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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

Reference number
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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

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Foreword

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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.

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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.

INTERNATIONAL STANDARD ISO 13067:2011(E)

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 EBSD map, 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 EBSD pattern 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

NOTE This parameter varies between EBSD manufacturers, but can include:

a) the average difference between the experimentally determined angles between diffracting planes and those angles calculated for the orientation determined by EBSD software;

b) the difference between the number of triplets (intersections of three Kikuchi bands) in the EBSD pattern 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 1 b) to 1 f)] 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

NOTE 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 EBSD patterns at each point in a grid [see Figure 1 a)] showing the pattern quality of the individual pixels

NOTE 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.

NOTE 2 Pattern quality maps can also indicate areas of heavy deformation and inadequate preparation, such as residual scratches.

NOTE 3 Small particles and features also contribute to the pattern quality map.

3.1.9

pseudosymmetry

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

NOTE 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 <111> pole for a cubic structure.

NOTE 2 Structures such as high-symmetry tetragonal crystals with an axial ratio, c/a, approximately equal to 1 are also likely to exhibit pseudosymmetry in EBSD patterns.

3.1.10
misorientation
given two crystal orientations, the misorientation is the rotation, often defined by an angle/axis pair, required to rotate one set of crystal axes into coincidence with the other set of crystal axes

3.1.11
disorientation
due to crystal symmetry, there can be several axis/angle pairs which represent the same misorientation, in which case the one having the smallest angle is called the disorientation

NOTE 1 For most crystal symmetries, there are multiple symmetrically equivalent axes for the disorientation with the smallest misorientation angle.

NOTE 2 Misorientation and disorientation are terms which are often used interchangeably. Disorientation is the more rigorous term here, but misorientation is the more frequently used.

3.1.12
forescatter imaging
orientation contrast produced from electrons which channel out of the specimen

3.1.13
electron-channelling contrast imaging
ECCI
orientation contrast produced from electrons which channel into the specimen

3.1.14
barrel distortion
difference in lateral magnification between the central and peripheral areas of an image such that the lateral magnification is less at the periphery

NOTE A square object in the centre of the field appears barrel-shaped (i.e. with convex sides).

3.1.15
pincushion distortion
difference in lateral magnification between the central and peripheral areas of an image such that the lateral magnification is greater at the periphery

NOTE A square object in the centre of the field appears cushion-shaped (i.e. with concave edges).

3.2 Terminology associated with grains and grain boundaries determined via EBSD

3.2.1
grain boundary
line separating adjacent regions of points in an EBSD orientation map with disorientation across the line greater than a minimum angle chosen to define the grain boundaries

3.2.2
grain
region of points with similar orientation (within a tolerance), completely enclosed by grain boundaries and greater than the minimum size defined to exclude isolated (often badly indexed) points as small grains

3.2.3
sub-grain boundary
line separating adjacent regions of points in a grain with a difference in orientation across the line smaller than that defining a grain but greater than that defining a sub-grain

NOTE Effectively, sub-grain boundaries are grain boundaries with a smaller misorientation limit than that defining a grain boundary. These boundaries can have a characteristic linear appearance and exhibit a characteristic misorientation.

3.2.4

sub-grain

region of points with similar orientation completely enclosed by boundaries greater than the minimum sub-grain boundary angle

3.2.5

special boundary

boundary between two grains having a special orientation relationship within a tolerance associated with identifying them in orientation maps

3.2.6

twin boundary

particular case of a special boundary between crystals oriented with respect to one another according to some symmetry rule, in which the boundary itself is planar and is a characteristic crystallographic plane (for both crystals) and, frequently, one crystal is the mirror image of the other

NOTE For example, in face-centred-cubic structures, the characteristic misorientation defining a common twin can be described as a 60° rotation about the <111> axis with the boundary plane normal to the rotation axis.

3.2.7

recrystallized grains

new set of undeformed grains formed by consuming deformed grains through nucleation and growth processes

NOTE Measurements of misorientation within grains by EBSD can be used to distinguish between deformed and undeformed grains.

3.2.8

phase

physically homogeneous volume in a material having the same crystal structure and chemical composition

3.3 Terminology associated within grain size measurement

There are a variety of ways of representing average grain size. This subclause outlines some of the more common terms used, and the reader is referred to Annex A for more details about other terms, about the standards available and about the applicability of methods for particular grain shapes and distributions.

3.3.1

line intercept

distance between the points at which a straight line crossing a grain intersects the grain boundary on each side

NOTE See ASTM E112 for more details.

3.3.2

equivalent circle diameter

D_{circle}

diameter of the circle with an area equivalent to the grain section area, given by:

$$D_{\text{circle}} = (4A/\pi)^{1/2}$$

where A is the area of the grain.

NOTE The ASTM grain size number, G, is given by:

$$G = -6{,}64\log_{10}D_{\text{circle}} - 2{,}95$$

where D_{circle} is measured in millimetres.

3.3.3
Feret diameter
perpendicular distance between two parallel lines drawn in a given direction tangential to the perimeter of an object on opposite sides of the object

NOTE 1 It is also known as the calliper diameter.

NOTE 2 Different variants of the Feret diameter are used. For example, the Feret diameter can be measured in the vertical and horizontal directions or in any two directions at right angles to each other.

3.3.4
grain shape
property whose value is determined by fitting an ellipse round the grain and measuring the aspect ratio, i.e. the ratio of the length of the minor axis to the length of the major axis

NOTE 1 It is sometimes referred to as grain elongation.

NOTE 2 The value lies in the range 0 to 1.

NOTE 3 There are several ways of fitting the ellipse round the grain, and different methods can result in small differences in the measured aspect ratio.

3.3.5
grain shape orientation
angle between the major axis of an ellipse fitted round the grain and the horizontal direction, usually measured counterclockwise

3.4 Terminology associated with data correction and uncertainty of EBSD maps

3.4.1
misindexing
assigning an incorrect orientation or phase to the measured EBSD pattern

NOTE This can occur for a number of reasons, e.g. pseudosymmetry effects, attempting to index a poor pattern or attempting to index a pattern from an unanticipated phase for which the indexing software is not configured.

3.4.2
non-indexing
non-assignment of an orientation due to insufficient quality of the EBSD pattern

NOTE This can occur for a variety of reasons, such as roughness of the specimen, dust on the specimen, overlapping patterns at the grain boundary, a poor-quality pattern due to the effects of strain, or if the pattern is from an unanticipated phase.

3.4.3
data cleaning
process chosen to accommodate non-indexed and misindexed data within the map, using a given set of parameters, typically based on the characteristics (orientation, phase) of a certain number of nearest neighbours [see Figures 1 b) to 1 f)]

NOTE A wide range of terms (not necessarily mathematically precise) is used in the various commercially available software packages for different data-cleaning operations, including noise reduction, extrapolation, dilation and erosion.

4 Acquiring a map by EBSD for grain size measurement

4.1 Hardware requirements

The reader is referred to ISO 24173 for equipment needed to acquire electron backscatter patterns, index the patterns (determine the orientation) and either step the beam across the specimen surface or, less commonly, step the stage, keeping the beam stationary to acquire a map.