BS EN 623-3:2001

Advanced technical ceramics — Monolithic ceramics — General and textural properties —

Part 3: Determination of grain size and size distribution (characterized by the linear intercept method)

The European Standard EN 623-3:2001 has the status of a
British Standard

ICS 81.060.30

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National foreword

This British Standard is the official English language version of EN 623-3:2001. It supersedes DD ENV 623-3:1993 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

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This British Standard, having been prepared under the direction of the Sector Committee for Materials and Chemicals, was published under the authority of the Standards Committee and comes into effect on 15 August 2001

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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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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 × 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°.

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 × 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.

(e) The report shall contain full documentation of the procedure employed.

NOTE Failure to observe these points will result in results which may be substantially at variance with the manual method.

9 Calculation of results

9.1 Method A

For both line and circle methods, calculate the mean linear intercept distance, g_{mli}, in micrometres, for each micrograph using the formula:

$$g_{\mathrm{mli}} = \frac{\left[L(t) - L(p)\right] \cdot 10^3}{N(\mathrm{i}) \cdot \mathrm{m}} ;$$

where: $L(t)$ is the total line length in millimetres; in the case of circles, the total circumference of the circles, in mm;
$L(p)$ is the total line length that crosses large pores, in mm;
$N(i)$ is the counted number of intersections on each micrograph;
m is the calibrated magnification of the micrograph.

Calculate the mean value of g_{mli} from the values determined for each of the individual micrographs used.

9.2 Method B

Calculate the mean linear intercept distance g_{mli}, in micrometres, of each discrete phase region or pores as follows:

$$g_{\mathrm{mli}} = \frac{[\Sigma L_i] \cdot 10^3}{N(g) \cdot \mathrm{m}} ;$$

where: L_i is the ith individual intercept length in millimetres;
Σ is the summation sign;
$N(g)$ is the number of discrete phase regions or pores counted;
m is the calibrated magnification of the micrograph.

10 Interferences and uncertainties

The nature of the microstructure of the test piece can affect the result determined by this test, especially in cases where there is a wide distribution of grain sizes (e.g. a bimodal distribution), or where it is difficult to find an adequate etching method to reveal grain boundaries. Method A assumes that the amount of continuous secondary phase is small compared with the major crystalline phase(s). As the widths of the layers of such secondary phase between grains of the primary phase increase, there will be an increasing overestimate of true mean grain size, and Method B should preferably be used. Method B also assumes that the total fine-scale porosity level is negligible.

The principal causes of uncertainty in this method are considered to be the random errors of selecting areas of the test piece from which to prepare micrographs and the positions on the micrograph in which to draw lines or circles. The former depends on the homogeneity of the microstructure within the test piece, and the latter on any subjective element in selecting line or circle positions.

Uncertainties arising from magnification and counting are considered to be negligible provided that the procedure described in this standard is followed.

NOTE An international round-robin has demonstrated the potential causes of scatter in undertaking measurement according to Method A. The findings are summarized in annex E.

11 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 standard, i.e. EN 623, Part 3.

g) A summary of the procedure for sampling, cutting, grinding, polishing and etching the test piece.

h) The observation technique employed (optical or scanning electron microscope).

i) The technique employed for calibration, and the resulting magnification.

j) Copies of the micrographs with their magnifications used for the measurement.

   NOTE 1 If AIA has been used, both the original and the digitally enhanced images should be provided.

k) If a manual method was employed, whether Method A or Method B was used, and if Method A, whether lines or circles were used for the analysis.

l) If an automatic or semiautomatic method was used, full documentation of the procedures employed, including details of image enhancement (if used), and the basis for the calculation method employed.

m) Any use of the angular correction method (see 7.4).

n) For Method A, the number of intercepts for each of the five lines or three circles on each of the three micrographs and the total line length, corrected for large pores, employed for the measurements, expressed in millimetres.

o) For Method A, the total number of intercepts.

p) For Method B, the phase types or pores measured, the individual intercept lengths, expressed in millimetres, and the total number of discrete phase regions or pores counted for each of the micrographs.

q) For Method A and Method B the calculated mean linear intercept size for each of the micrographs, expressed in micrometres to two significant figures, and the overall mean value.

r) If appropriate, the intercept size distribution using Method B for each discrete phase type.

