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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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Contents

Page

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

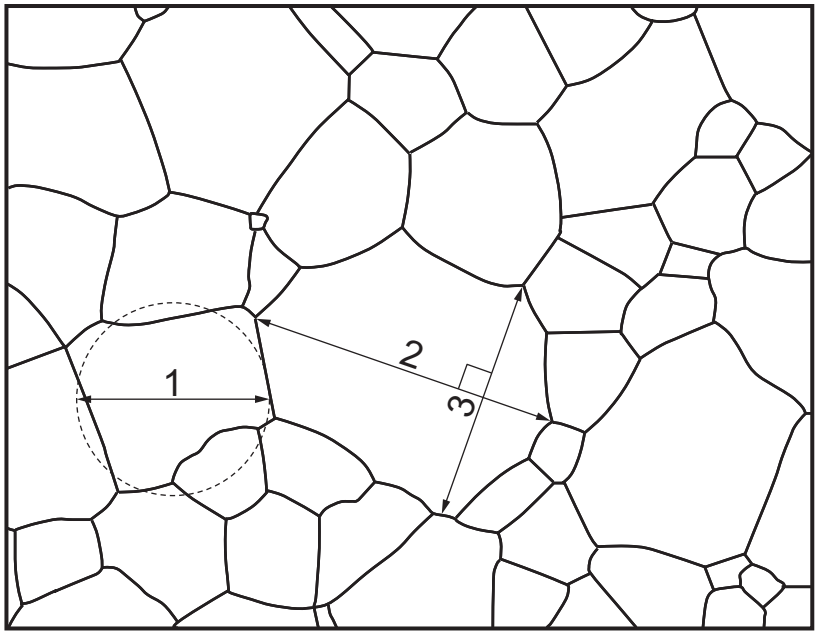
See Figure 1.

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.