



BSI Standards Publication

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

Part 1: Determination of grain size and size distribution (ISO 13383-1:2012)

National foreword

This British Standard is the UK implementation of EN ISO 13383-1:2016. It is identical to ISO 13383-1:2012. It supersedes BS EN 623-3:2001 and BS ISO 13383-1:2012 which are withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics.

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

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English Version

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Determination of grain size and size distribution (ISO 13383-1:2012)

Céramiques techniques - Caractérisation
microstructurale - Partie 1: Détermination de la taille
et de la distribution des grains (ISO 13383-1:2012)

Hochleistungskeramik - Mikrostrukturelle
Charakterisierung - Teil 1: Bestimmung der Korngröße
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CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

European foreword

The text of ISO 13383-1:2012 has been prepared by Technical Committee ISO/TC 206 “Fine ceramics” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 13383-1:2016 by Technical Committee CEN/TC 184 “Advanced technical ceramics” the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by October 2016, and conflicting national standards shall be withdrawn at the latest by October 2016.

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Endorsement notice

The text of ISO 13383-1:2012 has been approved by CEN as EN ISO 13383-1:2016 without any modification.

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Significance and use	3
5 Apparatus	4
5.1 Sectioning equipment	4
5.2 Mounting equipment	4
5.3 Grinding and polishing equipment	4
5.4 Etching equipment	4
5.5 Microscope	4
5.6 Calibrated rule or scale	5
5.7 Circle template	5
6 Test piece preparation	5
6.1 Sampling	5
6.2 Cutting	5
6.3 Mounting	5
6.4 Grinding and polishing	5
6.5 Etching	6
7 Photomicrography	6
7.1 General aspects	6
7.2 Optical microscopy	6
7.3 Scanning electron microscopy	6
7.4 Calibration micrographs	7
8 Measurement of micrographs	7
8.1 General	7
8.2 Method A1	8
8.3 Method A2	8
8.4 Method B	8
8.5 Use of automatic or semi-automatic image analysis for methods A and B	9
9 Calculation of results	10
9.1 Method A1	10
9.2 Method A2	10
9.3 Method B	10
10 Interferences and uncertainties	11
11 Test report	12
Annex A (informative) Grinding and polishing procedures	14
Annex B (informative) Etching procedures	16
Annex C (informative) Setting Köhler illumination in an optical microscope	18
Annex D (informative) Round-robin verification of Method A1	19
Annex E (informative) Round-robin verification of Method B	20
Annex F (informative) Grain size distribution measurement	21
Annex G (informative) Results sheet: Grain size in accordance with ISO 13383-1	22
Bibliography	23

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.

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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

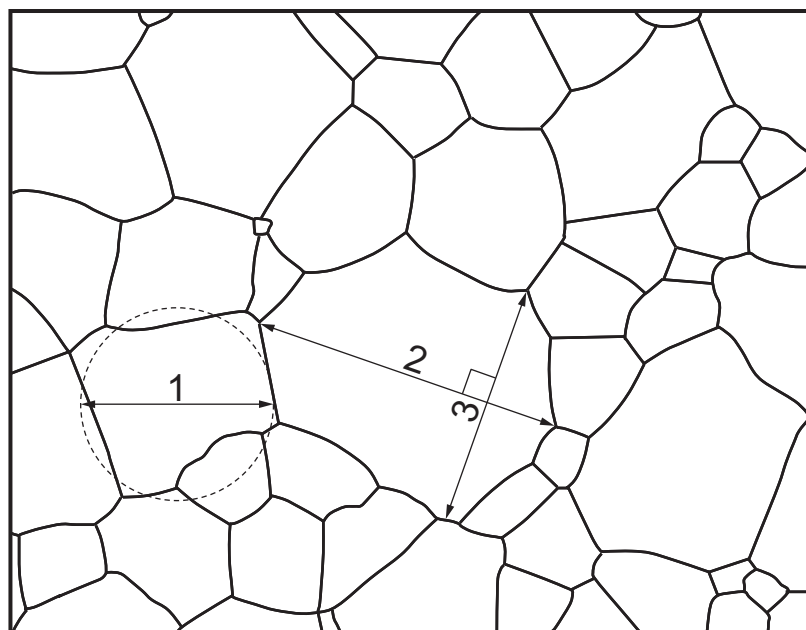
$d_{ci, perp}$

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, $d_{ci, \text{perp}}$

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