INTERNATIONAL STANDARD

ISO 13383-2

First edition
2012-09-01

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

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

Céramiques techniques — Caractérisation microstructurale —

Partie 2: Détermination de la fraction volumique des phases par évaluation de micrographes

Reference number
ISO 13383-2:2012(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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.

ISO 13383-2 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 2: Determination of phase volume fraction by evaluation of micrographs

1 Scope

This part of ISO 13383 specifies a manual method of making measurements for the determination of the volume fraction of major phases in fine ceramics (advanced ceramics, advanced technical ceramics) using micrographs of polished and etched sections, overlaying a square grid of lines, and counting the number of intersections lying over each phase.

NOTE 1 This method assumes that the true phase volume fractions are equivalent to area fractions on a randomly cut cross-section according to stereological principles.

NOTE 2 Guidelines for polishing and etching of advanced technical ceramics can be found in Annexes A and B of ISO 13383-1:2012.

The method applies to ceramics with one or more distinct secondary phases, such as found in Al_2O_3/ZrO_2, Si/SiC, or Al_2O_3/SiC_w.

If the test material contains discrete pores, these are to be treated as a secondary phase for the purpose of this method, provided that there is no evidence of grain pluck-out during polishing being confused with genuine pores.

NOTE 3 If the material contains more than about 20 % porosity, there is a strong risk that the microstructure will be damaged during the polishing process, and measurement of the volume fraction of pores may become misleading. Secondary phase volume fractions or porosity present at levels of less than 0,05 are subject to considerable error and potential scatter in results. A larger number of micrographs than the minimum of three is normally needed to improve the consistency and accuracy of the results.

NOTE 4 Many ceramics contain small amounts of secondary glassy phases. In order to make a reasonable estimate of glassy phase content, the glass material between crystalline grains should be readily observable, and thus should be at least 0,5 µm in width. The method in this part of ISO 13383 is not considered appropriate for narrow glassy films around grains.

NOTE 5 Microstructures are seldom homogeneous, and the phase contents can vary from micrograph to micrograph. It is essential to survey a sufficiently wide area of the prepared section to ensure that those areas selected for evaluation are representative, and do not contain eye-catching irregularities. This method assumes that the selected regions of a prepared cross-section are statistically representative of the whole sampled section.

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 currently outside the scope of this part, but some guidelines are given in Annex A.

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
phase volume fraction
volume occupied by a distinct, identifiable phase present in a material expressed as a fraction of the whole

3.2
primary phase
principal phase within a microstructure, typically comprising more than 50 % by volume or observed area in a cross-section

3.3
secondary phase
one or more distinct identifiable phases other than a primary crystalline phase in a material

NOTE A secondary phase can be in the form of discrete grains, or as a continuous phase surrounding some or all of the major phase grains. For the purposes of this part of ISO 13383, porosity may be treated as a secondary phase.

4 Apparatus

4.1 Sectioning equipment

Any suitable method may be used for preparing the test section from the item under investigation. If a diamond-bladed cut-off saw is employed, it is recommended that the grit size should not exceed 150 µm.

NOTE This grit size is designated as D151 according to ISO 6106 [5].

4.2 Mounting equipment

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

4.3 Grinding and polishing equipment

Suitable grinding and polishing equipment, employing diamond abrasive media.

NOTE A sequence of abrasives and techniques recommended for polishing are given in Annex A of ISO 13383-1:2012.

4.4 Microscope

An optical or scanning electron microscope with photomicrographic facilities.

NOTE Although the true magnification of the image is unimportant for making the measurement of the volume fraction, it is advised that a reference graticule may be used to determine magnification in an optical microscope, or a reference grid or latex spheres may be used for calibration of magnification in a scanning electron microscope, and as a check on the homogeneity of magnification across the field of view.

An optical microscope is additionally required for assessing the quality of polishing (see 5.4).

4.5 Transparent grid

Transparent square grid on, e.g. acetate film, and with line thickness not exceeding 0,1 mm.

