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Fine ceramics (advanced ceramics, advanced technical ceramics) — Testing method for macro-heterogeneity in microstructure

Céramiques techniques — Méthode d'essai relative aux macro-hétérogénéités dans la microstructure

Reference number
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Foreword

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The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Testing method for macro-heterogeneity in microstructure

1 Scope

This International Standard specifies a test method for determining macro-heterogeneities within the microstructure of fine ceramics that are above a certain size within the volume of the material and affect materials mechanical strength properties. This method is limited to fine ceramics with a porosity of less than 10 vol% and that are transparent for visible light in the form of a thin specimen.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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
coarse macro-heterogeneity
macro-heterogeneity, e.g. agglomerates, pores, inclusion, grains, with a size above 10 µm significantly affecting the mechanical strength

3.2
coarse macro-heterogeneity distribution
size distribution of coarse macro-heterogeneities

3.3
equivalent circle diameter
diameter of a circle having the same area as the coarse macro-heterogeneity

4 Principle

A variety of ceramics are composed of transparent materials and their macro-heterogeneity is observed in the transmission mode on a thin specimen.

5 Apparatus

5.1 Microscope, enabling the observation in the transmission mode. The microscope shall have a maximum magnification of the objective lens of ×10 and an illumination system with an adjustable aperture. A certified graticule shall be used for the calibration of the magnification.

5.2 Cutting machine, suitable machine capable of cutting the ceramics.

5.3 Grinding machine, suitable machine capable of grinding the ceramics.

5.4 Polishing machine, suitable machine capable of polishing the ceramics.

5.5 Micrometer, capable of measuring the thickness of the polished ceramic section with an accuracy of 2 µm.

5.6 Microscope slide, suitable transparent microscope slide for mounting the test-piece in the microscope.

5.7 Transparent film with circles of various diameter, to determine the equivalent circle diameter of defects.

6 Test specimen

The specimen shall be thin enough for observation in the transmission mode of the microscope. The typical thickness depends on the material and its porosity, and is between 30 µm and 200 µm.

The specimen shall be prepared as follows.

a) Cut a thin slab from the ceramic using a cutting machine. A thickness of the slab of more than 1 mm plus the final thickness is recommended. For a ceramic with thickness of less than 2 mm, this process can be omitted.

b) One face of the slab shall be ground using a grinding machine and polished with a polishing machine by using polishing grains of a sequence of decreasing sizes with the finishing grit size of less than 1 µm.

c) The other face of the slab shall be ground to adjust the thickness of the slab appropriately. Finally, the ground face shall be polished with the polishing machine by using polishing grains of a sequence of decreasing sizes as mentioned above to prepare a thin specimen for observation.

d) Finally, mount the thin specimen on the microscope slide for observation. Transparent thermoplastic resin is recommended for mounting.

e) The thickness of the specimen must be adjusted for adequate transparency before examination. With a specimen of adequate transparency, defects located at any depth in the specimen can be well focused. Scratches on both front and back surfaces are conveniently used to examine the applicability of this International Standard. If they are clearly seen from the opposite side, this International Standard can be applied.

7 Procedure

7.1 Measurement of specimen thickness

The thickness of the specimen shall be measured at the centre of the specimen with a micrometer to the nearest 0,002 mm.

7.2 Observation

Adjust the microscope as follows; set the numerical aperture of the condenser lens in the illumination system 0,1 or less. The objective lens used for observation should have the magnification under ×10.

7.3 Magnification

The minimum sizes of defects subjected for examination is 10 µm equivalent circle diameter. With the numerical aperture of the lens of 0,1, the maximum thicknesses of specimens which can be examined in one micrograph will be 48 µm and 28 µm multiplied by the refractive index of the specimen, for the objective lenses of magnifications ×4 and ×10, respectively.

The position of focus should be at half of the specimen thickness from the top surface. The refractive index of major phase shall be used for a specimen with multiple phases.

If the specimen is highly transparent, a thicker specimen can be used. A series of micrographs shall be taken by changing the position of focus through the depth direction of specimen. The interval in the position of focus shall be less than 48 µm and 28 µm multiplied by the refractive index of the specimen, for the objective lenses of magnifications ×4 and ×10, respectively.

The photographed areas shall not overlap and micrographs should cover at least 100 macro-heterogeneities which shall be analysed.

