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Carbon-fibre-reinforced composites — Determination of the resin, fibre and void contents

Composites renforcés de fibres de carbone — Détermination des teneurs en résine, en fibre et en vide

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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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 14127:2008), which has been technically revised.

The main changes are as follows:

- the new method: Method C (microscopic method) as means to determinate fibre content by volume and areal void content has been added;
- technical details related to the new method have been edited;
- procedure A3 has been modified by replacing the heating plate, beaker and watch glass with a heating mantle and round bottom flask;
- in subclauses 4.3, 6.3 and 7.3, where provisions for the number of test samples per assessment have been newly added;
- " m_r " has been corrected to " φ_r " in Formula (4).

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.

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Introduction

The constituent contents such as fibre content, resin content as well as void content are parameters characterize physical/structural properties of carbon-fibre-reinforced composites. Such properties are proven to have influence on mechanical performances of carbon-fibre-reinforced composites; thus, the constituent contents are always required as important index for processing quality control.

Microscopic method calculates the fibre volume content/areal void content from measured fibre area/void area and the area of cross-section of a specimen. The principle of this method differs from method A or B, so that method C might be available when some of the types of resin were so hard to remove or in the case of lacking information of the prepregs. Meanwhile, thanks to the development of electronic and information technology, equipment integrated with image analysis functions is commercially available. The microscopic method has multiple advantages of being efficient, safe, commercial and environment friendly.

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Carbon-fibre-reinforced composites — Determination of the resin, fibre and void contents

1 Scope

This document specifies methods for calculating the resin, fibre and void contents of a carbon-fibre-reinforced composite from the densities of the resin, the fibre and the composite and the mass of fibre in the composite (using method A), for calculating the fibre content from the thickness of the composite (using method B), and for calculating the fibre content by volume and areal void content through microscopic analysis (using method C).

Method A specifies three different resin removal procedures for the determination of the mass of fibre in the composite (viz a combustion procedure, a procedure by digestion in nitric acid and a procedure by digestion in a mixture of sulfuric acid and hydrogen peroxide). The selection of the procedure to be used is made by considering the combustibility of the resin used in the composite, its ability to decompose and the type of resin concerned. Method A is only of limited applicability when filled resins are present that can prevent complete dissolution and/or combustibility of the resin.

Method B (thickness measurement method) is only applicable to composites moulded from prepregs of known fibre mass per unit area.

Method C (microscopic method) is only applicable to carbon-fibre-reinforced composites with unidirectional, orthogonal and multidirectional laminates. It can also be used as reference for determination of the areal void content and fibre volume content of aramid- or glass-fibre-reinforced plastics, but is not applicable to fabric reinforced composites.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

ISO 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pycnometer method and titration method*

ISO 1183-2, *Plastics — Methods for determining the density of non-cellular plastics — Part 2: Density gradient column method*

ISO 1183-3, *Plastics — Methods for determining the density of non-cellular plastics — Part 3: Gas pyknometer method*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 9344, *Microscopes — Graticules for eyepieces*

ISO 10119, *Carbon fibre — Determination of density*

ISO 10934, *Microscopes — Vocabulary for light microscopy*

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472, ISO 10934 and the following apply.

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 https://www.electropedia.org/

3.1
fibre content by mass
ratio of the mass of fibre in a composite to the total mass of the composite

Note 1 to entry: It is expressed as a percentage.

3.2
fibre content by volume
ratio of the volume of fibre in a composite to the total volume of the composite

Note 1 to entry: It is expressed as a percentage.

3.3
void content
ratio of the volume of the voids (hollow spaces) in a composite to the total volume of the composite

Note 1 to entry: It is expressed as a percentage.

3.4
areal void content
ratio of the total area of the voids (hollow spaces) on the whole observed cross-section of the specimen taken from the composite sample

Note 1 to entry: It is expressed as a percentage.

4 Test specimens — General requirements

4.1 The locations from which the test specimens are taken shall be distributed randomly over the sample and be no nearer than 10 mm to any edge.

4.2 Delamination and cracking shall be prevented during the machining process. The edges of the test specimens shall be ground square and smoothed with abrasive paper (6.2.3).

4.3 At least three test specimens shall be taken unless otherwise specified by the party requesting the test.

5 Conditioning

This conditioning shall be carried out in one of the standard atmospheres specified in ISO 291.