NOTE 1 The grid spacing selected is not critical, but may conveniently be between 3 mm and 15 mm to minimize eyestrain. However, it is necessary that consideration of the requirements of 6.3 is taken into account.

NOTE 2 A suitable grid may be prepared as a computer plot with sufficient accuracy of line spacing for the purposes of this part of ISO 13383.

5 Test piece preparation

5.1 Sampling

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

NOTE Guidance on this issue may be found in EN 1006 (see Bibliography [18]). Depending on the objectives of performing the measurement, it is desirable to maintain knowledge of the positions within components or test pieces from which sections are prepared.

5.2 Cutting

The required section of test piece shall be cut using the sectioning equipment (see 4.1).

NOTE For routine inspection of materials, a small area of side no more than 10 mm is normally adequate as the section to be polished.

5.3 Mounting

Mount the test piece using an appropriate mounting medium.

NOTE 1 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 grinding and polishing.

NOTE 2 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 test pieces. The method of mounting selected should take into account the etching procedure to be used; see Annex B.

5.4 Grinding and polishing

Grind and polish the surface of the test piece. Care shall 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 4.4) at high magnification. At least 90 % of the test piece area shall be free from optically visible scratches, or other damage introduced by polishing, which will interfere with the determination. In particular, avoid the plucking out of discrete secondary phases from the surface giving the appearance of pores.

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 polishing are given in Annex A of ISO 13383-1:2012.

5.5 Etching

When a good quality polished surface has been achieved, the test piece shall be etched if necessary to reveal the individual phases. Any suitable technique shall be used, subject to agreement between parties.

NOTE 1 Some general guidelines recommending etching procedures for various commonly available advanced technical ceramics are given in Annex B of ISO 13383-1:2012.

NOTE 2 For optical evaluation, it is usually necessary to etch oxide materials in such a way that the individual phases are distinguished by having different contrast levels. For scanning electron microscope (SEM) evaluation, it may not be necessary to etch if a backscattered electron detector is used which has adequate resolution of net atomic number difference between the phases such that contrast is generated. If a secondary electron detector is used, it will usually be necessary to etch to produce topographic contrast unless the atomic number difference between the phases is large.

6 Photomicrography

6.1 General aspects

If it is found that the average grain size of each phase or the widths of continuous glassy phases between grains is less than 2 µm, prepare the test piece for scanning electron microscopy. For grain sizes between 2 µm and 4 µm, either scanning electron microscopy or optical microscopy are permitted. Otherwise, optical microscopy is adequate.

It is important to achieve sufficient contrast between phases in order to identify individual grains clearly and unambiguously.

6.2 Inspection

Inspect the sampled cross-section in the microscope. If the microstructure appears homogeneous, prepare micrographs from randomly selected areas.

NOTE If inhomogeneity of microstructure is suspected or specific regions of a section need to be investigated, this is permitted but must be reported in the report.

6.3 Number of micrographs

At least three micrographs shall be prepared at a magnification sufficient to identify clearly all the phases to be counted. In addition, at least 100 features in total of any given type shall be present to be counted in the set of micrographs.

NOTE For a nominally homogeneous material, it may be sufficient to use a small number of micrographs analysed with a small grid spacing, but for an inhomogeneous material, results representative of the average for the sampled section can be prepared reliably only by selecting a large number of micrographs of different areas, with less intensive counting from a larger grid.

6.4 Optical microscopy

Set up Köhler illumination in the microscope.

NOTE 1 Guidance on setting Köhler illumination conditions is given in Annex D of ISO 13383-1:2012.

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 randomly selected areas of the test-piece surface, taking into account the apparent homogeneity of the microstructure (see 6.2).

NOTE 2 As a guideline, the average size of discrete phase area to be counted should appear typically at least 3 mm across. If the total number of individual grains of any one phase to be counted in any one set of micrographs is less than one hundred, prepare more micrographs. Micrographs should be typically of a size 100 mm x 75 mm, but may with advantage be enlarged later to aid evaluation.