8 Measurement of macro-heterogeneities

8.1 Determination of specimen volume under examination

The volume, V, under examination is given by the image area, A_i, the thickness, t, of the slab, and the number, n_L, of locations under examination.

$$V = A_\mathrm{i} \times t \times n_\mathrm{L} \tag{1}$$

8.2 Measurement of the size of macro-heterogeneities

The net magnification shall be adjusted to make the defect of the minimum size appear to be over 5 mm equivalent circle diameter on the micrograph. A transparent film with circles of various diameters shall be used to determine the equivalent circle diameter. Determine the size for at least 100 macro-heterogeneities.

9 Analysis

9.1 Maximum and minimum size

Determine the largest and smallest macro-heterogeneity.

9.2 Size interval

At least five size intervals shall be taken between the size of the largest and smallest defects in the analysis. The intervals may not be equal, but at least five macro-heterogeneities should be present in each interval, except in the interval containing the largest macro-heterogeneity.

9.3 Size and population of defect

The size and the population of macro-heterogeneities for each size interval are given by Formulae (2) and (3).

$$d_{i,\mathrm{m}} = (d_{i,\mathrm{l}} + d_{i,\mathrm{u}})/2 \tag{2}$$

where

$d_{i,\mathrm{m}}$ is the mean size of interval i;

$d_{i,\mathrm{l}}$ is the lower limit of the interval i;

$d_{i,\mathrm{u}}$ is the upper limit of the interval i.

$$p_i = n_i/(d_{i,\mathrm{u}} - d_{i,\mathrm{l}})\ V \qquad (3)$$

where

p_i is the population on interval i;

n_i is the number of defects belonging to interval i.

The unit to express population is the number of macro-heterogeneities/mm^3/µm.

9.4 Graph

Plot the logarithm of the population as a function of the size of macro-heterogeneities.

10 Test report

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

a) a reference to this International Standard, i.e. ISO 18550;

b) the name of the specimen;

c) the preparation method of specimen:

   1) machining method;

   2) thickness of specimen;

d) the observation method:

   1) name of microscope;

   2) objective lens (type, magnification, optical system);

   3) numerical aperture in observation;

e) the micrographs [micrographs of specimen and microscale taken at the magnification of observation and the results (e), if required];

f) the measured results for size distribution of coarse non-uniform structure:

   1) size distribution of non-uniform structure (equivalent circle diameter);

   2) volume for observation.

Annex A
(informative)

Refractive indices of solid and liquids

Refractive index (R.I.) varies with the wave length. It decreases with increasing wave length. Materials of crystallographically cubic symmetry have a single value at each wave length. Those of non-cubic crystal structures have multiple values. Average values are given for them in Table A.1. Values at the wave length of 632,8 nm are listed in Table A.1.

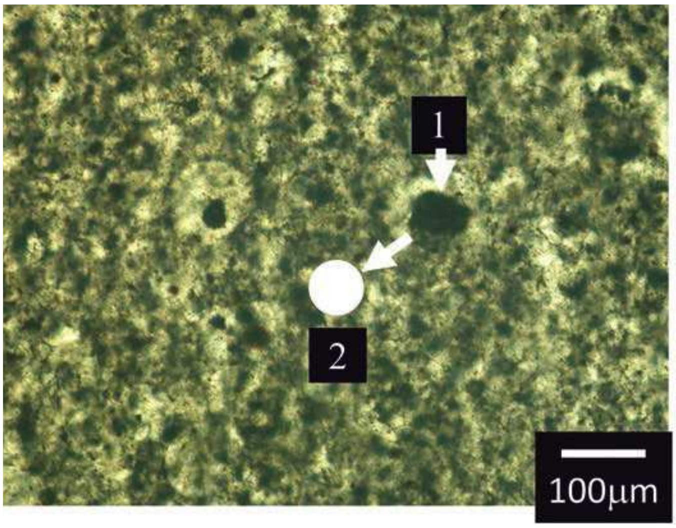
Table A.1 — Refractive indices (R.I.) of various materials at the wave length 632,8 nm

Materials	R.I.
Al_2O_3	1,770
AlN	2,165
MgO	1,735
SiC	2,635
SiO_2	1,457
Si_3N_4	2,023
$BaTiO_3$	2,411
TiO_2	2,874
ZrO_2	2,208
Source: http://www.filmetrics.com/refractive-index-database	

Annex B
(informative)

Examples of macro-heterogeneities observed in transmission mode

Macro-heterogeneities, e.g. agglomerates, pores, inclusion, grains, are found in essentially any ceramic. Figure B.1 shows the structure of alumina ceramics observed with a transmission optical microscope. Dark features of various shapes correspond to heterogeneities in the structure. Figure B.2 shows the population of macro-heterogeneities determined through the analysis of micrographs taken at various locations of the ceramics. A linear relationship is noted in this log-log plot. This relationship was noted in all other systems examined so far.