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6 Method A: Resin removal method

6.1 Principle

6.1.1 Procedure A1: Combustion procedure

The mass of a test specimen is determined before and after combustion of the resin in the upper part of the reducing (non-oxygen) flame of a Bunsen burner.

NOTE The combustion procedure makes use of the relative ease of decomposition of resins, compared to carbon fibres, in inert gases. The procedure consists of heating a specimen of composite material with the reducing flame of a Bunsen burner so that only the resin is removed by combustion. However, its application is limited to resins that decompose completely by combustion. Therefore, this procedure is not applicable to resins that are not completely combustible, such as epoxy novolac and brominated systems. There are also drawbacks such as the fact that the accuracy of the combustion procedure is slightly inferior to that of the nitric acid digestion procedure and the sulfuric acid/hydrogen peroxide digestion procedure. Nonetheless, it is useful as a rapid test procedure which can be carried out safely and simply.

Because of the lack of reliability of the combustion procedure, its use shall be as agreed between the purchaser and supplier.

6.1.2 Procedure A2: Nitric acid digestion procedure

The mass of a test specimen is determined before and after digestion of the resin with concentrated nitric acid, which does not attack the carbon fibres excessively.

NOTE Both the nitric acid digestion procedure and the sulfuric acid/hydrogen peroxide digestion procedure make use of the fact that digestion of resins in a hot bath of nitric acid or sulfuric acid/hydrogen peroxide mixture is rapid compared to carbon fibres (which resist digestion in such conditions). The procedure consists of soaking the composite in a hot bath of one of these reagents so that only the resins are removed by digestion. The nitric acid digestion procedure is applicable to all epoxy resins except acid anhydride curing substances. The sulfuric acid/hydrogen peroxide digestion procedure is applicable to all epoxy resins, phenolic resins and polyamide resins.

6.1.3 Procedure A3: Digestion in a sulfuric acid/hydrogen peroxide mixture

The mass of a test specimen is determined before and after digestion of the resin with an aqueous mixture of sulfuric acid and hydrogen peroxide, provided that the carbon fibres are not attacked.

See also the Note to 6.1.2.

6.2 Apparatus and reagents

6.2.1 General

6.2.1.1 Desiccator, containing a suitable drying agent (e.g. silica gel).

6.2.1.2 Analytical balance, accurate to 0,1 mg.

6.2.1.3 Abrasive paper, with grain size finer than 180 grit.

6.2.2 Procedure A1

6.2.2.1 Bunsen burner, compatible with the gas used.

6.2.2.2 Nichrome wire, about 0,2 mm in diameter.

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6.2.3 Procedure A2

6.2.3.1 Borosilicate-glass vacuum filter.

6.2.3.2 200 ml conical flask.

6.2.3.3 100 ml measuring cylinder.

6.2.3.4 Water reflux condenser, with a standard taper joint to fit the conical flask.

6.2.3.5 Air-circulation drying oven, capable of heating up to about 200 °C.

6.2.3.6 Acetone, as specified in ISO 6353-2.

6.2.3.7 Concentrated nitric acid, 62 % by mass.

6.2.4 Procedure A3

6.2.4.1 Borosilicate-glass vacuum filter.

6.2.4.2 Borosilicate-glass round bottom flask, minimum volume 200 ml.

6.2.4.3 100 ml measuring cylinder.

6.2.4.4 Reflux condenser, with a standard taper joint to fit the round bottom flask.

6.2.4.5 Heating mantle, capable of heating up to about 200 °C.

6.2.4.6 Air-circulation drying oven, capable of heating up to about 200 °C.

6.2.4.7 Acetone, as specified in ISO 6353-2.

6.2.4.8 Concentrated sulfuric acid, 96 % by mass.

6.2.4.9 30 % to 35 % hydrogen peroxide solution.

SAFETY PRECAUTIONS — When handling nitric acid, sulfuric acid or hydrogen peroxide, care shall be taken to prevent accidents. Wear rubber gloves, protective goggles, etc., in order to avoid direct contact of these reagents with the human body. If human skin does come into contact with one of these reagents, appropriate treatment shall be provided immediately. It is also necessary to ventilate the test area and/or room. Boiling 65 % hydrogen peroxide can decompose explosively; do not therefore allow it to concentrate by distillation. Waste chemicals should be kept separate and disposed of in a suitable way.

