SLOVENSKI STANDARD
SIST EN 2564:2001

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Aerospace series - Carbon fibre laminates - Determination of the fibre, resin and void contents

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Luft- und Raumfahrt - Kohlenstoffaser-Laminate - Bestimmung der Faser-, Harz- und Porenanteile

Série aérospatiale - Stratifiés de fibres de carbone - Détermination de la teneur en fibres, en résine et du taux de porosité

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49.025.40 Guma in polimerni materiali Rubber and plastics

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English version

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This European Standard was approved by CEN on 15 May 1998.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

This European Standard has been prepared by the European Association of Aerospace Manufacturers (AECMA).

After inquiries and votes carried out in accordance with the rules of this Association, this Standard has received the approval of the National Associations and the Official Services of the member countries of AECMA, prior to its presentation to CEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 1999, and conflicting national standards shall be withdrawn at the latest by January 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This standard specifies the methods for determining the fibre content by volume and mass and, by correlation, the resin content by volume and mass and void content by volume, of cured carbon fibre laminates, for aerospace applications.

2 Normative references

This European Standard incorporates by dated or undated reference provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 10119 Carbon fibre - Determination of density

ISO 1183 Plastics - Methods for determining the density and relative density of non-cellular plastics

EN 2743 Aerospace series - Reinforced plastics - Standard procedures for conditioning prior to testing [1)]

3 Principle

Determination of the difference in mass of specimens before and after extraction of the resin by sulphuric acid digestion.

Two methods are applicable:

- Method A is the reference method,
- Method B is a simplified method.

4 Apparatus and reagents

4.1 Balance accurate to ± 0,1 mg

4.2 Tweezers

4.3 Cutting device, such as a diamond blade saw or any other suitable apparatus

1) Published as AECMA Prestandard at the date of publication of this standard

4.4 250 ml double necked pear-shaped flask equipped with 50 ml dropping funnel, air inlet and a water pump

4.5 Beakers of various capacities, including 400 ml

4.6 20 ml sintered glass crucible (n°2 porosity) and suitable filtration assembly

4.7 Desiccator containing a suitable drying agent (for example silica gel, calcium chloride or phosphorus pentoxide)

4.8 Concentrated sulphuric acid (specific gravity: 1,84 to 1,89)

4.9 Hydrogen peroxide solution (300 g/l to 500 g/l)

4.10 Acetone (propanone)

4.11 Distilled water

4.12 Heat source with suitable temperature control

4.13 Fume cupboard (for method B, see 8.2)

4.14 Thermometer capable of measuring 180 °C

4.15 Sand bath (for method B, see 8.2)

4.16 Electric oven capable of maintaining a temperature of (120 ± 5) °C

4.17 Protective clothing, including rubber gloves, for handling hydrogen peroxide and sulphuric acid solutions

4.18 Safety screen

5 Specimens

5.1 Shape and dimensions

The specimens shall be rectangular 20 mm × 10 mm, thickness 2 mm.

Specimens of different dimensions may be used, subject to agreement between the user and manufacturer.

5.2 Number and distribution

A minimum of three specimens shall be used.

These shall be evenly distributed in the laminate and cut at least 10 mm from its edges.

6 Health, safety and environmental aspects

This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

7 Conditioning and test atmospheres

7.1 Conditioning

The specimens shall be conditioned in the test atmosphere (see 7.2) for a minimum of 2 h, unless otherwise specified.

7.2 Atmosphere for testing

EN 2743 B

7.3 Time interval between conditioning and testing

After conditioning, the sample shall be maintained in the test atmosphere. Unless otherwise specified, tests shall be carried out within 6 h.

8 Procedure

Use protective clothing and rubber gloves for handling hydrogen peroxide and sulphuric acid solutions. The reactions shall be carried out behind the safety screen for eye protection.

8.1 Method A

8.1.1 Dry the specimens to constant mass in the desiccator, that is until the difference in mass of two successive weightings is less than 2 mg. Record m_1.

8.1.2 Determine the density (ρ_c) of each specimen in accordance with ISO 1183, method A.

8.1.3 Drop the specimen into the double necked, pear-shaped flask and carefully pour 20 ml of concentrated sulphuric acid into the flask. Fit the dropping funnel to one neck and the water pump to the other.

