SLOVENSKI STANDARD
SIST ENV 623-3:2000

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Advanced technical ceramics - Monolithic ceramics - General and textural properties - Part 3: Determination of grain size

Advanced technical ceramics - Monolithic ceramics - General and textural properties - Part 3: Determination of grain size

Hochleistungskeramik - Monolithische Keramik - Allgemeine und strukturelle Eigenschaften - Teil 3: Bestimmung der Korngröße

Céramiques techniques avancées - Méthodes d'essai pour céramiques monolithiques - Propriétés générales et texturales - Partie 3: Détermination de la taille des grains

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English version

Advanced technical ceramics - Monolithic ceramics - General and textural properties - Part 3: Determination of grain size

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CEN members are required to announce the existance 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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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Contents list

Foreword

This European pre-standard has been prepared by CEN/TC 184 "Advanced technical ceramics".

EN 623 consists of four parts:

Part 1: Determination of the presence of defects by dye penetration tests
Part 2: Determination of density and porosity
Part 3: Determination of grain size (ENV)
Part 4: Determination of surface roughness (ENV)

CEN/TC 184 approved this European pre-standard by resolution 2/92 during its fifth meeting held in Brussels, 1992-03-31.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to announce this European pre-standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

1 Scope

This Part of ENV 623 describes procedures for polishing and etching test pieces of advanced technical monolithic ceramics, followed by the preparation of micrographs of the microstructure and methods of making measurements for the determination of grain size.

The method applies to single-phase ceramics, and to ceramics with a principal crystalline phase and a glassy grain-boundary phase of less than about 10 % by volume. The measurements described do not apply to ceramics with more than about 10 % by volume of pores or of continuous glassy phase, nor to ceramics with more than one crystalline phase. Such materials require alternative methods which allow the pores or phases to be distinguished and counted separately, and this is beyond the scope of the method.

NOTE : This standard does not cover automatic image analysis methods of determining grain size and other parameters. By agreement between parties, such methods may be used as an alternative to this Standard.

2 Normative references

This European Standard 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 listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ENV1006 : Advanced technical ceramics - sampling of monolithic ceramics

3 Definitions

3.1 Grain size

The size of the distinct crystallites in a material, and for the purposes of this method of test, that of the primary or major phase.

> NOTE : In materials which contain more than one phase, it may be necessary to characterise the different phases. The phases may be continuous or as isolated grains. In this method, only the primary phase is characterised, and minor phases are ignored.

3.2 Mean linear intercept grain size

The average value of the distance between grain boundaries as shown by randomly positioned lines drawn across a micrograph or other image of the microstructure.

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 125 µm to 150 µm and shall be cooled.

NOTE : The grit size is designated D151 by the Federation of European Abrasives Manufacturers.

4.2 Mounting equipment

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

4.3 Grinding and polishing equipment

Suitable grinding and polishing equipment, employing diamond abrasive media.

NOTE : Annex A recommends techniques and abrasives.

4.4 Microscope

An optical microscope or scanning electron microscope with photomicrographic facilities. For materials with a mean linear intercept grain size of less than about 4 µm a scanning electron microscope is recommended in order to obtain sufficient magnification of the photomicrograph. A reference graticule is required for determination of magnification in an optical microscope, and a reference square grid or latex spheres are required for calibration of magnification in a scanning electron microscope. In all cases, the calibration of dimensions of the references shall be traceable to national or international standards of length measurement.

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

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 full knowledge of the positions within components or test-pieces from which sections are prepared.

5.2 Cutting

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

NOTE : For routine inspection of materials, a small area of not more than 10 mm side is normally adequate as the section to be polished. Large areas generally take longer to polish.

5.3 Mounting

Mount the test piece using an appropriate proprietary mounting medium. If the ceramic has significant open porosity (>30%) it is advisable to vacuum impregnate the test piece with liquid mounting resin before encapsulating as this will provide some support during 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 plastic-based medium allows easy gripping and handling, especially of small irregularly shaped test pieces and of porous, friable materials.

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. The final surface shall be free from optically visible scratches or other damage introduced by polishing.

> NOTE : Care should be taken in choosing the sequence of grits and lap types. It is impossible within the scope of this Standard 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 grinding and polishing are given in Annex A.

5.5 Etching

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

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

6 Photomicrography

6.1 General aspects

If it is suspected that the average grain size of the test material is less than about 4 μm, it will be necessary to prepare the test piece for scanning electron microscopy. Otherwise, optical microscopy will generally be adequate.

6.2 Optical microscopy

Set up Kohler illumination in the microscope. 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 metallic coating by evaporation or sputtering. Prepare micrographs of at least three different areas of the test piece surface. There should be no more than 20 grains along any 75 mm line drawn on the micrograph so produced. If there are more grains than this, increase the magnification and prepare fresh micrographs. Unless already undertaken, prepare a micrograph of a graticule at the same magnification to provide a calibration of magnification. Micrographs should be of at least 100 mm x 75 mm size.

6.3 Scanning electron microscopy

If the test piece is not electrically conducting, mount the test piece on the test piece holder of the microscope and apply a thin evaporated or sputtered conductive coating. Insert the test piece into the microscope, ensuring that the surface to be characterised is normal to the electron beam to within 5^0.

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

Prepare micrographs at a suitable magnification (see 6.2) from at least three different areas of the test-piece. For calibration of the lateral and vertical magnification of the micrographs prepare similar images of a graticule or grid, or of calibrated spheres at the same working height of the microscope stage.

NOTE 2 : The photographic screen in the microscope may 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. If the image distortion is uniform across the field of view, i.e. lateral and vertical magnifications appear to be constant but different, it is possible to make corrections when measuring the micrographs (see Note 2 in clause 7.). For the purposes of this standard, the actual magnification should not vary by more than 5 % over the area of the screen.

7 Measurement of micrographs

Draw at least five thin straight lines of length exceeding 75 mm and of random position and orientation across each micrograph, giving a total line length of not less than 375 mm intersecting at least 100 grains. Measure each line length to the nearest millimetre and calculate the total line length l(t). Count the number n(i) of intersections of the lines with grain boundaries. If the line intersects the junction of three grains, count this as 1,5 intersections. If the line intersects a large pore, count this as one intersection. Measure the total length of line that crosses large pores l(p). If the line runs along a grain boundary, count this as one intersection.