6.3 Test specimen

The mass of the test specimens shall be at least 0,5 g and preferred of cuboid shape. The recommended dimensions are a thickness of less than 4 mm with 6 mm to 10 mm in length and width. Test at least 5 specimens every series for assessment of void content and at least 3 specimens every series for assessment of fibre volume content.

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6.4 Procedure

6.4.1 Density measurements

When using method A, the densities of the carbon fibre, the resin and the composite are needed for calculating the fibre content by volume, the resin content by volume and the void content by volume Determine the densities of the resin, the composite and the carbon fibre as follows.

a) Determine the density of the resin (ρ_r) in accordance with ISO 1183-1, ISO 1183-2 or ISO 1183-3.

b) Determine the density of the carbon fibre (ρ_f) in accordance with ISO 10119.

c) Determine the density of the composite (ρ_c) by the immersion method specified in ISO 1183-1. Use water as the immersion liquid.

   When measuring resin and composite densities by the immersion method, it is essential that a correction be made for the mass of the wire used to suspend the specimen.

6.4.2 Procedure A1

6.4.2.1 Weigh a test specimen with the analytical balance (6.2.1.2) to the nearest 0,1 mg (m).

6.4.2.2 Bind the specimen with nichrome wire (6.2.2.2) as shown in Figure 1. Weigh the specimen, including the nichrome wire, to the nearest 0,1 mg (m_1).

6.4.2.3 Light the Bunsen burner (6.2.2.1) with the air shut off. Adjust the height of the flame to between 15 cm and 20 cm. Gradually increase the supply of air to bring the height of the reducing flame (blue flame) to about 2 cm. Using tweezers to hold the nichrome wire binding the specimen, introduce the specimen horizontally into the flame a little above the top of the reducing zone of the flame, but without at any time touching the reducing zone, as shown in Figure 1.

6.4.2.4 Within five to twenty seconds, most of the resin in the specimen will be burnt off, producing a red flame and black soot. Continue to heat the specimen until its bottom surface becomes red hot. Then invert it until the other surface becomes red hot. When all the resin has been burnt off, the red flame will disappear. Continue heating until this happens. The total heating time will normally be 3 min or less.

6.4.2.5 Remove the specimen from the flame and place it on a metal plate to cool to room temperature. If the specimen is not weighed at once, place the plate plus specimen in a desiccator (6.2.1.1).

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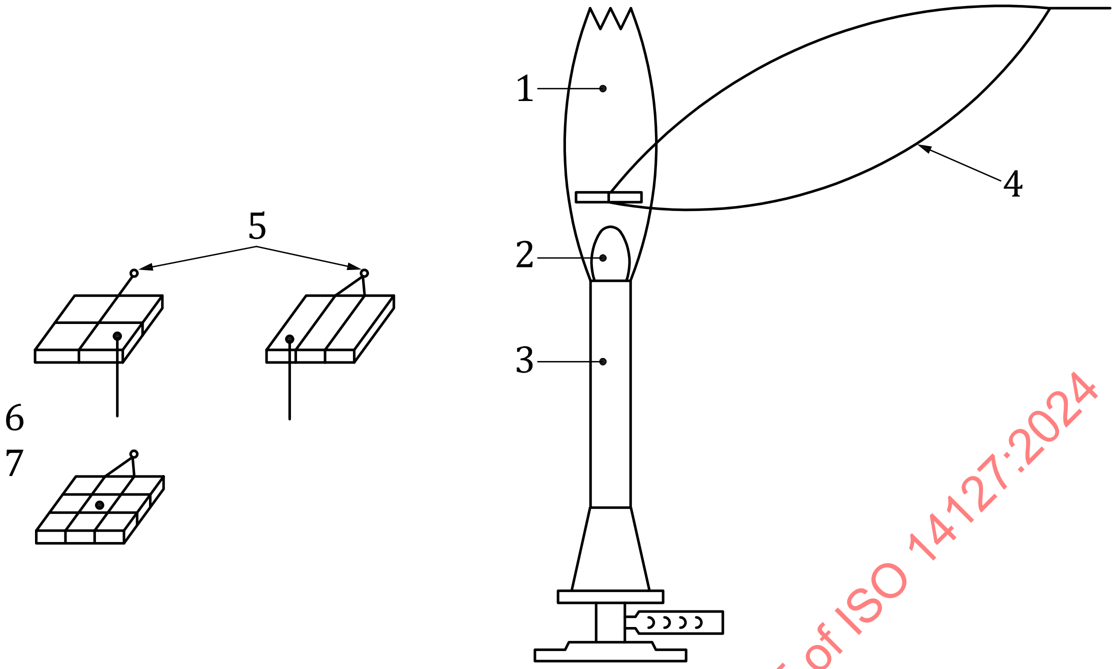
6.4.2.6 Weigh the specimen, including the nichrome wire, to the nearest 0,1 mg (m_2).



