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Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size

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National foreword

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

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Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size

*Analyse par microfaisceaux — Diffraction d'électrons rétrodiffusés —
Mesurage de la taille moyenne des grains*



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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.

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ISO 13067 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*.

Introduction

The mechanical and electromagnetic properties of engineering materials are strongly influenced by their crystal grain size and distribution . For example , strength , toughness and hardness are all important engineering properties that are strongly influenced by these parameters . Both bulk materials and thin films , even as narrow two dimensional structures , are influenced by grain size . For this reason , it is important to have standard methods for its measurement with commonly used and agreed terminology . This International Standard describes procedures for measuring average grain size from maps of local orientation measurements using electron backscatter diffraction .

Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size

IMPORTANT — The electronic file of this document contains colours which are considered to be useful for the correct understanding of the document. Users should therefore consider printing this document using a colour printer.

1 Scope

This International Standard describes procedures for measuring average grain size derived from a two-dimensional polished cross section using electron backscatter diffraction (EBSD). This requires the measurement of orientation, misorientation and pattern quality factor as a function of position in the crystalline specimen ^[1].

NOTE 1 While conventional methods for grain size determination using optical microscopy are well established, EBSD methods offer a number of advantages over these techniques, including increased spatial resolution and quantitative description of the orientation of the grains.

NOTE 2 The method also lends itself to the measurement of the grain size of complex materials, for example those with a significant duplex content.

NOTE 3 The reader is warned to interpret the results with care when attempting to investigate specimens with high levels of deformation.

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 16700, *Microbeam analysis — Scanning electron microscopy — Guidelines for calibrating image magnification*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 21748, *Guidance for the use of repeatability, reproducibility and trueness estimates in measurement uncertainty estimation*

ISO 23833, *Microbeam analysis — Electron probe microanalysis (EPMA) — Vocabulary*

ISO 24173:2009, *Microbeam analysis — Guidelines for orientation measurement using electron backscatter diffraction*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply. The reader is also referred to ISO 24173 and ISO 23833 for additional terms and definitions.

3.1 Terminology associated with EBSD measurement of grain size

3.1.1 step size

distance between adjacent points from which individual EBSD patterns are acquired during collection of data for an EBSD map

3.1.2

pixel

picture element

smallest area of an EBSDmap, with the dimensions of the step size, to which is assigned the result of a single orientation measurement made by stopping the beam at a point at the centre of that area

3.1.3

orientation

mathematical description of the angular relationship between the crystal axes of the analysis point and a reference frame, usually the specimen axes

3.1.4

indexed

a pixel is said to be indexed if the orientation calculated from the EBSDpattern acquired for that pixel meets a predetermined threshold for reliability

3.1.5

indexing reliability

numerical value that indicates the confidence/reliability that the indexing software places in an automatic analysis

NOT E This parameter varies between EBSDmanufacturers, but can include:

- a) the average difference between the experimentally determined angles between diffracting planes and those angles calculated for the orientation determined by EBSDsoftware;
- b) the difference between the number of triplets (intersections of three Kikuchi bands) in the EBSDpattern matched by the chosen orientation and the next best possible solution, divided by the total number of triplets.

3.1.6

orientation map

crystal orientation map

map like display of pixels derived from the sequential measurement of crystal orientation at each point in a grid (see Figures 1b) to 1f)) showing the crystallographic relationship between the pixels and the reference frame

3.1.7

pattern quality

measure of the sharpness of the diffraction bands or the range of contrast within a diffraction pattern

NOT E Different terms are used in different commercial software packages, including, for example, band contrast, band slope and image quality.

3.1.8

pattern quality map

map like display of pixels derived from the sequential collection of EBSDpatterns at each point in a grid (see Figure 1a)) showing the pattern quality of the individual pixels

NOT E 1 Since measures of pattern quality can change at features such as grain boundaries and with orientation, the pattern quality map can give an indication of grain shape and size.

NOT E 2 Pattern quality maps can also indicate areas of heavy deformation and inadequate preparation, such as residual scratches.

NOT E 3 Small particles and features also contribute to the pattern quality map.

3.1.9

pseudosymmetry

potential for an EBSDpattern to be indexed in several different ways due to internal similarities within the EBSD pattern

NOT E 1 Pseudosymmetry is a problem with some crystal orientations, usually when a main zone axis is in the centre of the pattern. Typical cases are a {0001} pole for a hexagonal structure and a $\langle 111 \rangle$ pole for a cubic structure.