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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

Céramiques techniques (céramiques avancées, céramiques techniques avancées) — Propriétés physiques des composites céramiques — Lignes directrices pour la détermination du taux de porosité et de la teneur en fibre sur une section polie par analyse d'images

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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 methods 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 methods apply 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

There are no normative references in this document.

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 the 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 affects 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 depends on the characteristic of material to be analysed. For these reasons, this document does not specify the details of polishing method, but describes the 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 the 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 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.

7 Test specimen

7.1 Sampling

The test specimen should be cut perpendicular to the fibre orientation. If the material is reinforced in multiple orientations, the test specimen should be cut directional perpendicular to the fibre orientation of interest. The recommendations for the cross section are as follows:

a) for 2D textile fabrics, the cross section should contain more than three plies;

b) for 3D textile fabrics, the cross section should contain more than three unit cells.

In cutting out the test specimen, care shall be taken to prevent the damage such as fibre cracking, fibre loss and so on.

7.2 Mounting

If the test specimen is hard to handle in grinding and polishing, mount the test specimen using appropriate mounting equipment. It is advisable to vacuum impregnate the test specimen with liquid mounting resin before encapsulating as this will provide some support during polishing.

NOTE It is not essential to encapsulate the test specimen. For example, it could be affixed to a metal holder. However, encapsulation in a polymer-based medium allows easy gripping and handling, especially of small irregularly shaped test specimens and of weak, friable material.

7.3 Grinding and polishing

Grind and polish the surface of the test specimen. Care shall be taken to ensure that grinding produces a planar surface with a minimum of damage. Employ successively smaller grit sizes, at each stage removing the damage from the previous stage until there is no change in appearance when examined by an optical microscope at high magnification. Care should be taken in choosing the sequence of grits and lap types. The final surface shall be free from optically visible scratches, or other damage introduced by polishing, which would interfere with the determination.

NOTE It is impossible within the scope of this document to make specific recommendations for all types of material. The general principle to be adopted is the minimization of subsurface damage, and its removal by progressively fined grits while retaining a flat surface. Some guidelines on grinding and polishing are given in in ISO 13383-1:2012, Annex A.

8 Procedures

8.1 Calibration

Before image acquisition, measure a calibrated rule or scale using a stage micrometer and calculate the length per pixel.

8.2 Image acquisition

Place the test specimen on the microscope stage so that the test specimen surface is perpendicular to the optic axis. Obtain a grayscale image so as to contain the unit structures described in Clause 7. The resolution should be around one tenth the size of the object for detection. If multiple images are stitched to one image, it is recommended to use the same conditions for all images. Enhance the contrast of image in order to discriminate the void area and the filament area accurately. The edge of the sample causes poor contrast. Obtain a grayscale image so as to exclude the sample edge.

NOTE 1 In the digital image of the polished section taken using an optical microspore or SEM, the void area is darker than other areas.

NOTE 2 In general, the grayscale level range of filaments is similar to that of matrix.

