BRITISH STANDARD

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Advanced technical ceramics — Monolithic ceramics — General and textural properties

Part 5: Determination of phase volume fraction by evaluation of micrographs

ICS 81.060.30

British Standards

National foreword

This British Standard is the UK implementation of EN 623-5:2009. It supersedes DD ENV 623-5:2002 which is withdrawn.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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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 623-5:2009) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2010, and conflicting national standards shall be withdrawn at the latest by January 2010.

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

This document supersedes ENV 623-5:2002.

EN 623 consists of five parts, under the general title "Advanced technical ceramics - Monolithic ceramics - General and textural properties":

- Part 1: *Determination of the presence of defects by dye penetration*
- Part 2: *Determination of density and porosity*
- Part 3: *Determination of grain size and size distribution (characterized by the Linear Intercept Method)*
- Part 4: *Determination of surface roughness*
- Part 5: *Determination of phase volume fraction by evaluation of micrographs*

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1 Scope

This part of EN 623 specifies a manual method of making measurements for the determination of volume fraction of major phases in 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.

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 can 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 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 European Standard is not considered appropriate for narrow glassy films around grains.

This method assumes that the selected regions of a prepared cross-section are statistically representative of the whole sampled section.

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.

Some users of this European Standard can wish to apply automatic or semiautomatic image analysis to micrographs or directly captured microstructural images. This is currently outside the scope of this European Standard, but some guidelines are given in Annex C.

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.

EN 1006, *Advanced technical ceramics - Monolithic ceramics - Guidance on the selection of test pieces for the evaluation of properties*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

3 Terms and definitions

For the purposes of this part of EN 623, 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
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 the major phase grains. For the purposes of this European Standard, porosity may be treated as a secondary phase.

4 Apparatus

4.1 Sectioning equipment

A suitable diamond-bladed cut-off saw to prepare the initial section for investigation. The saw shall be metal bonded with a diamond grit size of 125 µm to 150 µm and shall be cooled.

NOTE This grit size is designated D151 according to ISO 6106, see [1].

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.

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 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 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 minimise 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 European Standard.

5 Test piece preparation

5.1 Sampling

The test pieces shall be sampled in accordance with the guidelines given in EN 1006, and subject to agreement between parties.

NOTE 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 diamond saw (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. 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 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 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 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, discrete secondary phases may be plucked out from the surface giving the appearance of pores. This shall be avoided.

NOTE Care should be taken in choosing the sequence of grits and lap types. It is impossible within the scope of this part of EN 623 to make specific recommendations for all types of material. The general principle to be adopted is the minimisation of subsurface damage, and its removal by progressively finer grits whilst retaining a flat surface. Some guidelines on polishing are given in Annex A.

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.

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 evaluation by scanning electron microscopy (SEM), 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 suspected that the average grain size of each phase or the widths of continuous glassy phases between grains is less than 2 µm, it will be necessary to prepare the test piece for SEM. Between 2 µm and 4 µm either SEM or optical microscopy may be used. Otherwise, optical microscopy will normally be 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. If inhomogeneity of microstructure is suspected, select representative areas of relevance for measurement.

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 Guidance on setting Köhler illumination conditions is given in Annex D.

Examine the test piece at a magnification sufficient to resolve the individual grains clearly. If the contrast obtained is insufficient, e.g. in white or translucent materials, apply a suitable 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). 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 x 75) mm, but may with advantage be enlarged later to aid evaluation.

6.5 Scanning electron microscopy (SEM)

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 characterised 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 should typically be of a size (100 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 comments in 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. Count the number of grid intersections n_j 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.

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 Equation (1):

$$V f j = \frac{n j}{N} \tag{1}$$

where

n_j is the total number of grid intersections 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 Equation (2):

$$V f j = \frac{n j}{S} = \frac{n j}{(N - n_p)} \tag{2}$$

where

n_j is the number of grid intersections lying over solid phase j;

n_p is the number of grid intersections lying over pores;

S 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 minimised 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 E contains information from a round robin activity associated with the development of this European Standard which illustrates these concerns.

