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Advanced technical ceramics - Monolithic ceramics - General and textural properties - Part 3: Determination of grain size and size distribution (characterized by the Linear Intercept Method)

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Hochleistungskeramik - Monolithische Keramik - Allgemeine und strukturelle Eigenschaften - Teil 3: Bestimmung der Korngröße und der Korngrößenverteilung (Linienschnittverfahren)

Céramiques techniques avancées - Céramiques monolithiques - Propriétés générales et texturales - Partie 3: Détermination de la taille des grains et de la distribution granulométrique (selon la méthode de l'intersection linéaire)

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Advanced technical ceramics - Monolithic ceramics - General and textural properties - Part 3: Determination of grain size and size distribution (characterized by the Linear Intercept Method)

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

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

This European Standard was approved by CEN on 19 April 2001.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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Foreword

This European Standard 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of an identical text or by endorsement, at the latest by November 2001, and conflicting national standards shall be withdrawn at the latest by November 2001.

This European Standard supersedes ENV 623-3:1993.

Annexes A, B, C, D, E, F and G are informative.

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

1 Scope

This Part of EN 623 describes manual methods of making measurements for the determination of mean linear intercept grain size of advanced technical ceramics using photomicrographs of polished and etched test pieces. This is not the true mean grain diameter, but a somewhat smaller parameter representing the average path length of a line drawn across a two-dimensional section. The relationship to true grain dimensions depends on grain shape and degree of microstructural anisotropy. This standard contains two methods, A and B.

Method A applies to single-phase ceramics, and to ceramics with a principal crystalline phase and a glassy grain-boundary phase of less than about 5% by volume for which intercept counting suffices. Method B applies to ceramics with more than about 5% by volume of pores or secondary phases, or ceramics with more than one major crystalline phase where individual intercept lengths are measured, which can optionally be used to create a size distribution. This latter method allows the pores or phases to be distinguished and the mean linear intercept size for each to be calculated separately.

NOTE A method of determining volume fraction(s) of secondary phase(s) is under development as ENV 623-5; this will provide a means of determining whether Method A or Method B should be applied in borderline cases.

Some users of this standard may wish to apply automatic or semiautomatic image analysis to micrographs or directly captured microstructural images This is permitted by this standard provided that the technique employed simulates the manual method (see clause 4 and 8.4).

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 are 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 (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 standard, the following terms and definitions apply.

3.1
grain size

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

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 Significance and use

The mean grain size and the distribution of grain sizes of a ceramic material play an important role in determining many properties, and thus grain size characterization is an important tool for ensuring consistency of manufacture. There are many measures of grain size and/or shape, but the linear intercept method provides the simplest possible method from a two dimensional section through the material. However, it must be recognised that the numerical value obtained for the mean linear intercept size is somewhat smaller than most other measures of grain size because intercepts can cross grains at any position, and not necessarily along the largest axis. The relationship between mean linear intercept size and a true three-dimensional grain size is not simple, and depends on the grain shape and the average number of facets.

NOTE Annex A contains a bibliography of sources dealing with stereology and methods of sizing three-dimensional objects.

This Standard provides a simple method of measuring intercept distances in single-phase materials based on counting the number of intersections along given lengths of randomly orientated and positioned lines or randomly positioned circles drawn onto a micrograph of a suitably sectioned, polished and etched test-piece. The length of lines crossing large pores residing at grain boundaries can be ignored, thus eliminating any bias that porosity may introduce, but small pores within grains should be ignored. In materials which contain more than one phase, the phases may be continuous or as isolated grains. It may be necessary to characterize the different phases separately. The principal purpose of this standard is to permit characterization of the major phases. The same intercept principle as for single-phase materials

can be used, but the individual intercept lengths across each phase must be measured, rather than just counted. The characterization of minor phases may require different treatment, which is outside the scope of this Standard.

If the material possesses a microstructure which has a preferred orientation of the primary or secondary phases, the results of this measurement may not be representative of the true character of the material. Rather than using randomly orientated lines, it may be necessary to make measurements restricted to specific orientations. If undertaken, this must be reported in the report.

This Standard does not cover methods of measuring mean grain size by counting using calibrated microscope stage movement or projection onto screens, accompanied by visual observation. While this latter method may produce an equivalent result to the analysis of micrographs, it does not provide a means of verification of the results of the measurement, since no permanent record is obtained.

If automatic or semiautomatic image analysis (AIA) is to be used it must be recognised that different AIA systems approach the measurement in different ways, and may use different parameters to linear intercept distance, such as those based on grain area by pixel counting. In order to obtain results equivalent to those of the manual method described in this standard, the AIA system needs to be programmed to operate in a similar way to the manual method. By agreement between parties, such a near-equivalent AIA method may be used as an alternative to the manual method, and if undertaken must be reported in the report.

5 Apparatus

5.1 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 mm to 150 mm and shall be cooled.

NOTE The grit size is designated D151 in ISO 6106, see annex A.

5.2 Mounting equipment

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

5.3 Grinding and polishing equipment

Suitable grinding and polishing equipment, employing diamond abrasive media.

NOTE Annex B recommends techniques and abrasives.

5.4 Microscope

An optical or scanning electron microscope with photomicrographic facilities. 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 quality of polishing (see 6.4).

5.5 Calibrated rule or scale

A calibrated rule or scale reading to better than 0.5 mm and accurate to better than 0,5%.

6 Test piece preparation

6.1 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 the measurement, it is desirable to maintain full knowledge of the positions within components or test pieces from which sections are prepared.

6.2 Cutting

The required section of the test piece shall be cut using the diamond saw (see 5.1).

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.

