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Fine ceramics (advanced ceramics, advanced technical ceramics) — Physical properties of ceramic composites — Guidelines for determination of void and fibre contents in polished cross section by image analysis

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Contents

Page

Foreword

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This document was prepared by Technical Committee ISO/TC *206*, *Fine ceramics*,

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Physical properties of ceramic composites — Guidelines for determination of void and fibre contents in polished cross section by image analysis

1 Scope

This document describes the method for the determination of void and fibre with specific orientation contents in a polished cross section of continuous fibre-reinforced ceramic matrix composites by image analysis.

The method applies to all ceramic matrix composites with continuous fibre reinforcement: bidirectional (2D) and tridirectional (3D).

The methods also apply to carbon-fibre-reinforced carbon matrix composites (also known as: carbon/carbon or C/C).

NOTE The result obtained by the method is not volume content but area content.

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 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology 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
amount of fibre present in a cross section of composite

4 Principle

The test specimens are cut out of representative locations of samples or materials. The cutting sections of test specimens are carefully polished and subjected to observation with an optical microscope or similar. Digital images or photographs are taken at high magnification and analysed.

Voids are detected and discriminated based on the grayscale level differences compared to those of matrix and fibres. The threshold value is determined from the histogram of the image. The image is binarized and the void pixels are counted. The void area content is calculated as the ratio of the void pixels to the total pixels.

There are two different determination methods for the fibre area content. One is a simplified method and the other is a detailed method.

In the simplified method, the mean filament area is calculated from several tows chosen from the polished section at random. The number of tows is counted in the image or photograph. The fibre content in the polished section is determined as the product of the mean filament area per tow and the number of tows.

In the detailed method, the area of all filament sections in the image is detected by a pattern matching method with image analysis software, because the shape of the filament cross section is considered to be almost circular. By counting the number of pixels detected as filaments, the fibre content is determined as the pixel ratio of the filament area to the total image size.

5 Significance and use

The results obtained by the methods are not volume content but area content. In the case of fibre content, the result is the area content of the fibre of specific orientation. If a random cross section is representative of the volumetric fibre distribution, the volume content can be determined by measuring adequate number of sections and calculating the mean value. Even the area content is useful to investigate the material properties and control the material quality for the purpose of material development.

Since the image quality affect the analysis result significantly, the preparation of test specimen is crucial for the image analysis. The polishing method, however, includes the know-how or the practical skills of laboratories and also depends on the characteristic of material to be analysed. For these reasons, this document does not specify the details of polishing method, but describes general principle.

NOTE With respect to fine ceramics, some guidelines on grinding and polishing are given in ISO 13383-1:2012, Annex A.

The detailed method to determine the fibre area content is performed by a pattern matching method to detect the filament area in the image. This algorithm is based on the characteristic that its cross section of filament is circle. Therefore, all filaments shall be cut perpendicular to their axes within ±15° so that the cross sections remain circle. In some woven materials it is difficult to cut the test specimen perpendicular to the fibre axis due to fibre waving. This method is not suitable for such woven materials.

6 Apparatus

6.1 Microscope should have high resolution to enable good object observation (i.e. a resolution of around one tenth of filament diameter or void size). A scanning electron microscope (SEM) may be used. A microscope with an automatic stage and automatic focus to stitch a number of digital images is recommended to cover lager cross sections for observation.

6.2 Calibrated rule or scale should be accurate within ±0,5 %. Its reading should be 0,5 mm or better.

6.3 Image analysis software Image analysis software is capable of the following.

— making a grayscale histogram, setting a threshold value automatically or manually and binarizing an image based on the threshold value;

— detecting a circular shape and counting the number of detected pixels.