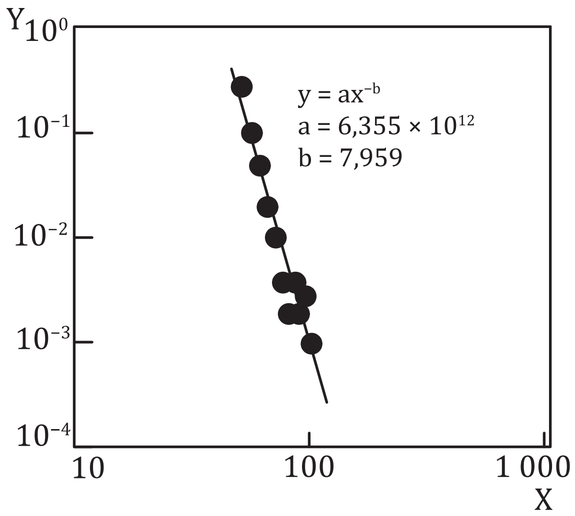


Key

1 flaw

2 equivalent circle, flaw size = equivalent circle diameter

Figure B.1 — Optical micrograph of a ceramic and the determination of equivalent circle diameter of a flaw



Key

X flaw equivalent circle diameter/µm

Y population of flaw/mm^{-3} μm^{-1}

Figure B.2 — Size distribution of macro-heterogeneities in an alumina ceramic

Examples of other macro-heterogeneities are shown in Figures B.3 and B.4 for a silicon nitride ceramic and a zirconia ceramic, respectively.

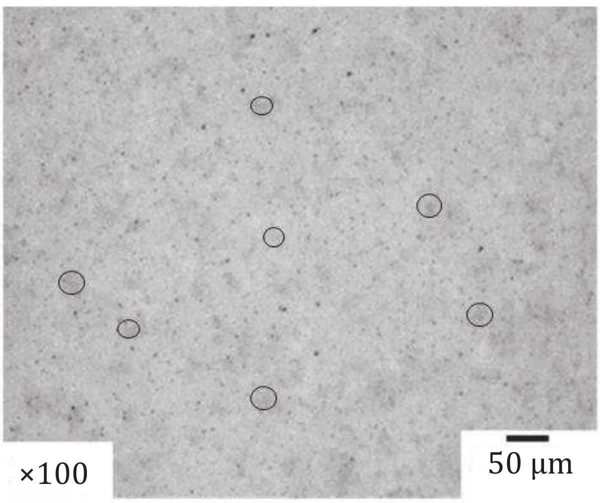


Figure B.3 — Macro-heterogeneities in a silicon nitride ceramic (100 µm thick)

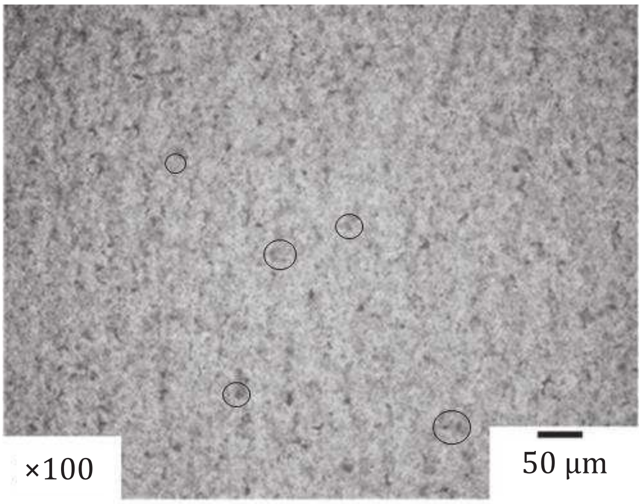


Figure B.4 — Macro-heterogeneities in a zirconia ceramic (100 µm thick)

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