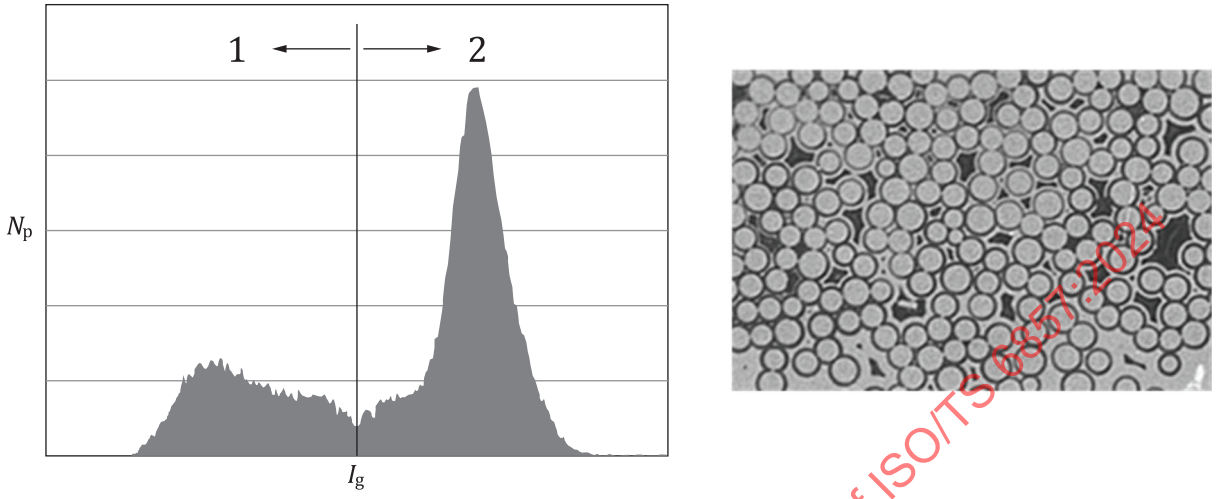
8.3 Void area content

8.3.1 Binarization

Develop the grayscale histogram with image analysis software. Since the grayscale level in a void area is lower (darker) and that in other areas is higher (lighter), in general, the histogram shows a distribution with two peaks (bimodality distribution) as shown in Figure 1. Set the threshold value to that indicated in the valley of the figure and binarize the image.

Lower void area content decreases the number of low grayscale pixels relatively and lowers the peak of the void pixels in the histogram. If setting a threshold value for binarization is difficult, adjust the contrast and attempt taking the image again.

The image analysis to the stitched file can be performed, although the size of image file becomes large. It is recommended to not have a compression of the image resulting from file stitching.



Key

1 void
2 fibre and matrix
I_g gray scale intensity
N_p number of pixels

Figure 1 — Grayscale histogram

8.3.2 Pixel counting

Count the number of black and white pixels, P_b, P_w, for the binary image using image analysis software.

8.3.3 Removal of smaller voids

Label all the detected voids and obtain their areas with image analysis software. Exclude the smaller voids agreed between the Parties.

8.4 Fibre area content by simplified method

8.4.1 Tow counting

Count the number of tows in the image. If a tow intersects the frame border, count the number of tows to the first decimal place considering the mean tow area as shown in Figure 2.

8.4.2 Determination of mean filament area per tow

Measure the filament area in a tow with a planimeter, image analysis software or other methods and obtain the filament area in a tow. Continue this measurement for 30 % or more tows in the image and determine the mean area per tow. If it is difficult to measure the mean tow area directly, count the number of filaments and obtain the mean filament diameter. The mean filament diameter can be obtained from either direct

measurement for an adequate number of filaments or the inspection certificate by a filament manufacturer. The filament area in a tow is determined by Formula (1):

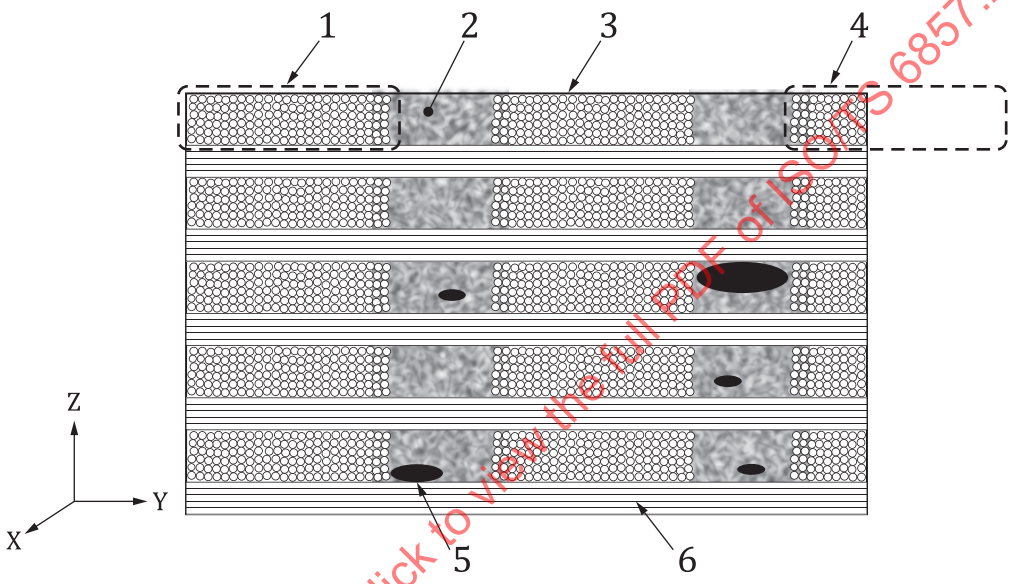
$$A_f = \frac{\pi m \bar{d}^2}{4} \quad (1)$$

where

A_f is the filament area in a tow;

m is the number of filaments in a tow;

$\bar{d}$ is the mean filament diameter.