6.5 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 in 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 6.4) from at least three different randomly selected areas of the test piece, using either secondary electron imaging or backscattered electron imaging.

NOTE 2 Although the contrast between phases can be enhanced using backscattered electron imaging, a noisier image than in secondary electron imaging may result and may render the boundaries between contrasting phases indistinct. It can be helpful to use secondary electron images for counting the phase proportions, but backscattered images to aid identification of each phase.

If the number of grains of the phase to be counted is less than 100 in total over all the micrographs, increase the number of areas photographed. Micrographs shall be typically be of a size 100 mm x 75 mm, but may with advantage be enlarged later to aid evaluation.

NOTE 3 It is possible that the photographic screen in the microscope will 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. For the purposes of this test method, distortions of typically up to 5 % may be acceptable provided that the phases being counted are distributed homogeneously across the entire area of the micrograph.

7 Measurement of micrographs

If desirable, enlarge the photomicrograph to a size suitable for easier observation of the features. Examine the dimensions of the smallest features to be counted. Select a suitable grid spacing and prepare a square grid (see 4.5, 6.3 and Clause 9) such that the grid area covers the entire micrograph. Tape the micrograph to a smooth surface. Overlay the grid such that the entire area of the micrograph is covered by the grid, with no grid intersections immediately over the edges of the micrograph (Figure 1). Count the number of grid intersections n_{ij} of the grid that lie over each phase j. If the grid intersection lies exactly over the boundary between two phases, count this as one-half of a grid intersection for each phase. If porosity is to be estimated, use the same rule for when a grid intersection lies exactly on the edge of a pore. Count the total number of grid intersections over the area of the micrograph. If pores are not being counted, count the number of grid intersections lying over the crystalline or glassy phases in the material.

NOTE It can be helpful in counting to screen with pieces of paper those lines of intersections above and below the one being counted; this reduces eye strain and the risk of miscounting, as shown in Figure 1.

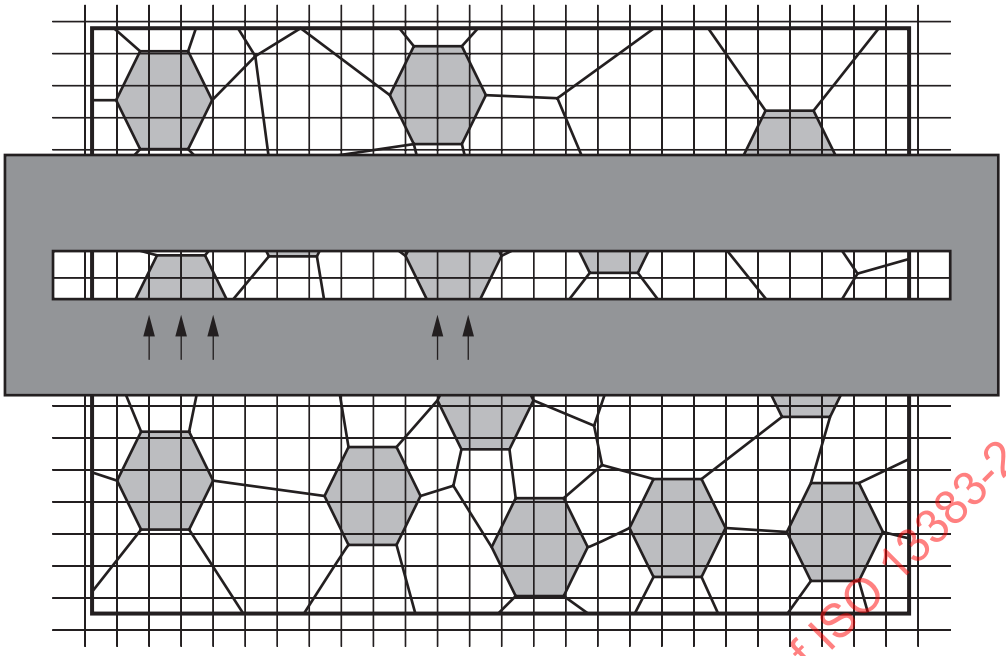

Figure 1 — Schematic diagram of microstructure overlaid with square grid and with optional screen applied. Intersections of the grid with the dark phase are arrowed