Key

1	flame
2	reducing zone of flame
3	bunsen burner
4	tweezers
5	nichrome wire
6	specimen reinforced with unidirectional carbon fibres
7	specimen reinforced with woven carbon fibre fabric

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Figure 1 — Procedure A1

6.4.3 Procedure A2

6.4.3.1 Weigh a test specimen with the analytical balance (6.2.1.2) to the nearest 0,1 mg (m).

6.4.3.2 Using a measuring cylinder (6.2.3.3), measure 50 ml of concentrated nitric acid (6.2.3.7) into a 200 ml conical flask (6.2.3.2).

6.4.3.3 Place the flask in an oil bath maintained at 120 °C ± 2 °C and place the test specimen in the flask.

6.4.3.4 Attach a reflux condenser (6.2.3.4) to the flask and heat the flask for 90 min.

6.4.3.5 Dry a glass filter (6.2.3.1) in an air-circulation oven (6.2.3.5) at 105 °C ± 5 °C for 90 min, then cool it in a desiccator to room temperature and weigh it to the nearest 0,1 mg. Continue drying, cooling and weighing until constant mass is reached, i.e. until two consecutive weighings do not differ by more than 1 mg. Label this mass as m_3.

6.4.3.6 After digestion, cool the solution and filter it through the glass filter, using vacuum suction, to separate off the fibre.

6.4.3.7 Wash out the flask with 20 ml to 30 ml of concentrated nitric acid, filtering the washings through the glass filter.

6.4.3.8 Wash the fibre retained on the filter with several approximately 100 ml portions of distilled water from a washing bottle and then with acetone (6.2.3.6).

6.4.3.9 Place the glass filter in an air-circulation oven and dry it at 105 °C ± 25 °C for 90 min.

6.4.3.10 After drying, remove the glass filter from the oven and place it in a desiccator for 30 min to cool to room temperature.

6.4.3.11 Weigh the glass filter plus fibre to the nearest 0,1 mg.

6.4.3.12 Continue drying, cooling and weighing until constant mass is reached, i.e. until two consecutive weighings do not differ by more than 1 mg. Label this mass as m_4.

6.4.4 Procedure A3

6.4.4.1 Weigh a test specimen with the analytical balance (6.2.1.2) to the nearest 0,1 mg (m).

6.4.4.2 Using a measuring cylinder (6.2.4.3), measure 30 ml of concentrated sulfuric acid (6.2.4.6) into a 200 ml round bottom flask (6.2.4.2).

6.4.4.3 Attach a reflux condenser (6.2.4.4) to the flask.

6.4.4.4 Place the specimen in the concentrated sulfuric acid (6.2.4.8) and heat the flask in a heating mantle (6.2.4.5) (at 180 °C ± 10 °C) so that the sulfuric acid begins to fume. When all the resin has dissolved and the sulfuric acid solution has become dark brown in colour, stop heating.

6.4.4.5 Fill a syringe with about 20 ml of hydrogen peroxide solution (6.2.4.9). Slowly add small drops of hydrogen peroxide solution to the flask. When all the fibres have floated up to the surface, leaving the sulfuric acid solution clear, add 2 ml more hydrogen peroxide solution and heat for 10 min prior to cooling to room temperature.

6.4.4.6 Dry a glass filter (6.2.4.1) to constant mass (m_3), following the procedure described in 6.4.3.5.

6.4.4.7 Filter the contents of the flask through the glass filter, using vacuum suction, to separate off the fibre.

6.4.4.8 Wash out the flask with 20 ml to 30 ml of concentrated sulfuric acid, filtering the washings through the glass filter.

