SLOVENSKI STANDARD

SIST ENV 623-5:2007

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Sodobna tehniļna keramika - Monolitna keramika - Sploġne in strukturne lastnosti - 5. del: Doloļanje volumskega 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 strukturelle Eigenschaften - Teil 5: Bestimmung des Volumenanteils von Phasen durch Auswertung von Mikrogefügeaufnahmen

Céramiques techniques avancées - Méthodes d'essai pour céramiques monolithiques - Propriétés générales et texturales

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

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This European Prestandard (ENV) was approved by CEN on 12 March 2002 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Contents

Foreword

This document ENV 623-5:2002 has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", the secretariat of which is held by BSI.

Annexes A to F are all informative.

This Prestandard includes a Bibliography.

EN 623 consists of five Parts:

Part 1: *Determination of the presence of the presence of defects by dye penetration tests*

Part 2: *Determination of density and porosity*

Part 3: *Determination of grain size*

Part 4: *Determination of surface roughness*

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this European Prestandard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Prestandard 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 Prestandard 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 containing eye-catching irregularities.

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

2 Normative references

This European Prestandard incorporates by dated or undated reference provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

ENV 1006 Advanced technical ceramics - Methods of testing monolithic ceramics - Guidance on the sampling and selection of test pieces

EN ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)

3 Terms and definitions

For the purposes of this European Prestandard, 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 Prestandard, 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 Prestandard.

5 Test piece preparation

5.1 Sampling

The test pieces shall be sampled in accordance with the guidelines given in ENV 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 Prestandard 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 SEM evaluation, 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.

6 Photomicrography

6.1 General aspects

If it suspected that the average grain size of each phase or the widths of continuous glassy phases between grains is less than 2 µm, it will be necessary to prepare the test piece for scanning electron microscopy. Between 2 µm and 4 µm either scanning electron microscopy or optical microscopy may be used. Otherwise, optical microscopy will normally be adequate.

It is important to achieve sufficient contrast between phases in order to identify individual grains clearly and unambiguously.

6.2 Inspection

Inspect the sampled cross-section in the microscope. If the microstructure appears homogeneous, prepare micrographs from randomly selected areas. If inhomogeneity of microstructure is suspected, select representative areas of relevance for measurement.

6.3 Number of micrographs

At least three micrographs shall be prepared at a magnification sufficient to identify clearly all the phases to be counted. In addition, at least 100 features in total of any given type shall be present to be counted in the set of micrographs.

NOTE For a nominally homogeneous material, it may be sufficient to use a small number of micrographs analysed with a small grid spacing, but for an inhomogeneous material, results representative of the average for the sampled section can be prepared reliably only by selecting a large number of micrographs of different areas, with less intensive counting from a larger grid.

6.4 Optical microscopy

Set up Köhler illumination in the microscope.

NOTE Guidance on setting Köhler illumination conditions is given in annex D.

Examine the test piece at a magnification sufficient to resolve the individual grains clearly. If the contrast obtained is insufficient, e.g. in white or translucent materials, apply a suitable thin metallic coating by evaporation or sputtering. Prepare micrographs of at least three different randomly selected areas of the test-piece surface, taking into account the apparent homogeneity of the microstructure (see 6.2). As a guideline, the average size of discrete phase area to be counted should appear typically at least 3 mm across. If the total number of individual grains of any one phase to be counted in any one set of micrographs is less than one hundred, prepare more micrographs. Micrographs should be typically of a size (100 x 75) mm, but may with advantage be enlarged later to aid evaluation.

6.5 Scanning electron microscopy

Mount the test piece on the test piece holder of the microscope. If the test piece is not electrically conducting, apply a thin evaporated or sputtered conductive coating. Insert the test-piece in the microscope, ensuring that the surface to be characterised is normal to the electron beam to within 5°.

NOTE 1 This ensures that the image does not suffer from excessive distortion or loss of focus due to the angle of viewing.

Prepare micrographs at a suitable magnification (see 6.4) from at least three different randomly selected areas of the test piece, using either secondary electron imaging or backscattered electron imaging.

NOTE 2 Although the contrast between phases can be enhanced using backscattered electron imaging, a noisier image than in secondary electron imaging may result and may render the boundaries between contrasting phases indistinct. It can be helpful to use secondary electron images for counting the phase proportions, but backscattered images to aid identification of each phase.

If the number of grains of the phase to be counted is less than 100 in total over all the micrographs, increase the number of areas photographed. Micrographs should typically be of a size (100 x 75) mm, but may with advantage be enlarged later to aid evaluation.

