SLOVENSKI STANDARD
kSIST prEN 623-5:2009

01-marec-2009

Sodobna tehniļna keramika - Monolitna keramika - Sploġne in strukturne lastnosti - 5. del: Doloļanje volumenskega deleġa faz z vrednotenjem mikrostrukture

Advanced technical ceramics - Monolithic ceramics - General and textural properties - Part 5: Determination of phase volume fraction by evaluation of micrographs

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

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

Ta slovenski standard je istoveten z: **prEN 623-5**

ICS:

81.060.30	Sodobna keramika	Advanced ceramics

kSIST prEN 623-5:2009 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

FINAL DRAFT
prEN 623-5

December 2008

ICS 81.060.30

Will supersede ENV 623-5:2002

English Version

Advanced technical ceramics - Monolithic ceramics - General and textural properties - Part 5: Determination of phase volume fraction by evaluation of micrographs

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

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

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If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Ref. No. prEN 623-5:2008: E

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Foreword

This document (prEN 623-5:2008) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This document is currently submitted to the Unique Acceptance Procedure.

This document will supersede 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*

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 Al_2O_3/ZrO_2, Si/SiC,or Al_2O_3/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.

prEN 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.

5 Test piece preparation

5.1 Sampling

The test pieces shall be sampled in accordance with the guidelines given in prEN 1006, and subject to agreement between parties.

NOTE Depending on the objectives of performing the measurement, it is desirable to maintain knowledge of the positions within components or test pieces from which sections are prepared.

5.2 Cutting

The required section of test-piece shall be cut using the diamond saw (see 4.1).

NOTE For routine inspection of materials, a small area of side no more than 10 mm is normally adequate as the section to be polished.

5.3 Mounting

Mount the test piece using an appropriate mounting medium. If the ceramic is suspected to have significant open porosity in some regions (see Clause 1) it is advisable to vacuum impregnate the test piece with liquid mounting resin before encapsulating as this will provide some support during grinding and polishing.

NOTE It is not essential to encapsulate the test piece. 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 pieces and of weak friable test pieces. The method of mounting selected should take into account the etching procedure to be used; see Annex B.

5.4 Grinding and polishing

Grind and polish the surface of the test piece. Care should 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 (see 4.4) at high magnification. At least 90 % of the test piece area shall be free from optically visible scratches, or other damage introduced by polishing, which will interfere with the determination. In particular, discrete secondary phases may be plucked out from the surface giving the appearance of pores. This shall be avoided.

NOTE Care should be taken in choosing the sequence of grits and lap types. It is impossible within the scope of this part of EN 623 to make specific recommendations for all types of material. The general principle to be adopted is the minimisation of subsurface damage, and its removal by progressively finer grits whilst retaining a flat surface. Some guidelines on polishing are given in Annex A.

5.5 Etching

When a good quality polished surface has been achieved, the test piece shall be etched if necessary to reveal the individual phases. Any suitable technique shall be used, subject to agreement between parties.

NOTE 1 Some general guidelines recommending etching procedures for various commonly available advanced technical ceramics are given in Annex B.

NOTE 2 For optical evaluation, it is usually necessary to etch oxide materials in such a way that the individual phases are distinguished by having different contrast levels. For evaluation by scanning electron microscopy (SEM), it may not be necessary to etch if a backscattered electron detector is used which has adequate resolution of net atomic number difference between the phases such that contrast is generated. If a secondary electron detector is used, it will usually be necessary to etch to produce topographic contrast unless the atomic number difference between the phases is large.