8 Calculation of results

For the case where porosity is to be counted as one of the phases, calculate the volume fraction of each phase using Formula (1):

$$V_{fj} = \frac{n_{ij}}{N} \tag{1}$$

where

n_{ij} is the total number of grid intersections lying over phase j;

N is the total number of grid intersections lying over the micrograph.

For the case where porosity is to be ignored, see Formula (2):

$$V_{fj} = \frac{n_{ij}}{\sum n_{ij}} = \frac{n_{ij}}{(N - n_p)} \tag{2}$$

where

n_{ij} is the number of grid intersections lying over solid phase j;

n_p is the number of grid intersections lying over pores;

Σn_{ij} is the sum of all grid intersections lying over all solid phases.

9 Interferences and uncertainties

The nature of the microstructure of the material can affect the result determined in this test. The test is effective when a sufficient number of grid intersections of each phase are counted. This can be achieved

either by intensive analysis of the minimum number of three micrographs, or by less intensive analysis of a larger number of micrographs. For intensive analysis, the grid shall be small enough such that there is a good chance that a grid intersection will lie over each grain. Failure to do this means that the results are subject to increasing possible random error depending on exactly where the grid is positioned. The random error is minimized by adhering to the above guideline, but will always exist because of random positioning of the grid on the micrograph. Typically, for a homogeneous material with randomly distributed phases results from a given series of three micrographs counting at least 100 grains of each type should give phase volume fractions consistent to ± 0,02.

If the material appears inhomogeneous, either more areas should be analysed intensively to establish the extent of the inhomogeneity, or if an average result only is required, a larger grid spacing can be used for less intensive analysis provided that at least 100 grains of each phase type in total are counted. The procedure adopted should be reported.

The counting process requires visual observation of the phase lying underneath each grid intersection. Clean, well-defined phase boundaries are required. If the phase boundaries are poorly defined as a result of limited optical or SEM resolution, it is necessary to adopt a consistent criterion for assessing which side of the true boundary the grid intersection overlies. Failure to do this can lead to under or overestimation of phase volume fraction, and is particularly dangerous for small volume fractions.

The micrographs should not contain features which are ambiguous. Grain pluckout during polishing could inadvertently be treated as porosity and, vice versa, features seen within shallow pores might be counted as solid grains. Particular caution should be taken to avoid subsurface grains giving strong signals in backscattered electron images, or edge highlights in secondary electron images hiding individual grains.

NOTE Annex B contains information from a round-robin activity associated with the development of this part of ISO 13383 which illustrates these concerns.

10 Test report

The report of the test shall be in accordance with the reporting provisions of 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 part of ISO 13383 i.e. ISO 13383-2:2012;

g) the observation technique employed (optical or scanning electron microscope);

h) a summary of the procedure for sampling, cutting, grinding, polishing and etching the test piece;

i) copies of the micrographs used for the measurements;

NOTE 1 It is good practice to provide magnifications on all micrographs, even though not a requirement of this part of ISO 13383.

j) the grid size employed;

k) the number of grid intersections lying over each of the defined phases, including pores if appropriate, on each of the micrographs;

l) the total number of grid intersections on each micrograph;

m) the calculated volume fractions of each phase in each micrograph, and the overall result from all micrographs, expressed as a decimal number to two significant figures;

n) any remarks on the appearance of the microstructure, and difficulties of observing the individual phases;

o) signatures of the persons responsible for the test and authorizing the report;

NOTE 2 For routine presentation of results, it is useful if a standardized format is adopted. A recommended scheme is given in Annex C.

p) any comments on the test or test results, including any necessary deviations from the procedure given in this part of ISO 13383.