6.4.4.9 Wash the fibre retained on the filter with approximately 500 ml of distilled water from a washing bottle, ensuring that the washing water is close to pH 7. Then wash the fibre with acetone.

6.4.4.10 Place the glass filter in an air-circulation oven (6.2.4.6) and dry it at 105 °C ± 5 °C for 90 min.

6.4.4.11 After drying, remove the glass filter from the oven and place it in a desiccator for 30 min to cool to room temperature.

6.4.4.12 Weigh the glass filter plus fibre to the nearest 0,1 mg.

6.4.4.13 Continue drying, cooling and weighing until constant mass is reached, i.e. until two consecutive weighings do not differ by more than 1 mg. Label this mass as m_4.

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6.5 Expression of results

6.5.1 In the case of procedure A1 (see 6.4.2), calculate the fibre content by mass from Formula (1):

$$w_f = \frac{m - m_1 + m_2}{m} \times 100 \qquad (1)$$

where

w_f is the fibre content (mass %);

m is the initial mass of the test specimen (g);

m_1 is the initial mass of the test specimen and nichrome wire (g);

m_2 is the mass of the test specimen and nichrome wire after combustion (g).

In the case of procedure A2 (see 6.4.3) and procedure A3 (see 6.4.4), calculate the fibre content from Formula (2):

$$w_f = \frac{m_4 - m_3}{m} \times 100 \qquad (2)$$

where

m_3 is the mass of the glass filter (g);

m_4 is the mass of the test specimen and glass filter after digestion (g).

6.5.2 Calculate the fibre content by volume, the resin content by volume and the void content by volume from Formulae (3), (4) and (5):

$$\varphi_f = \frac{w_f \times \rho_c}{\rho_f} \qquad (3)$$

$$\varphi_r = \frac{(100 - w_f) \times \rho_c}{\rho_r} \qquad (4)$$

$$\varphi_v = 100 - (\varphi_f + \varphi_r) \qquad (5)$$

where

φ_f is the fibre content (volume %);

φ_r is the resin content (volume %);

φ_v is the void content (volume %);

w_f is the fibre content (mass %);

ρ_c is the density of the test specimen (g/cm^3);

ρ_f is the density of the fibre (g/cm^3) (see Note);

ρ_r is the density of the resin (g/cm^3).

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If the calculated value of the void content is negative, it is necessary to re-examine the densities of the resin, the fibre and the composite. In such cases, a solution shall be negotiated between the purchaser and supplier.

NOTE The density of the fibre can be obtained from the supplier's documentation.

7 Method B: Thickness measurement method

7.1 Principle

The thickness of the composite is measured over the entire surface of the composite. Using known values of the mass per unit area and the density of the reinforcement, the fibre content of the composite can be calculated.

7.2 Apparatus and materials

7.2.1 **Ball micrometre** or **equivalent instrument**, reading to an accuracy of 0,01 mm, to measure the specimen thickness.

7.3 Test specimen

The mass of the test specimens shall be at least 0,5 g and preferred of cuboid shape. The recommended dimensions are a thickness of less than 4 mm with 6 mm to 10 mm in length and width. Test at least 5 specimens every series for assessment of void content and at least 3 specimens every series for assessment of fibre volume content.

7.4 Procedure

7.4.1 Determine the density of the carbon fibre (ρ_f) in accordance with ISO 10119.

7.4.2 Measure the thickness with a ball micrometre or equivalent instrument (7.2.1) at at least 10 points scattered uniformly across the surface of the composite.

7.4.3 It should be noted that the surface smoothness of the composite will affect the results of this measurement. The fibre content of a composite can be determined for the whole composite or for specific areas of the composite. Hence, a correction might be needed for a non-uniform resin surface, such as that caused by the use of a peel ply during manufacture.

7.5 Expression of results

Calculate the fibre content by volume and the resin content by volume of the composite from Formulae (6) and (7):

$$\varphi_f = \frac{N \times \rho_{A,p}}{d \times \rho_f} \times 10^{-1} \qquad (6)$$

$$\varphi_r = 100 - \varphi_f \text{ (see Note 2)} \qquad (7)$$

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where

N is the number of plies in the composite;

$\rho_{A,p}$ is the mass per unit area of the fibre in the plies of the prepreg (g/cm^2) (see Note 1);

d is the thickness of the composite (mm);

ρ_f is the density of the fibre (g/cm^3) (see Note 3).

