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Fine ceramics (advanced ceramics, advanced technical ceramics) — Microstructural characterization —

Part 1:
Determination of grain size and size distribution

Céramiques techniques — Caractérisation microstructurale —

Partie 1: Détermination de la grosseur du grain et de la distribution granulométrique

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13383-1 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

ISO 13383 consists of the following parts, under the general title *Fine ceramics (advanced ceramics, advanced technical ceramics) — Microstructural characterization*:

— *Part 1: Determination of grain size and size distribution*

— *Part 2: Determination of phase volume fraction by evaluation of micrographs*

Fine ceramics (advanced ceramics, advanced technical ceramics) — Microstructural characterization —

Part 1: Determination of grain size and size distribution

1 Scope

This part of ISO 13383 describes manual methods of making measurements for the determination of grain size of fine ceramics (advanced ceramics, advanced technical ceramics) using photomicrographs of polished and etched test pieces. The methods described in this part do not yield the true mean grain diameter, but a somewhat smaller parameter depending on the method applied to analyse a two-dimensional section. The relationship to true grain dimensions depends on the grain shape and the degree of microstructural anisotropy. This part contains two principal methods, A and B.

Method A is the mean linear intercept technique. Method A1 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 A2 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) can be found in ISO 13383:2; this will provide a means of determining whether Method A1 or Method A2 should be applied in borderline cases.

Method B is the mean equivalent circle diameter method, which applies to any type of ceramic with or without a secondary phase. This method may also be employed for determining grain aspect ratio and a size distribution.

Some users of this part of ISO 13383 may wish to apply automatic or semiautomatic image analysis to micrographs or directly captured microstructural images. This is permitted by this part provided that the technique employed simulates the manual methods (see Clause 4 and 8.4).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

For the purposes of this document, 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
g_{mli}
average value of the distance between grain boundaries as shown by randomly positioned lines drawn across a micrograph or other image of the microstructure

3.3
equivalent circle grain diameter
d_{ci}
diameter of a circle which closely matches the perimeter of a grain

See Figure 1.

3.4
maximum (Feret) grain size
$d_{ci, max}$
maximum dimension of a grain viewed in two dimensions

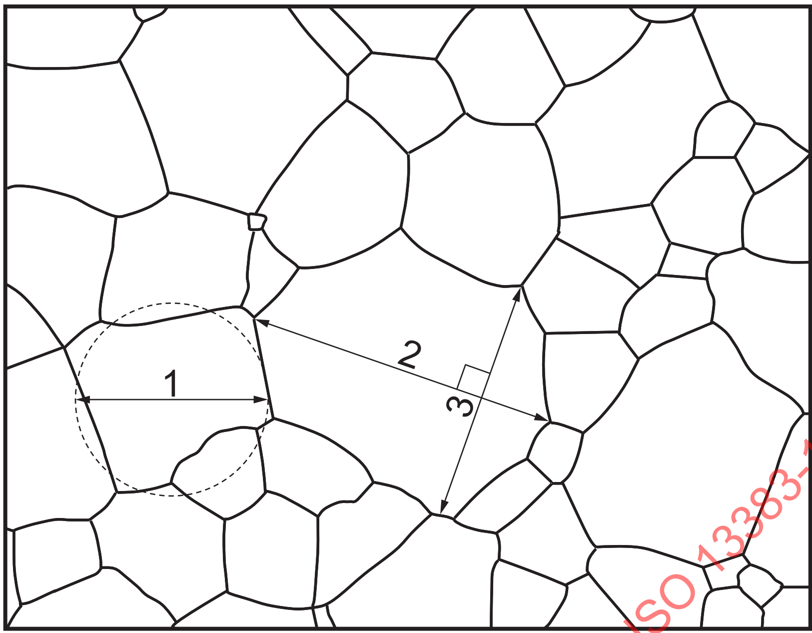
See Figure 1.

NOTE This is also termed maximum caliper diameter in ASTM E930.

3.5
maximum orthogonal grain size
$d_{ci, perp}$
for the purposes of determination of grain aspect ratio, the largest dimension of a grain normal to its maximum (Feret) grain dimension, viewed in two dimensions

See Figure 1.