Key

1	tow	4	0,3 tow
2	matrix	5	void
3	X direction filaments	6	Y direction filaments

Figure 2 — Illustration of polished cross-section

8.5 Fibre area content by detailed method

8.5.1 Filament detection

Detect each filament section with the circular shape recognition function of image analysis software. If pattern recognition of the image is difficult, the edges of filament as shown in Figure 3 are detected first and pattern recognition can be performed on the detected edges.

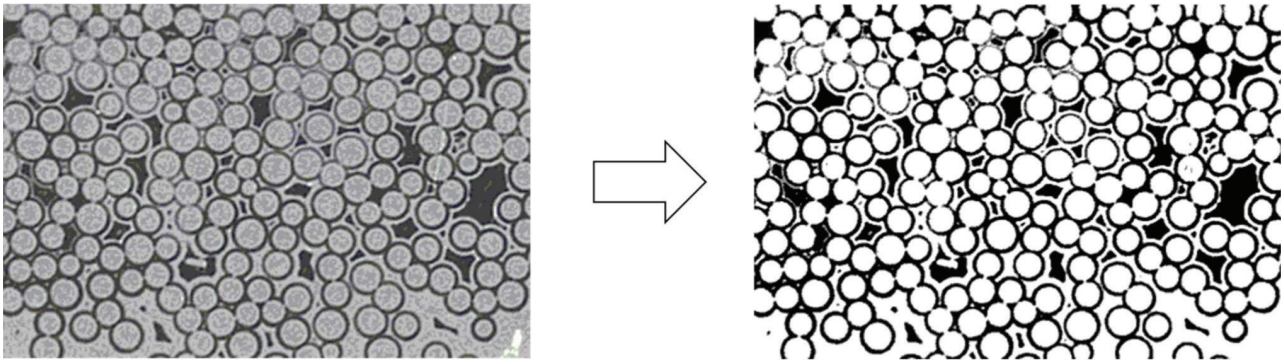


Figure 3 — Result of edge detection

8.5.2 Pixel counting

Count the number of pixels detected as the filament area P_f.

9 Calculation

9.1 Void area content

Calculate the void area content to the first decimal place by using Formula (2):

$$A_v = \frac{P_b - P_{cut}}{P_b + P_w} \times 100 \quad (2)$$

where

A_v is the void content in each image (%);

P_{cut} is the total number of void pixels agreed between the parties as the cut-off void;

P_b is the total number of pixels with a brightness of 0 (black) in a binary image;

P_w is the total number of pixels with a brightness of 255 (white) in a binary image.

9.2 Fibre area content by simplified method

Calculate the fibre content to the first decimal places by using Formula (3):

$$A_f = \frac{n\overline{A_f}}{A_t} \times 100 \quad (3)$$

where

A_f is the fibre content in a polished cross-section;

n is the number of tows;

$\overline{A_f}$ is the mean fibre area in a tow;

A_t is the total image area.

9.3 Fibre area content by detailed method

Calculate the fibre content to the first decimal places by using Formula (4):

$$A_f = \frac{P_f}{P_t} \times 100 \quad (4)$$

where

A_f is the fibre content in a polished cross-section;

P_f is the number of pixels detected in a filament;

P_t is the total number of pixels of image analysed.

10 Report

The test report shall contain, at least, the following information:

a) reference to this document, i.e. ISO/TS 6857:2024;

b) test laboratory and operator;

c) date of measurement;

d) description of the test material (material type, manufacturing data, etc.);

e) preparation of test specimen (cutting method, polishing method, etc.);

f) measurement devices;

g) images before and after analysis;

h) image analysis software (including version);

i) individual values of the void area content and/or fibre area content;

j) mean values of the void area content and/or fibre area content;

k) cases different from the procedures or requirements of this document;

l) any unusual features observed.