8.1.4 Pour a suitable amount of the hydrogen peroxide solution (e.g. 20 ml of 500 g/l concentration or 40 ml of 300 g/l concentration) into the dropping funnel and heat the flask gently to (160 ± 10) °C until the acid starts to fume. Discontinue heating of the flask and allow the hydrogen peroxide solution to drip into the acid at a rate of about one drop every 2 s, increasing to one drop per second after 5 min. If, after all the hydrogen peroxide solution has been used, the solution is still brown, add further 5 ml portions of hydrogen peroxide at the latter rate and continue heating until the solution becomes and remains clear.

8.1.5 Stop heating. When the neck of the flask is cool enough to handle, ensure the air inlet is open, turn the water pump off, remove the attachments from the flask and cool the flask and contents down to ambient temperature in running water.

8.1.6 Pour the contents of the flask into a 400 ml beaker containing 100 ml of distilled water, wash with distilled water any fibres adhering to the flask into this beaker.

8.1.7 Filter the contents of the beaker through the sintered glass crucible, previously dried and weighed (m_2), washing the beaker and contents of the crucible with distilled water until washings are free from acid, then wash with 10 ml of acetone.

8.1.8 Dry the crucible and contents at 120 °C for at least 45 min, cool in a desiccator for 20 min and weigh (m_3) to ± 1 mg.

8.2 Method B

This simplified method may be used in laboratories where a large number of tests are carried out provided that adequate safety precautions are in place.

The procedure is carried out in a fume cupboard, using open beakers instead of the pear-shaped flask, the beaker being heated on a sand bath.

In this case, only 300 g/l hydrogen peroxide solution shall be used.

8.2.1 Dry the specimens to constant mass in the desiccator, that is until the difference in mass of two successive weightings is less than 2 mg. Record m_1.

8.2.2 Determine the density (ρ_c) of each specimen in accordance with ISO 1183, method A.

8.2.3 Place each specimen in a beaker with 20 ml of concentrated sulphuric acid.

8.2.4 Place each beaker on the sand bath at (160 ± 10) °C.

8.2.5 When the resin begins to break down, which is shown by a black coloration of the sulphuric acid, add 25 ml of 300 g/l hydrogen peroxide solution very slowly.

The solution becomes clear and the fibres rise to the surface. Continue heating until the evolution of gas ceases and white fumes of sulphur trioxide re-appear. If the solution becomes dark again, repeat the operation.

8.2.6 When the solution remains clear, remove the beakers from the sand bath and allow them to cool down to ambient temperature .

8.2.7 Pour the contents of each beaker into a 400 ml beaker containing 100 ml of distilled water, wash with distilled water any fibres adhering to the first beaker into the second.

8.2.8 Filter the contents of each beaker through a sintered glass crucible, previously dried and weighed (m_2), washing the beaker and contents of the crucible with distilled water until the washings are free from acid, then wash with 10 ml of acetone.

8.2.9 Dry the crucible and contents at 120 °C for at least 45 min, cool in a desiccator for 20 min and weigh (m_3) to ± 1 mg.

9 Expression of results

9.1 Fibre content by mass (W_f)

$$W_f = 100 \times \frac{(m_3 - m_2)}{m_1}$$

where:

W_f is the fibre content as a percentage of the initial mass;

m_1 is the initial mass of the specimen, in grammes

m_2 is the mass of the sintered glass crucible, in grammes;

m_3 is the final total mass of the sintered glass crucible and the residue after acid digestion, in grammes.

9.2 Fibre content by volume (V_f)

$$V_f = W_f \cdot \frac{\rho_c}{\rho_f}$$

where:

V_f is the fibre content as a percentage of the initial volume;

W_f is the fibre content as a percentage of the initial mass;

ρ_c is the specimen density, in grammes per cubic centimetre;

ρ_f is the fibre density, in grammes per cubic centimeter, determined to ISO 10119 or given in the supplier's release report.

9.3 Resin content by mass (W_r)

$$W_r = 100 - W_f$$

where:

W_r is the resin content as a percentage of the initial mass;

W_f is the fibre content as a percentage of the initial mass.

9.4 Resin content by volume (V_r)

$$V_r = (100 - W_f) \cdot \frac{\rho_c}{\rho_r}$$

where:

V_r is the resin content as a percentage of the initial volume;

W_f is the fibre content as a percentage of the initial mass;

ρ_c is the specimen density expressed, in grammes per cubic centimetre;

ρ_r is the density of the cured resin, in grammes per cubic centimetre, determined to ISO 1183 (method A) on a cured resin specimen or given in the supplier's release report.