3.6
grain aspect ratio
ratio of maximum (Feret) grain size to the maximum orthogonal grain size measured perpendicular to it



Key

1 Equivalent circle grain diameter, d_{ci}

2 Maximum grain (Feret) size, $d_{ci, max}$

3 Maximum orthogonal grain size perpendicular to 2, $d_{ci, perp}$

Figure 1 — Equivalent circle diameter and definition of aspect ratio

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, and these are usually of different numerical values for a given microstructure.

NOTE The Bibliography contains sources dealing with stereology and methods of sizing three-dimensional objects.

The principal purpose of this part of ISO 13383 is to permit characterization of the major phases. However, 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 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 part of ISO 13383.

Method A, the linear intercept method, provides the simplest possible method from a two-dimensional section through the material. However, it must be recognized 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. This part of ISO 13383 provides simple methods 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.

Method B, the mean equivalent circle diameter method, provides an alternative approach based on identifying the radius of a circle which most closely approximates the boundary of the grain. This measure usually gives a result which is a little larger than that from the mean linear intercept method because it is based on area and not random intercept length. The method may also be used to measure grain aspect ratio, and is therefore more appropriate for microstructures with elongated grains.

NOTE This method is taken from JIS R1670 [1].

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 Test Report. Method B may be more appropriate.

This part of ISO 13383 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 recognized that different AIA systems approach the measurement in different ways, usually based on pixel counting. In order to obtain results equivalent to those of the manual methods described in this part of ISO 13383, the AIA system needs to be programmed to operate in a similar way to the manual method. By agreement between the parties concerned, such a near-equivalent AIA method may be used as an alternative to the manual method, and if undertaken must be reported in the Test Report.

5 Apparatus

5.1 Sectioning equipment

A suitable fine-grained diamond-bladed cut-off saw with a liquid cooling or other device to prepare the initial section for investigation.

NOTE A grit size of 125 µm to 150 µm is recommended, designated as D151 in ISO 6106 [2].

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 A recommends techniques and abrasives.

5.4 Etching equipment

Etching equipment appropriate to the etching process to be used to reveal grain boundaries in the material being examined.

NOTE Annex B provides some guidelines for etching methods.

5.5 Microscope

An optical or scanning electron microscope with photomicrographic facilities. A calibrated stage micrometer 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 the quality of polishing (see 6.4).

5.6 Calibrated rule or scale

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

5.7 Circle template

For method B, a stencil cut with circles of diameter in 1 mm increments, or a transparent sheet with circles drawn in a series of 1 mm increments. The line thickness on a transparent sheet shall not exceed 0,2 mm.

6 Test piece preparation

6.1 Sampling

The test pieces shall be sampled in a manner subject to agreement between the parties concerned.

NOTE Guidance on this issue may be found in EN 1006 (see Bibliography [3]). 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 sectioning device (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 B.

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 5.5) 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 part of ISO 13383 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 while retaining a flat surface. Some guidelines on grinding and polishing are given in Annex A.

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 appropriate to the ceramic material class shall be used, subject to agreement between the parties concerned. Excessive intensity of etching shall be avoided.

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

7 Photomicrography

7.1 General aspects

Either optical microscopy or scanning electron microscopy may be used, the latter being required if the grain structure is on a scale finer than can be resolved adequately by optical microscopy, according to the requirements for the minimum observed sizes of grains or second phases in the prepared images.

NOTE Typically, if the mean linear intercept size of the principal phase is less than about 2 µm for Method A1, or less than about 4 µm for Methods A2 and B, then scanning electron microscopy should be used.

7.2 Optical microscopy

Set up Köhler illumination in the microscope.

NOTE 1 Guidance on setting up Köhler illumination is given in Annex C.

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 areas of the test piece surface.

NOTE 2 The important aspect of area selection is that it should be random and representative of the test material. Depending on the purpose of the investigation, it should be agreed between the parties concerned whether it is more important to employ several images from a single polished sample, or individual images from a number of samples in a batch. Furthermore, if the material appears to be inhomogeneous, or to have a wide distribution of grain sizes, it may be advantageous to evaluate more areas less intensively than in the case of a very uniform microstructure.

As a guideline for Method A, the average size of each distinct grain should appear at least 2 mm and preferably at least 3 mm across in the evaluated image. 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. Printed micrographs should be typically of a size at least 100 mm x 75 mm, but may with advantage be enlarged 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 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 7.2) from at least three different areas of the test piece, using the same visual guidelines as for optical images.