NOTE 1 The mass per unit area of the fibre in prepregs can be obtained either from the supplier's documentation or by direct measurement using specimens taken from an area adjacent to the area of the prepreg used in the manufacture of the composite.

NOTE 2 The calculation of the resin content assumes a 0 % void content.

NOTE 3 The density of the fibre can be obtained from the supplier's documentation.

8 Method C: Microscopic method

8.1 Principle

8.1.1 Procedure C1: Determination of fibre volume content

The area of fibre in the observation area of a carbon-fibre-reinforced plastic specimen is measured by image-analyser or optical microscope, and the ratio of fibre area to the observation area is defined as fibre volume content of the specimen.

8.1.2 Procedure C2: Determination of areal void content

The total void area on the whole observed cross-section of a carbon-fibre-reinforced plastic specimen is measured by optical microscope, image analyser and the ratio of the measured void area to the area of cross-section is defined as areal void content of the specimen.

Because of difference between the term of areal void content and void content, and of the result deviation caused by the choosing of observation equipment and calculating means, its use shall be as agreed between the purchaser and supplier.

NOTE The identifiable void size depends on the accuracy of the observation equipment (such as the maximum magnification of the equipment and its resolution), and the computational accuracy is decided by the means of calculating the void area. In general, the accuracy of area calculation using a semi-automatic or fully automatic image analysis software is higher than that of area counting using transparent grid papers.

8.2 Apparatus and materials

8.2.1 General

8.2.1.1 Castable resins, such as clear epoxy resins.

8.2.1.2 Water proof abrasive papers, choosing No.320, No. 400, No. 600, No. 800.

8.2.1.3 Polishing cloth, choosing velvet and woollen.

8.2.1.4 Polishing paste, choosing W0,5 or W1 artificial diamond polishing paste.

8.2.1.5 Grinding and polishing equipment.

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8.2.1.6 Micrometre or equivalent instrument, reading to an accuracy of 0,01 mm.

8.2.2 Procedure C1

8.2.2.1 A metallographic microscope with magnification factor over 1 200.

8.2.2.2 Millimetre transparent graph papers, for counting.

8.2.2.3 Image analyser, with quantitative measurement analysis software (particle area, area percentage) and data processing system.

8.2.2.4 A planimeter.

8.2.3 Procedure C2

8.2.3.1 Microscope, with eyepiece mesh as specified in ISO 9344, micrometre and other accessories.

8.2.3.2 Image analyser.

8.3 Test specimen

8.3.1 General

For unidirectional laminate composites, the specimens shall be cut off along the cross-section perpendicular to the axial direction of the fibre, whose length is 20 mm, width 10 mm and the depth as same as the sample. Test at least 5 specimens every series for assessment of void content and at least 3 specimens every series for assessment of fibre volume content. For orthogonal and multidirectional laminate composites, at least three specimens with length of 20 mm, width of 10 mm and height as same as the sample shall be cut off along each cross-section perpendicular to every axial direction of the fibre.

8.3.2 Specimen preparation

8.3.2.1 Measurement of specimens

Measure the length and width of the cross section of the specimen before determination of the void content with a micrometre or equivalent instrument (8.2.1.6), accurate to 0,01 mm.

8.3.2.2 Specimen embedding

Embed the specimens with castable resins (8.2.1.1), making sure that the observation surface is perpendicular to the related orientation of fibre.

8.3.2.3 Specimen grinding and polishing

Grind the well embedded specimens against the grinding machine (8.2.1.5) with rough to fine abrasive papers (8.2.1.2) in the flowing water. Then polish the specimen with appropriate polishing cloth (8.2.1.3) and polishing paste (8.2.1.4) on the polishing machine, until the cross-section morphology is clearly visible under the microscope. The specimen shall be thoroughly rinsed out by every replacement of the abrasive paper during the process of grinding and polishing. If there is polishing paste plugging the pores, an ultrasonic cleaner can be used.