NOTE 2 Annex F contains a method by which data may be ranked for the purposes of preparing an intercept size distribution.

s) Any remarks on the general appearance of the microstructure, whether isotropic or anisotropic, the presence of secondary phases, whether the grain size is obviously bimodal, or the grain shape is anisotropic.

t) Signatures of persons responsible for the test and authorising issue of report.

NOTE 3 For routine presentation of results it is useful if a standardized format is adopted. A recommended scheme is presented in annex G.

u) Comments on the test or test results, including any deviations from the procedure required by this standard.

Annex A
(informative)

Bibliography on stereology and grain size measurement

ASTM E112, *Standard test method for determining average grain size*, *ASTM Annual Book of Standards,* Vol. 3.01.

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Annex B
(informative)

Grinding and polishing procedures

Preparation of polished sections of ceramics requires different procedures from those conventionally employed for metallic materials, which typically commence with a coarse grinding stage using fixed grit silicon carbide papers of grit sizes of 30 mm or greater (see reference B.1 for information on grit size coding). For ceramic materials, this type of procedure can produce considerable amounts of subsurface damage in the form of extended microcracks which can then influence the microstructural appearance obtained, unless precautions are taken to minimize such damage and to remove all traces of it in subsequent grinding steps. Unless care is taken, the final surface may contain damage which manifests itself as microcracks and grain tear-out, the presence of which can influence the results of any microstructural characterization measurement. Thus, selection of appropriate polishing procedures, including the sequence of grit sizes, the times of abrasion, and the applied pressure are all important. Optimum conditions vary considerably depending on the type of material being prepared. Guidelines on how to choose a grinding method may be found in Hübner and Hausner (see reference B.2).

As an example, a series of metal-bonded diamond grinding discs give high material removal rates for initial flattening. However, grit sizes greater than 30 mm may introduce damage, especially in materials of poor toughness, and smaller grit sizes used for longer periods of time may produce a better result. Loose diamond abrasives remove material more slowly than grit of the same size fixed in a disc, and may cause more damage. Subsequent grinding steps may need to be of longer duration. The use of a shock-absorbing system, such as a soft metal lap (e.g. tin) into which loose grit becomes lodged, or a metal-plastic composite lap with fixed diamond grit, gives a good balance between speed of abrasion and surface damage.

The grinding of silicon carbide ceramics can cause special difficulties. Klimek (see reference B.3) recommends that the diamond abrasive used should not be larger than 6 mm, since a larger size of abrasive tends to shatter large SiC grains rather than to produce cutting.

After a planar surface is achieved with the initial grinding stage, a sequence of finer grit sizes may be employed to remove grinding damage from previous steps. The precise sequence of stages chosen will depend on equipment available, and may have to be optimized for each type of material. The general principle should be that each step should be of sufficient duration to remove evidence of damage from the previous stage. The final polishing stage should not be undertaken until a good quality finish is obtained. The use of napped cloths for polishing is not recommended because on many types of ceramic it can cause pluck-out of grains (especially with high-alumina ceramics) or loss of flatness of surface. Polishing procedures have been described by Clinton (see reference B.4). A series of articles on microstructural preparation of ceramics with polishing details is given in reference B.5.

The following five-stage procedure is recommended as a starting point for fine-grained ceramics, and gives surfaces of sufficient quality for examination at high magnification in the scanning electron microscope:

1. 30 µm diamond on a hard composite lap;

2. 6 µm diamond on a softer composite lap;

3. 1 µm diamond on a hard napless cloth (or a tin lap);

4. 0,25 µm diamond on a hard napless cloth;

5. colloidal silica in alkaline solution on a hard napless cloth.

NOTE The last step is intended to remove scratches from the polished surface, which it does very successfully. However, there is a risk of pores becoming filled with polishing debris which is impossible to remove, and this step should not be used if evaluation of porosity content is required. Such pick-up should not influence grain size measurement. It is recommended that the lap is kept wet at all times, and that polishing is continued with water for a short while at the end to prevent the build-up of deposits on the surface.