NOTE 2 The appearance of micrographs may vary depending on the accelerating voltage employed. Voltages of less than 15 kV may be advantageous in improving contrast.

7.4 Calibration micrographs

7.4.1 Optical microscopy

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

7.4.2 Scanning electron microscopy

For calibration of the lateral (X-direction) and vertical (Y-direction) magnifications of the scanning electron micrographs, prepare similar images of a calibrated grid, or of calibrated spheres, at the same operating voltage and working distance of the microscope stage as that used for taking micrographs.

NOTE The photographic screen or image capture system 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 field of view, since it is easy to detect distortions of the grid. If the image distortion is uniform across the field of view, i.e. X and Y magnifications appear to be constant but different, it is possible to make corrections when measuring the micrographs. The effective magnification of each drawn line (see 8.2) can be calculated by noting its angle relative to the X direction on the micrographs and applying an angular correction to the X direction magnification. This procedure may only be adopted by agreement between the parties concerned, and be reported (see Clause 11).

Use the same procedure as for optical micrographs (see 7.4.1) to calculate the X and Y direction magnifications. 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 calculated X and Y direction magnifications 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 part of ISO 13383.

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 A1 or Method B. If they appear to contain 5 % or more of a secondary phase, either continuous or as discrete grains, employ the procedure given in Method A2 as an alternative to A1. If the requirement is for determining additionally a grain size distribution, use Method A2 or Method B.

Whichever method is employed, the confidence in the average grain size determination depends on the spread of apparent grain sizes and the number of independent grain dimensions measured. For a single-phase ceramic with visually uniform and isotropic size and shape of grains, counting about 100 grains or grain intercepts in total over all micrographs employed will provide an estimate of average grain size to within about ±10 % of the true average. For ceramics which do not meet this criterion, a larger number of grains or grain intercepts generally needs to be counted to achieve this level of confidence. If a more accurate estimate is required, a larger number of grains or grain intercepts needs to be counted.

Thus, for routine quality control purposes on a uniform-grained material which has demonstrable consistency, counting about 100 grains in total over three representative areas may be sufficient. For a material with initially unknown microstructure, or which may be multiphase, or which has a preferred grain orientation or a wide grain size distribution, typically 300, perhaps 500, grains in total may be required.

NOTE 1 For some applications, it may be more important to sample systematically a large number of test items or areas within a test piece rather than focus on the minimum of three randomly selected areas.

NOTE 2 If it is uncertain whether sufficient grains or grain intercepts have been counted, a 'cumulative moving average' size should be computed as the count proceeds. Plotting the cumulative moving average against the number of grains or intercepts counted provides a visual trend of progress towards a stable final result within the uncertainty band required for the estimate.

Methods A1 (see 8.2), A2 (see 8.3) and B (see 8.4) are intended for manual measurement of printed micrographs of a material treated as 'unknown' and requiring precautions in measurement. Additional factors when using semi-automatic or fully automatic image analysis systems are described in 8.5. The described requirement for at least five randomly orientated drawn lines in Methods A1 and A2 may, with justification, be appropriately relaxed when the material is known to be of uniform, isotropic and consistent microstructure. Similarly, the total number of intersections or grains counted may be reduced, but in no case shall less than 100 features be counted (a minimum of 30 per micrograph).

8.2 Method A1

Unless otherwise justified, draw at least five thin straight lines of random position and orientation across each micrograph intersecting at least 100, preferably up to 300, grains in total over the minimum of three micrographs.

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.

NOTE 2 Random orientation of the lines ensures that the influence of any texture, local or general, is minimized.

Measure each line length to the nearest 0,5 mm using the calibrated rule or scale (see 5.6) and calculate the total line length $L(\mathrm{t})$. Count the number $N(\mathrm{i})$ 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 minor secondary phase, either discrete or continuous, count this as one intersection. Measure the total length of line that crosses large pores or inclusions, $L(\mathrm{p})$. If the line runs exactly 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 circle 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 $L(\mathrm{t})$. Count the number $N(\mathrm{i})$ 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 minor secondary phase, either discrete or continuous, count this as one intersection. Measure the approximate arc length that crosses large pores $L(\mathrm{p})$.

