FINAL DRAFT International Standard

ISO/FDIS 14127

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

ISO/TC **61**/SC **13**

Secretariat: **JISC**

Voting begins on:
2024-04-23

Voting terminates on:
2024-06-18

Reference number
ISO/FDIS 14127:2024(en)

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Published in Switzerland

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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 document 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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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.

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.

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*

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.

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.

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.