10 Test report

The report of the test shall be in accordance with the reporting provisions of EN ISO/IEC 17025 and shall include at least the following information:

a) name and address of the testing establishment;

b) date of the test;

c) on each page, a unique report identification and page number;

d) customer name and address;

e) reference to this European Standard, i.e. determined in accordance with EN 623-5;

f) authorizing signature;

NOTE 1 For routine presentation of results it is useful if a standardised format is adopted. A recommended scheme is given in Annex F.

g) any deviation from the method described, with appropriate validation, i.e. demonstrated to be acceptable to the parties involved;

h) details of the test piece, including material type, manufacturing code, batch number, etc.;

i) observation technique employed (optical or scanning electron microscope);

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

k) copies of the micrographs used for the measurements;

NOTE 2 It is good practice to provide magnifications on all micrographs, even though not a requirement of this European Standard.

l) grid size employed;

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

n) total number of grid intersections on each micrograph;

o) 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;

p) any remarks on the appearance of the microstructure, and difficulties of observing the individual phases.

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 [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 minimise 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 characterisation measurement. Thus, selection of appropriate polishing procedures, including the sequence of grit sizes, the times of abrasion and the applied pressure are all important. Optimum conditions vary considerably depending on the type of material being prepared. Guidelines on how to choose a grinding method may be found in Hübner and Hausner (see [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 a disc, and may cause more damage. Subsequent grinding steps may need to be of longer duration. The use of a shock-absorbing system, such as a soft metal lap (e.g. tin) into which loose grit becomes lodged, or a metal-plastic composite lap with fixed diamond grit, gives a good balance between speed of abrasion and surface damage.

The grinding of silicon carbide ceramics can cause special difficulties. Klimek (see [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 (see [4]). A series of articles on microstructural preparation of ceramics with polishing details is given in [5].

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

1) 30 µm diamond on a hard composite lap;

2) 6 µm diamond on a softer composite lap;

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

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

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

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

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

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

Bibliographic lists of etching methods have been given by Clinton and Petzow (see [4] and [6]), and further information is given in [7] to [9]. Table B.1 gives examples.

Table B.1 – Examples of etching methods

Ceramic	**Method**	**Conditions**
Alumina (> 99,5 %)	Thermal	1 500 °C, 2 h (see text)
Alumina (lower purity)	Chemical or thermal	10 % HF, 20 s; 1 450 °C, 1 h (see text)
Zirconia-toughened alumina	Thermal	1 500 °C, 15 min (see text)
Yttria-Tetragonal zirconia polycrystals (TZP)	Thermal	1 300 °C, 2 h to 1 420 °C, 15 min (see text)
Ce-TZP	Thermal	1 450 °C, 5 min (see text)
Sialons and sintered silicon nitrides	Plasma etch (see [10] and [11])	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.

Thermal etching used for oxide ceramics can give good clear delineation of grain boundaries, but there is a risk of modifying the microstructure of the product in the process. The maximum temperature for this process should be at least 150 °C below the original firing temperature of the ceramic (for the same time period) to minimise 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 can 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. It is possible that the true grain boundaries will not all be revealed.

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

Annex C
(informative)

Use of automatic image analysis (AIA)

C.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 E.

C.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 digitisation 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.

C.3 Micrograph requirements

In order to minimise 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 E, 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.

C.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 D
(informative)

Setting Köhler illumination in an optical microscope

D.1 Purpose

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

D.2 Definition

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

D.3 Setting up for Köhler illumination

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

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

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

Annex E
(informative)

Round robin verification of this procedure

Background research and a round robin developed in conjunction with this European Standard 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 summarised as follows:

1) 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;

2) 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 95 % confidence on mean values from all participants was less than ± 1,0 %;

3) 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;

4) 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;

5) the variation in 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.

Annex F
(informative)

Results sheet

Testing establishment					
Report reference no.					
Client name and address					
Date of receipt of test piece					
Date of test					
Test piece reference number and manufacturing details					
Size of sampled ceramic					
Number of test pieces					
Test piece preparation	Method of cutting Method of mounting Method of grinding/polishing Method of etching				
Method of observation	Type of microscope Method of recording images Magnification of micrographs and calibration method, if available				
Results from each micrograph	Phase type	(1)	(2)	(3)	Total
Including/excluding pores	No. of intersections over phase				
	Phase volume fraction				
Overall results from all micrographs	Phase type	(1)	(2)	(3)	Total
	No. of intersections over phase				
	Phase volume fraction				
Remarks Problems with preparation Features of microstructure					
Micrographs					
Signature of person undertaking the test					
Authorising signature					

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