NOTE 3 For the purposes of this part of ISO 13383, a large pore is one which resides at grain boundaries. Small pores entrained within grains should be ignored.

8.3 Method A2

Unless otherwise justified, draw at least five randomly positioned and randomly orientated straight lines across each micrograph such that a total of at least 100, preferably up to 300, 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 5.6). Count the total number of phase regions or pores, $N(\mathrm{g})$, measured.

8.4 Method B

Overlay the micrographs in turn with the stencil or transparent sheet. For each complete grain in the micrograph (i.e. ignoring grains intersected by the micrograph border), identify the circle that best fits the boundary of the grain using the visual criterion that approximately equal areas of grain should be inside the circle as outside (see Figure 2). Note the circle diameter. All complete grains in the micrographs shall be measured. At least 100 grains, preferably up to 300, shall be measured.

If it is required to measure the grain aspect ratio, use the same procedure, but for each grain match the circles to the longest (Feret) grain diameter and to the maximum grain diameter perpendicular to the longest grain diameter (see Figure 1). Note these diameters. Alternatively, a calibrated rule may be used.

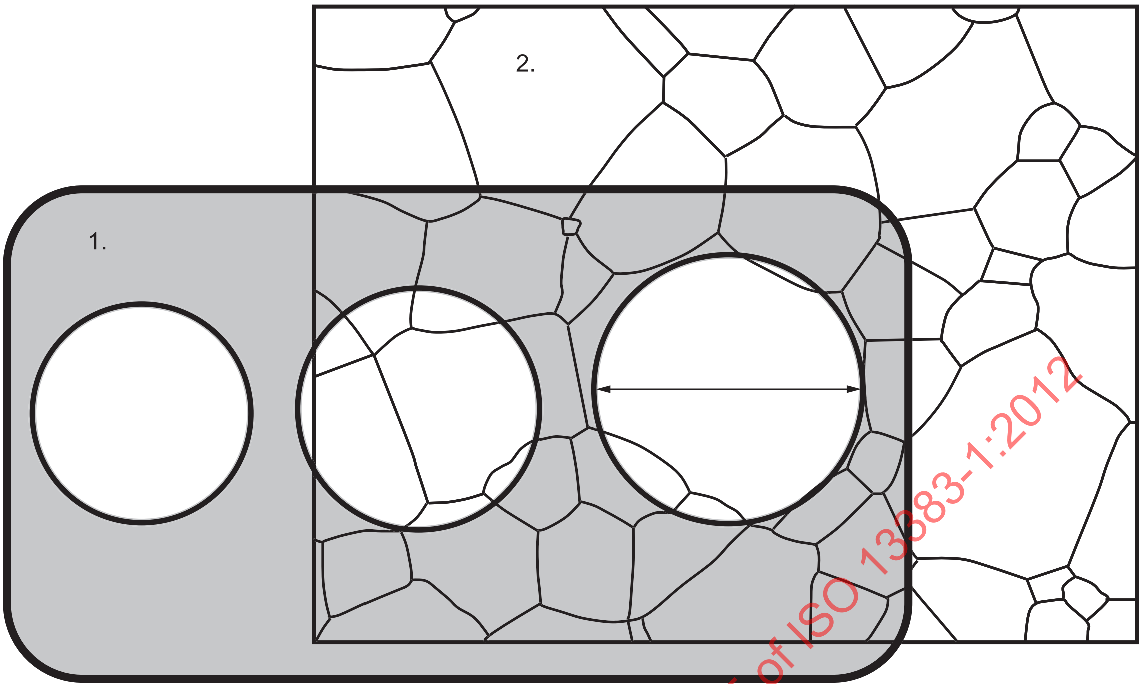


Figure 2 — Use of a stencil, 1, overlaid on a micrograph, 2, and visually matched to the selected grain being measured.

8.5 Use of automatic or semi-automatic 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 part of ISO 13383, 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 in order to reveal grain boundaries more clearly, this should be performed manually rather than using any proprietary software until confidence is built up that the software method produces equivalent results.

b) For Methods A1 and A2, the image should 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 generally acceptable since it does not allow for anisotropy unless justified by inspection.

c) The equivalent image analysis approach to Method B is to measure individual grain areas that do not intersect the edges of the micrographs, using pixel counting, and then to compute individual equivalent circle diameters. Care is needed to ensure uniform contrast across each grain and narrow grain boundaries, otherwise an underestimate of grain size will result.