NOTE 3 It is possible that the photographic screen in the microscope will not have constant magnification at all points. A square grid makes a suitable reference for ascertaining the degree of distortion in the screen, since it is easy to detect distortions of the grid. For the purposes of this test method, distortions of typically up to 5 % may be acceptable provided that the phases being counted are distributed homogeneously across the entire area of the micrograph.

7 Measurement of micrographs

If desirable, enlarge the photomicrograph to a size suitable for easier observation of the features. Examine the dimensions of the smallest features to be counted. Select a suitable grid spacing and prepare a square grid (see 4.5 and 6.3, and comments in clause 9) such that the grid area covers the entire micrograph.

Tape the micrograph to a smooth surface. Overlay the grid such that the entire area of the micrograph is covered by the grid, with no grid intersections immediately over the edges of the micrograph. Count the number of grid intersections n_j of the grid that lie over each phase *j*. If the grid intersection lies exactly over the boundary between two phases, count this as one-half of an grid intersection for each phase. If porosity is to be estimated, use the same rule for when a grid intersection lies exactly on the edge of a pore. Count the total number of grid intersections over the area of the micrograph. If pores are not being counted, count the number of grid intersections lying over the crystalline or glassy phases in the material.

NOTE It can be helpful in counting to screen with pieces of paper those lines of intersections above and below the one being counted; this reduces eye strain and the risk of miscounting.

8 Calculation of results

For the case where porosity is to be counted as one of the phases, calculate the volume fraction of each phase using the equation (1):

$$V_{f\,j} = \frac{n_{i\,j}}{N} \qquad (1)$$

where

n_j is the total number of grid intersections over phase *j*;

N is the total number of grid intersections lying over the micrograph.

For the case where porosity is to be ignored, see equation (2):

$$V_{f\,j} = \frac{n_{i\,j}}{\Sigma\, n_{i\,j}} = \frac{n_{i\,j}}{(N - n_p)} \qquad (2)$$

where

n_j is the number of grid intersections lying over solid phase *j*;

n_p is the number of grid intersections lying over pores;

S is the sum of all grid intersections lying over all solid phases.

9 Interferences and uncertainties

The nature of the microstructure of the material can affect the result determined in this test. The test is effective when a sufficient number of grid intersections of each phase are counted. This can be achieved either by intensive analysis of the minimum number of three micrographs, or by less intensive analysis of a larger number of micrographs. For intensive analysis, the grid shall be small enough such that there is a good chance that a grid intersection will lie over each grain. Failure to do this means that the results are subject to increasing possible random error depending on exactly where the grid is positioned. The random error is minimised by adhering to the above guideline, but will always exist because of random positioning of the grid on the micrograph. Typically, for a homogeneous material with randomly distributed phases results from a given series of three micrographs counting at least 100 grains of each type should give phase volume fractions consistent to ± 0,02.

If the material appears inhomogeneous, either more areas should be analysed intensively to establish the extent of the inhomogeneity, or if an average results only is required, a larger grid spacing can be used for less intensive analysis provided that at least 100 grains of each phase type in total are counted. The procedure adopted should be reported.

The counting process requires visual observation of the phase lying underneath each grid intersection. Clean, well-defined phase boundaries are required. If the phase boundaries are poorly defined as a result of limited optical or SEM resolution, it is necessary to adopt a consistent criterion for assessing which side of the true boundary the grid intersection overlies. Failure to do this can lead to under or overestimation of phase volume fraction, and is particularly dangerous for small volume fractions.

The micrographs should not contain features which are ambiguous. Grain pluckout during polishing could inadvertently be treated as porosity and, *vice versa*, features seen within shallow pores might be counted as solid grains. Particular caution should be taken to avoid subsurface grains giving strong signals in backscattered electron images, or edge highlights in secondary electron images hiding individual grains.

NOTE Annex E contains information from a round robin activity associated with the development of this Prestandard which illustrates these concerns.

10 Test report

The report of the test shall be in accordance with EN ISO/IEC 17025 and shall contain the following:

a) the name of the testing laboratory;

b) a unique identification of the report;

c) the name and address of the client;

d) details of the test piece, including material type, manufacturing code, batch number, etc.;

e) the date of receipt of the test item(s) and of the test;

f) a reference to this European Prestandard, i.e. ENV 623-5;

g) the observation technique employed (optical or scanning electron microscope);

h) a summary of the procedure for sampling, cutting, grinding, polishing and etching the test piece;