Before moving from one stage to the next, the test piece should be carefully cleaned of abrasive grit using an ultrasonic bath and a suitable liquid cleaning agent, and should be examined in an optical microscope to ensure the surface is uniform and that damage from the previous stage is minimized.

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Annex C
(informative)

Etching procedures

With many ceramic materials it is necessary to reveal the positions of grain boundaries for the purpose of this test. A variety of techniques are available for doing this, but the choice and the severity of the process may depend on the precise nature of the material and the technique used to observe the microstructure. Some experimentation is often needed to set appropriate conditions for unfamiliar materials. Over-etching is to be avoided, since it can modify the appearance of the microstructure. It is recommended that the optimum etching conditions are determined in a step-wise fashion to ensure that over-etching does not occur. It may be necessary to use more severe etching for SEM images than for optical images in order to produce adequate contrast at grain boundaries.

Bibliographic lists of etching methods have been given by Clinton and Petzow (see references C.1 and C.2), and further information is given in references C.3 to C.5. Table C.1 shows some examples.

Table C.1

Ceramic	Method	Conditions
Alumina (> 99.5 %)	Thermal	1 500 °C, 2 h (see note)
Alumina (lower purity)	Chemical or thermal	10 % HF, 20 s; 1 450 °C, 1 h (see note)
Zirconia-toughened alumina	Thermal	1 500 °C, 15 min (see note)
Yttria-TZP	Thermal	1 300 °C, 2 h to 1 420 °C, 15 min (see note)
Ce-TZP	Thermal	1 450 °C, 5 min (see note)
Sialons and sintered silicon nitrides	Plasma etch (see C.6 and C.7)	CF_4 plasma etch, 40 s
Hot-pressed silicon nitride	Chemical	NaOH, 400-450 °C, 1-10 min CF_4 plasma etch, 40 s
Aluminium nitride	Relief polished	Colloidal silica, alkaline solution
Sintered silicon carbide	Chemical	Modified Murakami’s reagent, e.g. 3 g KOH, 30 g $K_3Fe(CN)_6$, 60 ml H_2O, boil for 2 min to 20 min

Thermal etching used for oxide ceramics can give good clear delineation of grain boundaries, but there is a risk of modifying the microstructure of the product in the process. The maximum temperature for this process should be at least 150 °C below the original firing temperature of the ceramic (for the same time period) to minimize the risks. In addition, the presence of glassy secondary phases can cause problems of contamination of the grain surfaces as it is usually mobile at the required thermal etching temperatures.

Chemical methods, particularly those involving melts, can be difficult to control and reproduce. Ensure that the test piece is clean and free from grease before using aqueous etchants. If a test piece is over-etched, smaller grains may disappear. Ceramics with continuous secondary phases are generally more easily etched than those without, but caution is required if the primary phase is also continuous, e.g. in reaction-bonded silicon carbide, or in some high-alumina ceramics. The true grain boundaries may not all be revealed.

Many etching processes, particularly thermal etching, will require that the test piece mounting or impregnation medium is removed beforehand.

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Annex D
(informative)

Setting Köhler illumination in an optical microscope

D.1 Purpose

The principal purpose behind setting up the correct illumination is to ensure that the intensity across the image width is uniform for the purposes of photomicrography.

D.2 Definition

Köhler illumination is achieved when an image of the illumination source is projected by a collecting lens into the plane of the aperture diaphragm positioned in the front focal plane of the condenser lens. This latter lens, in turn, projects an image of the illuminated field diaphragm at the condenser lens into the object plane.

D.3 Setting up for Köhler illumination

The following instructions are the basic principles. Different microscopes may have different means of achieving these steps, and reference to the equipment handbook is recommended.

Switch on the illumination system. Choose a reflective specimen, e.g. a metal graticule, and a low magnification objective lens, typically ×10. Focus the microscope on the specimen in the normal way. Fully open the condenser aperture iris. Remove the eyepiece and sight down the microscope tube. Observe the image of the lamp filament in the back focal plane of the objective (alternatively, if fitted, a Bertrand lens can be introduced and this image observed without removing the eyepiece). Adjust the lateral position of the lamp filament until it appears centrally in the field of view. Adjust the condenser lens position (or the lamp collector lens, depending on the system design) until the image is sharp and in focus at the same time as the condenser iris diaphragm. Replace the eyepiece (or remove the Bertrand lens). Close the collector lens aperture until the field of view begins to darken, and then open it a little. The objective is now collecting the maximum angle cone of light without excess scattering or internal reflections.