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

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

f) The Test Report shall contain full documentation of the procedure employed.

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

9 Calculation of results

9.1 Method A1

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

$$g_{mli} = \frac{[L(t) - L(p)] \times 10^3}{N(i) \times m} \quad (1)$$

where

$L(t)$	is the total line length expressed in millimetres; in the case of circles, the total circumference of the circles, expressed in millimetres;
$L(p)$	is the total line length that crosses large pores or inclusions, expressed in millimetres;
$N(i)$	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 A2

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

$$g_{mli} = \frac{[\Sigma L_i] \times 10^3}{N(g) \times m} \quad (2)$$

where

L_i	is the i^{th} individual intercept length, expressed 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 micrographs.

If appropriate, create a linear intercept size distribution.

NOTE See Annex F for a suitable procedure.

9.3 Method B

Calculate the mean equivalent circle diameter grain size, g_{ecd}, expressed in micrometres, as follows:

$$g_{ecd} = \frac{[\Sigma d_{ci}] \times 10^3}{N(d) \times m} \quad (3)$$

where

d_{ci} is the equivalent circle diameter of the i th grain, in mm;

Σ is the summation sign;

N(d) is the number of grains measured;

m is the calibrated magnification of the micrographs.

If the mean grain aspect ratio is to be determined, calculate the mean aspect ratio, R, as follows:

$$R = \frac{[\Sigma R_i] \times 10^3}{N(d)} \quad (4)$$

where

R_i is the aspect ratio of the i th grain determined as $d_{ci,\ \mathrm{max}}/d_{ci,\ \mathrm{perp}}$;

$d_{ci,\ \mathrm{max}}$ is the Feret maximum grain length, in mm;

$d_{ci,\ \mathrm{perp}}$ is the maximum grain dimension perpendicular to the Feret maximum grain length, in mm.

If appropriate, create an equivalent circle diameter size distribution.

NOTE See Annex F for a suitable procedure.

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 A1 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 a secondary phase between grains of the primary phase increase, there will be an increasing overestimate of true mean grain size, and Method A2 should preferably be used. Method A2 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 part of ISO 13383 is followed.

NOTE 1 An international round-robin has demonstrated the potential causes of scatter in undertaking measurement according to Method A1. The findings are summarized in Annex D.

Method B has a stepwise approach to measurement, based on the circle diameter steps of the stencil or transparent sheet, and relies on human judgement to fit an appropriate circle to each irregular grain. Provided that sufficient grains are counted, the mean result should not have a bias corresponding to more than one measurement step.

NOTE 2 A national round-robin has demonstrated the consistency of this method and its relationship to method A1. The findings are summarized in Annex E.

The statistical uncertainty in the mean linear intercept size is controlled by the number of grain intercepts counted or grain diameters measured. Counting the minimum of 100 grains gives an estimate consistent to typically about ±8 %, expressed as a standard error of the mean (about ±16 %, expressed as the limits of the 95 % confidence interval). Counting 300 grains reduces this value to about ±3 % (about ±6 % expressed as the limits of the 95 % confidence interval). Note should be made of these uncertainty levels when comparing an experimental value with a specification level.

Methods A and Method B produce different numerical results because the measurement methods have different bases. Stereologically, the average of the randomly located intercept lengths in Method A is likely to be somewhat smaller than the average diameter of the circle of area equivalent to randomly located cross-sectional planar intersections.

11 Test report

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

a) the name of the testing laboratory;

b) a unique identification of the report;

c) the name and address of the client;

d) relevant 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 part of ISO 13383, i.e. ISO 13383-1:2012;

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), the technique employed for calibration, and the resulting magnification;

i) 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.

j) if a manual method was employed, whether Method A1, or Method A2, or Method B was used, and if Method A1, whether lines or circles were used for the analysis;

k) if an automatic or semi-automatic method was used, full documentation of the procedures employed, including details of image enhancement (if used), and the basis for the calculation method employed;

l) any use of the angular correction method (see 7.4);

m) for Method A1, the number of intercepts for each of the lines or circles on each of the micrographs, and the total line length corrected for large pores, inclusions and other features excluded from the evaluation, employed for the measurements, expressed in millimetres;

n) for Method A2, the discrete phase type or types measured, the individual intercept lengths expressed in millimetres, and the total number of grains of each discrete phase regions counted for each of the micrographs;

o) for Method A1 and Method A2, the calculated mean linear intercept size for each of the micrographs, expressed in micrometres to two significant figures, and the overall mean value;

p) if appropriate, the intercept size distribution using Method A2 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.