Annex A
(informative)

Use of automatic image analysis (AIA)

A.1 Background

There is increasing use of automatic image analysis for estimating phase volume fraction, but there is not yet sufficient experience of analysis procedures to permit a standard to be prepared. The following gives information on possible suitable procedures evaluated during the course of the study reported in Annex B.

A.2 Analysis techniques

Depending on the supplied software, there are a number of techniques that can be programmed into automatic image analyzers, but all rely on digitization of the captured image. The original analogue image in the microscope is usually captured via a video camera or micrograph scanning device and is converted into a series of pixels which contain information on position and brightness/intensity. The series of pixels can be interrogated, for example, to count all those with a given brightness. The number counted, divided by the total number of pixels in the image gives the volume fraction. Alternatively, the image can be sampled in the same way as the manual method by selecting only a small proportion of the total number of pixels representing the intersections of a grid. Other methods suitable when the phase brightness in the image is inconsistent involve a manual stage where boundaries are drawn manually around each of the identified phases, rather than relying on selecting a brightness level or range of levels. It is recommended that allowance is made for apparent grain boundary width, which can with advantage be narrowed electronically to reduce the possible counting error.

A.3 Micrograph requirements

In order to minimize the need for human interaction in the process, it is desirable to acquire micrographs which have uniform brightness across each phase. It is possible that this will not be easy to achieve. In the optical microscope, it is recommended that the illumination of the field of view is very even. The contrast established in the scanning electron microscope should be very uniform with a minimum of edge brightness or etched grain boundary topographic width in secondary electron images, which is best achieved by using low voltages. Alternatively, backscattered electron images eliminate undesirable edge brightness, but there is an increase in noise. As shown by the round robin reported in Annex B, in a good image, the scatter between laboratories is similarly as low as that achieved using the manual method, but when the image contains phases showing wide intensity variations that do not prevent their correct identification by the human eye, the scatter between laboratories using AIA is much wider.

A.4 Calibration

There is currently no means of checking that an AIA system is giving the correct results, except for when black and white images are used (a trivial case). The options for programming within AIA systems should be checked out using such an image. The ability of the system and its user to obtain correct results should be checked by using manually evaluated micrographs. In this way, a check can be made that intensity level variations across grains and at phase boundaries are being correctly assessed.

Annex B
(informative)

Round-robin verification of this procedure

Background research and a round robin developed in conjunction with this part of ISO 13383 were targeted at setting the conditions for measurement using the manual grid method and at evaluating the repeatability of the method amongst a number of participants. The findings can be summarized as follows, see Bibliography [16], [17]:

a) In a manual method simulation using automatic image analysis, it was shown that the volume fractions as determined by counting the fraction of grid intersections lying over each phase converged to consistent values when the grid spacing was approximately the apparent phase grain size in the micrograph. Employing larger grid spacings than this gave results strongly dependent on grid spacing and position over the micrograph;

b) In the round robin set up to operate under the conditions described above, the manual method reveals statistically consistent and reproducible results when applied to single micrographs of a two-phase barium titanate ceramic and an alumina/5 % zirconia ceramic. The standard deviation of the volume fraction results was within ± 2,5 % (expressed as a phase volume fraction) and the expanded uncertainty (k = 2) on mean values from all participants was less than ± 1,0 %;

c) The scatter in the results of the manual method was found to be mainly produced by the random positioning of the grid on a given micrograph, and by the distribution of the phases in each area examined;

d) The determined volume fractions of a microstructure were found also to be dependent on the exposure conditions used for preparing the micrograph. Whether using an SEM or an optical microscope, decisions have to be made concerning the contrast and brightness, the magnification and the representative area of interest;

e) The variation in the apparent volume fraction can be large between individual micrographs of randomly chosen areas. It was established that it is necessary to make measurements on a representative number of micrographs at magnification required to obey the criterion in 1) above before convergence of the average result could be achieved.