If the objective lens is subsequently changed, the optimum Köhler illumination should be checked unless it has previously been established that the same positions of adjustment apply to all lenses in the instrument.

Annex E
(informative)

Round-robin verification of the procedure in this standard

An international round-robin under the auspices of VAMAS Technical Working Area 3 was conducted in 1992 to verify Method A described in this standard, and to determine the likely level of variability resulting from the randomness of positioning lines or circles on the microstructure. Twenty-five organisations took part in a multistage exercise designed to establish:

1. The scatter in mean linear intercept size due to random positioning of lines or circles on a computer-drawn, pore-free “grain” structure formed through a Dirichlet tessellation, in which the grain boundaries are very distinct.

2. The scatter in mean linear intercept size due to random position of lines and circles on a given real microstructural image showing significant porosity and in which grain boundaries were less distinct.

3. The scatter in mean linear intercept size due to randomness in choice of an area to measure.

4. Any significant differences between line and circle methods.

The findings (see references E.1 and E.2) indicated that:

1. Under conditions in which counting of grain boundaries is easy, provided that at least 100 intercepts are recorded, random positioning factors result in a scatter about the mean result of ≤ ±10 % at a confidence level of 95 %.

2. For a given micrograph, under conditions in which grain boundaries need to be carefully interpreted by the observer, and in the presence of porosity, the scatter about the mean result is ≤ ±20 % at a confidence level of 95 % when several sets of data were excluded on the grounds that too few intercepts had been recorded, or that concentric circles had been incorrectly used.

3. Under conditions in which a randomly selected area of a participant-prepared polished and thermally etched test piece was examined, the scatter about the mean result is ≤ ±25 % at a confidence level of 95 %; the apparent grain size increased with the thermal etching temperature employed, presumably as smaller grains disappeared as the boundaries became more deeply etched.

4. There was no significant difference between the results for the line and circle methods provided that at least 100 grains were counted, and that the circles were not drawn concentrically, and were of radii at least 10 times the mean grain size.

References:

E.1 Dortmans, L.J.M.G., Morrell, R., De With, G., *Round-robin on grain size measurement for advanced technical ceramics*, VAMAS Technical Report No. 12, issued through National Physical Laboratory, Teddington, UK, 1993.

E.2 Dortmans, L.J.M.G., Morrell, R., De With, G., *Round-robin on grain size measurement for advanced technical ceramics*, J. Eur. Ceram. Soc., 12 [1993], 205-13.

Annex F
(informative)

Grain size distribution measurement

Using the individual intercept lengths measured in accordance with Method B (see 7.2), rank the intercept lengths in ascending order of size and prepare a cumulative distribution plot of fractional rank $n/(N(g) + 1)$ against intercept size g_{li}, where n is the rank number, and $N(g)$ is the total number of intercept lengths. Identify the intercept sizes at the 10 % ($g_{\mathrm{li},10}$), 50 % ($g_{\mathrm{li},50}$) and 90 % ($g_{\mathrm{li},90}$) positions in the distribution.

NOTE It should be noted that $g_{\mathrm{li},50}$ will not normally have the same value as g_{mli} determined by the counting method because grain size distributions are typically log-normal in character, a factor ignored with the counting method.

Annex G
(informative)

Results sheet — Grain size in accordance with EN 623-3

Testing establishment		
Report reference number		
Client name and address		
Date of receipt of test piece		
Date of test		
Test piece reference number and manufacturing details		
Size of sampled ceramic		
Number of test pieces		
Test piece preparation	Method of cutting Method of mounting Method of grinding and polishing Method of etching	
Method of observation	Type of microscope Calibration method Calibrated magnification Method of recording images Method of processing images (manual in accordance with EN 623-3, automatic)	
Results for each micrograph	Total line length Number of intercepts for each line Mean linear intercept size	
Remarks Problems with preparation Features of microstructure Aspect ratio of grains		
Micrographs		
Signature of person undertaking measurement		
Authorizing signiture		

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