6.3 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 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 materials. The method of mounting selected should take into account the etching procedure to be used; see annex C.

6.4 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 5.4) at high magnification. The final surface shall be free from optically visible scratches, or other damage introduced by polishing, which would interfere with the determination.

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 minimization 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 B.

6.5 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 C.

7 Photomicrography

7.1 General aspects

If the grain structure of the test material is too small for optical microscopy adequately to resolve and count grain boundary intersections (Method A) or measure the individual grains (Method B), scanning electron microscopy is to be used.

NOTE Typically, if the mean linear intercept size of the principal phase is less than 2 µm for Method A, or 4 µm for Method B, then scanning electron microscopy should be used.

7.2 Optical microscopy

Set up Köhler illumination in the microscope.

NOTE Guidance on setting Köhler illumination 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 metallic coating by evaporation or sputtering. Prepare micrographs of at least three different areas of the test piece surface. As a guideline for Method A the average size of each distinct grain should appear typically at least 3 mm across. For Method B, the typical size of discrete phase areas or pores should appear at least 5 mm across. If the grains or phase areas appear smaller than these levels, increase the magnification and prepare fresh micrographs. Micrographs should be typically of a size 100 mm x 75 mm, but may with advantage be enlarged later to aid evaluation.

7.3 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 into the microscope, ensuring that the surface to be characterized is normal to the electron beam to within 5^{o}.

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

Prepare micrographs at a suitable magnification (see 7.2) from at least three different areas of the test piece.

7.4 Calibration micrographs

7.4.1 Optical microscopy

For optical microscopy, unless already undertaken, prepare a micrograph of a graticule at the same magnification as that used for preparing micrographs to provide a calibration of magnification. Measure the size of the spacing of the calibrated graticule as shown by a micrograph and calculate the magnification.

7.4.2 Scanning electron microscopy

For calibration of the lateral and vertical magnifications of the scanning electron micrographs, prepare similar images of a graticule or grid, or of calibrated spheres, at the same working distance of the microscope stage as that used for taking micrographs.

NOTE 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 (X-direction) and vertical (Y-direction) magnifications appear to be constant but different, it is possible to make corrections when measuring the micrographs. The effective magnification of each drawn line can be calculated by noting its angle relative to the horizontal on the micrographs and applying an angular correction to the X-direction magnification. This procedure may only be adopted by agreement between parties, and shall be reported (see clause 11).

Use the same procedure as for optical micrographs (see 7.4.1) to calculate the magnification horizontally and vertically. If calibration spheres have been used, measure the horizontal and vertical dimensions of at least six spheres and calculate the respective mean values. If the vertical and horizontal magnifications calculated are different by more than 5% or individually vary by more than 5% across the screen, the distortion of the image is not acceptable for the purposes of this standard.

8 Measurement of micrographs

8.1 General

Inspect the micrographs. If they appear to be essentially single phase and to contain less than 5% of a secondary phase, use Method A. If they appear to contain 5% or more of a secondary phase, either continuous or as discrete grains, employ the procedure given in Method B. If the requirement is for determining additionally a grain size distribution, use Method B.

8.2 Method A

Draw at least five thin straight lines of random position and orientation across each micrograph intersecting at least 100 grains.

NOTE 1 On a micrograph of typical size 100 mm x 75 mm showing grains averaging 3 mm across satisfying the requirements of 7.1, five lines of length 75 mm will provide an adequate number of grain intersections for this test method.

Measure each line length to the nearest 0,5 mm using the calibrated rule or scale (see 5.5) 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, a wide grain boundary, or a secondary phase, either discrete or continuous, 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.

Alternatively, on each micrograph draw at least three circles of diameter not less than 10 times the expected mean grain size using a pair of compasses and randomly positioning the centres. Measure the diameters of the circles *d* to the nearest 0,5 mm using the calibrated rule or scale (see 5.5), and calculate the sum of their circumferences *L(t)*. Count the number *N(i)* of intersections of each circle with the grain boundaries. If the intersection coincides with the junction of three grains, count this as 1,5 intersections. If the line intersects a large pore, a wide grain boundary, or a secondary phase, either discrete or continuous, count this as one intersection. Measure the approximate arc length that crosses large pores *L(p)*.

NOTE 2 For the purposes of this standard, a large pore is one which resides at grain boundaries. Small pores entrained within grains should be ignored.

8.3 Method B

Draw at least five randomly positioned and randomly orientated lines across the micrograph such that at least 100 discrete phase regions or pores of the type to be assessed are intersected. Ignore grains which touch the edge of the micrograph. Using a visual aid as necessary, measure the distance, L_i, between intersections of grain boundaries across each phase region or pore to the nearest 0,5 mm using the calibrated rule or scale (see 5.5). Count the total number of phase regions or pores, *N(g)*, measured.

8.4 Use of automatic or semiautomatic image analysis for methods A and B

If it is desired to apply an automatic or semi-automatic image analyser to the measurement of micrographs or directly recorded images, in order that the results are comparable with the manual method described in this standard the following points are to be noted:

(a) Care must be taken that the contrast change at a grain boundary is sufficient for the detection system to identify it as such. If the captured image requires enhancement to more clearly reveal grain boundaries, this should be performed manually rather than using any proprietary software until confidence is built up that the software method produces equivalent results.

(b) The image must be line-scanned in at least five random directions, which may be achieved either through software design or by rotating the image to random orientations and taking horizontal line scans. Scanning in only one direction on the test piece is not acceptable since it does not allow for anisotropy.

(c) The analyser must be calibrated for magnification using micrographs or images of a graticule or grid, as for the manual methods.

(d) The calculation routine incorporated in the software must operate in the same way as this manual method in order that large pores are discounted.