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8.4 Procedure

8.4.1 Procedure C1

8.4.1.1 Procedure C1.1: Determination of fibre volume content using image analyser

8.4.1.1.1 Place the well-prepared specimen on the platform of the image analyser (8.2.2.3).

8.4.1.1.2 Adjust the luminance of the observation plane and the focusing plane to obtain the clear fibre cross section morphology. There shall be no voids in the observation plane.

8.4.1.1.3 Adjust the magnification of the image analyser to greater than 500 ×, so that the single fibre is able to be distinguished.

8.4.1.1.4 Calculate the percentage of the fibre area to the observed area. Record the result. Measure at least three observation fields for each specimen.

8.4.1.2 Procedure C1.2: Determination of fibre volume content using microscope

8.4.1.2.1 Place the well-prepared specimen on the platform of the microscope (8.2.2.1).

8.4.1.2.2 Photomicrograph three observation fields on one specimen all at the magnification of 200 ×, for determination of the area of each observation field (A_i) and the number of fibres (N_i) in every field. There shall be no voids in the observation field.

8.4.1.2.3 Take a micrograph at the magnification of 1 200 × (or greater) for determination of the a single fibre cross-sectional area (A_f).

8.4.1.2.4 Cover the micrograph with a calibrated millimetre transparent graph paper (8.2.2.2). Determinate the average cross-sectional area ($\overline{A_f}$) of 25 fibres on the micrographs obtained by the means in 8.4.2.2.3, using a planimeter (8.2.2.4) or other methods. The fibre's cross-sectional area can be calculated from the measured fibre diameter, if the fibre has a circular cross section.

8.4.2 Procedure C2:

8.4.2.1 Procedure C2.1: Determination of areal void content using image analyser

8.4.2.1.1 The pore content can be measured on the sample or photomicrograph with an image analyser (8.2.3.2).

8.4.2.1.2 Place the well-prepared specimen on the platform of the image analyser.

8.4.2.1.3 Adjust the magnification of the image analyser until the cross-section of the specimen can be clearly observed. Photomicrograph the specimen first or measure the absolute void area directly over the entire cross-section, or direct determination of the absolute pore area over the entire pattern, or calculate the percentage of the pore area to the cross-section area of the specimen. Record the results.

8.4.2.2 Procedure C2.2: Determination of areal void content using microscope with eyepiece mesh

8.4.2.2.1 Place the well-prepared specimen on the platform of a microscope (8.2.3.1).

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8.4.2.2.2 Observe the entire section of the specimen rapidly at magnification of 100 × and adjust the magnification to make most of the pore areas greater than a quarter cell.

8.4.2.2.3 Record the number of cells falling on the pore areas, with a quarter of a cell as the minimum counting unit. Count for 1/2 when it is greater than 1/4 cell; for 3/4 of greater than 1/2 cell; for 1 of greater than 3/4 cell. The area of each eyepiece grid cell shall be calibrated with a microscale at the selected magnification.

8.4.2.2.4 It should be noted that the number of voids, the maximum and the minimum of the void area, the total area of voids and the void content in the measured area of each specimen shall be determined by using no matter which means mentioned in procedure C2.

8.5 Expression of results

8.5.1 In the case of procedure C1.1, calculate the fibre content by volume from Formula (8):

$$\varphi_f = \frac{\sum_{i=1}^{n} \varphi_{fi}}{n} \tag{8}$$

where

φ_f is the fibre content (volume %);

φ_{fi} is the fibre content in the i-th observation field (volume %);

n is the number of observation fields of the specimen.

8.5.2 In the case of procedure C1.2 (using microscope), calculate the fibre content by volume in each observation field from Formula (9), and then calculate the fibre content by volume from Formula (8) in 8.5.1:

$$\varphi_{fi} = \frac{N_i \cdot \overline{A_f}}{A_i} \times 100 \tag{9}$$

where

φ_{fi} is the fibre content in each observation field (volume %);

$\overline{A_f}$ is the average cross-sectional area of the fibre (μm^2);

A_i is the area of the i-th observation field (μm^2);

N_i is the number of fibres in the i-th observation field.

8.5.3 In the case of procedure C2.1, calculate the areal void content from Formula (10):

$$\varphi_v = \frac{\sum_{i=1}^{n} A_{vi}}{A} \times 100 \tag{10}$$

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