q) for Method B, the discrete phase type or types measured, the individual equivalent circle diameters expressed in millimetres, the total number of grains measured, and the mean equivalent circle diameter, expressed in micrometres to two significant figures;

r) if appropriate, the equivalent circle diameter distribution using the data from Method B;

NOTE 3 Annex F contains a method by which data may be ranked for the purposes of preparing an equivalent circle diameter 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; any comments on the test or test results, including any necessary deviations from the procedure required by this part of ISO 13383;

t) signatures of persons responsible for the test and authorising an issue of the report;

NOTE 4 For routine presentation of results, it is useful if a standardized format is adopted. A recommended scheme is presented in Annex G.

Annex A
(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 µm or greater (see [A.1] for information on grit size coding). For ceramic materials, this type of procedure can produce considerable amounts of sub-surface 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 [A.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 µm 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 discs, 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 [A.3] recommends that the diamond abrasive used should not be larger than 6 µm, 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 [A.4]. A series of articles on microstructural preparation of ceramics with polishing details is given in reference [A.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:

a) 30 µm diamond on a hard composite lap;

b) 6 µm diamond on a softer composite lap;

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

d) 0,25 µm diamond on a hard napless cloth;

e) colloidal silica in alkaline solution on a hard napless cloth.

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 particulate-free liquid cleaning agent, and should be examined in an optical microscope to ensure that the surface is uniform and that damage from the previous stage is minimized.

Bibliography for Annex A

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[A.2] Hübner, G., Hausner, H., Material-orientated preparation of sintered ceramic bodies, Prakt. Metallogr., 1983, 20, 289-296.

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[A.4] Clinton, D.J., A guide to polishing and etching of technical and engineering ceramics, Institute of Ceramics, Stoke-on-Trent, Staffs, UK, 1987.

[A.5] Carle, V., et al., Ceramography of high-performance ceramics - description of materials, preparation, etching techniques and description of microstructures:-Part II: Silicon carbide. Prakt. Metallogr., 1991, 28, 420-34. Part III - Zirconium dioxide (ZrO_2) (by Schäfer, U., et al.), ibid, 1991, 28, 468-83. Part IV - Aluminium nitride (AlN) (by Predel, F., et al.), ibid, 1991, 28, 542-52.

Annex B
(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 is 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 scanning electron microscopy (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 [B.1] and Petzow [B.2], and further information is given in references [B.3] to [B.5]. Table B.1 shows some examples.

Table B.1 — Some examples of etching procedures

Ceramic	**Method**	**Typical conditions**
Alumina (>99.5 %)	Thermal	1 500 °C, 2 h (see below)
Alumina (lower purity)	Chemical or thermal	10 vol. % HF, 20 s; 1 450 °C, 1 h (see below)
Zirconia-toughened alumina	Thermal	1 500 °C, 15 min (see below)
Yttria-TZP	Thermal	1 300 °C, 2 h to 1 420 °C, 15 min (see below)
Ce-TZP	Thermal	1 450 °C, 5 min (see below)
Sialons and sintered silicon nitrides	Plasma etch (see [B.6] and [B.7])	CF_4 plasma etch, 40 s
Hot-pressed silicon nitride	Chemical	NaOH, 400 °C - 450 °C, 1 min –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
* Not all silicon nitrides can be etched effectively by this method, depending on their exact composition.		

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 50 °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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[B.6] Chatfield, C. and Norstrom, H., Plasma etching of sialon. J. Amer. Ceram. Soc., 1983, 64(9), C-168.

[B.7] Täffner, U., Hoffman, M.J., Krämer, M., Comparison of different physical/chemical methods of etching for silicon nitride ceramics. Prakt. Metallog., 1990, 27, 385-90.

Annex C
(informative)

Setting Köhler illumination in an optical microscope

C.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.

C.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.

C.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 stage micrometer, and a low-magnification objective lens, typically x10. 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.