A_f} \times 10^{-6}$$

where

F_f is the maximum tensile force, in newtons;

A_f is the cross-sectional area of the yarn, in square metres, given by the equation:

$$A_f = \frac{T_{tf}}{\rho_f} \times 10^{-6}$$

T_{tf} being the linear density of the unsized yarn, in tex, calculated from the linear density determined in accordance with ISO 1889 and the size content determined in accordance with ISO 10548;

ρ_f being the density of the yarn, in kilograms per cubic metre, determined in accordance with ISO 10119.

10.1.2 Calculate the arithmetic mean of five individual tensile-strength determinations and report this mean as the result. If required by the product specification or by the person requesting the test, calculate the standard deviation and coefficient of variation of the individual determinations using normal statistical calculation methods.

10.2 Tensile modulus of elasticity (see Figure 1)

10.2.1 Method A

The tensile modulus of elasticity E_f as determined by method A is calculated, in gigapascals, from the following equation:

$$E_f = \frac{\Delta F}{A_f} \times \frac{L_0}{\Delta L} \times 10^{-9}$$

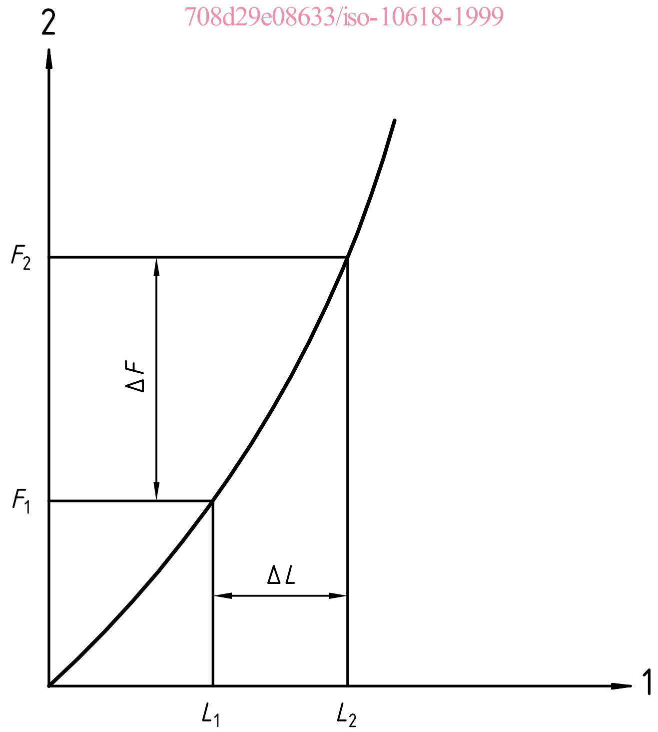
where

ΔF is the variation in the force, in newtons, corresponding to the variation in the length, in millimetres, between 400 mN/tex and 800 mN/tex;

A_f is the cross-sectional area of the yarn, in square metres (see 10.1.1);

L_0 is the extensometer gauge length, in millimetres;

ΔL is the variation in the length, in millimetres, corresponding to the variation in the force between 400 mN/tex and 800 mN/tex.



Key

1 Extension (mm)
2 Force (N)

Figure 1 — Force-extension relationship during tensile testing

10.2.2 Method B

Method B is the reference method for determining the tensile modulus of elasticity E_f. The modulus is calculated, in gigapascals, from the following equation:

$$E_f = \frac{\Delta F}{A_f} \times \frac{L_0}{\Delta L} \times 10^{-9}$$

where

ΔF is the variation in the force, in newtons, corresponding to the variation in the length, in millimetres, between the strain limits given in Table 1 for the fibre type concerned;

A_f is the cross-sectional area of the yarn, in square metres (see 10.1.1);

L_0 is the extensometer gauge length, in millimetres;

ΔL is the variation in the length, in millimetres, corresponding to the variation in the force between the strain limits given in Table 1 for the fibre type concerned.

Table 1 — Relationship between fibre type and strain limits

Value of strain at break ε typical of fibre	Strain limits
$\varepsilon \geqslant$ 1,2 %	0,1 % to 0,6 %
0,6 % $\leqslant \varepsilon <$ 1,2 %	0,1 % to 0,3 %
0,3 % $\leqslant \varepsilon <$ 0,6 %	0,05 % to 0,15 %
NOTE The typical value of the strain at break (percent elongation at maximum load) may be determined by extensometry or calculated from typical strength and tensile-modulus values for the type of carbon fibre under test.	

10.2.3 Calculate the arithmetic mean of five individual tensile-modulus determinations and report this mean as the result. It will be assumed that method B, the reference method, has been used unless it is specifically stated in the test report that method A or another method has been used.

If required by the product specification or by the person requesting the test, calculate the standard deviation and coefficient of variation of the individual determinations using normal statistical calculation methods.

10.3 Strain at maximum load (percent elongation at failure)

The strain at maximum load ε is calculated from the following equation:

$$\varepsilon = \frac{L_u - L_0}{L_0}$$

where

L_0 is the extensometer gauge length, in millimetres;

L_u is the extensometer reading, in millimetres, at maximum load.

The strain at maximum load is usually given in micrometres per metre. The percent elongation at failure is obtained by multiplying the strain at maximum load by 100.