

Advanced technical ceramics — Monolithic ceramics — General and textural properties

Part 5: Determination of phase volume fraction by evaluation of micrographs

ICS 81.060.30

National foreword

This British Standard is the UK implementation of EN 623-5:2009. It supersedes DD ENV 623-5:2002 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics.

A list of organizations represented on this committee can be obtained on request to its secretary.

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fraction by evaluation of micrographs**

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monolithiques - Propriétés générales et textures - Partie 5:
Détermination de la fraction volumique de phase par
évaluation des microphotographies

Hochleistungskeramik - Monolithische Keramik -
Allgemeine und strukturelle Eigenschaften - Teil 5:
Bestimmung des Volumenanteils von Phasen durch
Auswertung von Mikrogefügeaufnahmen

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Foreword

This document (EN 623-5:2009) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by January 2010, and conflicting national standards shall be withdrawn at the latest by January 2010.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes ENV 623-5:2002.

EN 623 consists of five parts, under the general title “Advanced technical ceramics - Monolithic ceramics - General and textural properties”:

- Part 1: *Determination of the presence of defects by dye penetration*
- Part 2: *Determination of density and porosity*
- Part 3: *Determination of grain size and size distribution (characterized by the Linear Intercept Method)*
- Part 4: *Determination of surface roughness*
- Part 5: *Determination of phase volume fraction by evaluation of micrographs*

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1 Scope

This part of EN 623 specifies a manual method of making measurements for the determination of volume fraction of major phases in advanced technical ceramics using micrographs of polished and etched sections, overlaying a square grid of lines, and counting the number of intersections lying over each phase.

NOTE 1 This method assumes that the true phase volume fractions are equivalent to area fractions on a randomly cut cross-section according to stereological principles.

NOTE 2 Guidelines for polishing and etching of advanced technical ceramics can be found in Annexes A and B.

The method applies to ceramics with one or more distinct secondary phases, such as found in $\text{Al}_2\text{O}_3/\text{ZrO}_2$, Si/SiC, or $\text{Al}_2\text{O}_3/\text{SiC}_w$.

If the test material contains discrete pores, these can be treated as a secondary phase for the purpose of this method provided that there is no evidence of grain pluck-out during polishing being confused with genuine pores.

NOTE 3 If the material contains more than about 20 % porosity there is a strong risk that the microstructure will be damaged during the polishing process, and measurement of volume fraction of pores may become misleading.

Secondary phase volume fractions or porosity present at levels of less than 0,05 are subject to considerable error and potential scatter in results. A larger number of micrographs than the minimum of three is normally needed to improve the consistency and accuracy of the results.

NOTE 4 Many ceramics contain small amounts of secondary glassy phases. In order to make a reasonable estimate of glassy phase content, the glass material between crystalline grains should be readily observable, and thus should be at least 0,5 μm in width. The method in this European Standard is not considered appropriate for narrow glassy films around grains.

This method assumes that the selected regions of a prepared cross-section are statistically representative of the whole sampled section.

NOTE 5 Microstructures are seldom homogeneous, and the phase contents can vary from micrograph to micrograph. It is essential to survey a sufficiently wide area of the prepared section to ensure that those areas selected for evaluation are representative, and do not contain eye-catching irregularities.

Some users of this European Standard can wish to apply automatic or semiautomatic image analysis to micrographs or directly captured microstructural images. This is currently outside the scope of this European Standard, but some guidelines are given in Annex C.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 1006, *Advanced technical ceramics - Monolithic ceramics - Guidance on the selection of test pieces for the evaluation of properties*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

3 Terms and definitions

For the purposes of this part of EN 623, the following terms and definitions apply.

3.1

phase volume fraction

volume occupied by a distinct, identifiable phase present in a material expressed as a fraction of the whole

3.2

secondary phase

one or more distinct identifiable phases other than a primary crystalline phase in a material

NOTE A secondary phase can be in the form of discrete grains, or as a continuous phase surrounding some or all the major phase grains. For the purposes of this European Standard, porosity may be treated as a secondary phase.

4 Apparatus

4.1 Sectioning equipment

A suitable diamond-bladed cut-off saw to prepare the initial section for investigation. The saw shall be metal bonded with a diamond grit size of 125 µm to 150 µm and shall be cooled.

NOTE This grit size is designated D151 according to ISO 6106, see [1].

4.2 Mounting equipment

Suitable metallurgical mounting equipment and media for providing firm gripping of the test piece for polishing.

4.3 Grinding and polishing equipment

Suitable grinding and polishing equipment, employing diamond abrasive media.

NOTE A sequence of abrasives and techniques recommended for polishing are given in Annex A.

4.4 Microscope

An optical or scanning electron microscope with photomicrographic facilities.

NOTE Although the true magnification of the image is unimportant for making the measurement of volume fraction, it is advised that a reference graticule may be used to determine magnification in an optical microscope, or a reference grid or latex spheres may be used for calibration of magnification in a scanning electron microscope, and as a check on the homogeneity of magnification across the field of view.

An optical microscope is additionally required for assessing polishing (see 5.4).

4.5 Transparent grid

Transparent square grid on, e.g. acetate film, and with line thickness not exceeding 0,1 mm.

NOTE 1 The grid spacing selected is not critical, but may conveniently be between 3 mm and 15 mm to minimise eyestrain. However, it is necessary that consideration of the requirements of 6.3 is taken into account.

NOTE 2 A suitable grid may be prepared as a computer plot with sufficient accuracy of line spacing for the purposes of this European Standard.