

Fine ceramics (advanced ceramics, advanced technical ceramics) - Microstructural characterization - Part 2: Determination of phase volume fraction by evaluation of micrographs (ISO 13383-2:2012)

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## European foreword

The text of ISO 13383-2:2012 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 13383-2:2016 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of an identical text or by endorsement, at the latest by October 2016, and conflicting national standards shall be withdrawn at the latest by October 2016.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

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# 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  $\text{Al}_2\text{O}_3/\text{ZrO}_2$ ,  $\text{Si}/\text{SiC}$ , or  $\text{Al}_2\text{O}_3/\text{SiC}_w$ .

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  $\mu\text{m}$  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*