INTERNATIONAL STANDARD

ISO 13067

Second edition
2020-07

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
ISO 13067:2020(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 13067:2011), which has been technically revised. The main changes compared to the previous edition are as follows:

— Data from a round robin (Annex B) have been used to:
  — Include information on expected precision (Clause 7 and Annex B);
  — Include more detail on sources of errors (Clause 7);
  — Clarify statements on minimum numbers of grains measured (5.8) and acceptable clean up procedures (6.3–6.3);
  — Clarify the distinction between sectional grain size measured on a 2D section and average grain size determined from some 2D measurements of grain sections which can be related by stereology to the 3D grain size;
  — Additionally, improvements have been made to the description of calculation of average values (6.5) and representation of the data (6.6).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 document 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

1 Scope

This document 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]. The measurements in this document are made on two dimensional sections. The reader should note carefully the definitions used (3.3) which draw a distinction between the measured sectional grain sizes, and the mean grain size which can be derived from them that relates to the three dimensional grain size.

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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 23833, *Microbeam analysis — Electron probe microanalysis (EPMA) — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 24173 and ISO 23833 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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* (3.1.1), to which is assigned the result of a single *orientation* (3.1.3) 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

[SOURCE: ISO 24173:2009, 3.16, modified to include different reference frames.]

3.1.4
indexed
meets the predetermined threshold for reliability for the *orientation* (3.1.3) of a *pixel* (3.1.2) calculated from the EBSD pattern acquired for that pixel

3.1.5
indexing reliability
numerical value that indicates the confidence/reliability that the indexing software places in an automatic analysis

Note 1 to entry: 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* (3.1.2) derived from the sequential measurement of crystal *orientation* (3.1.3) at each point in a grid [see Figures 1 b) to 1 f)] showing the crystallographic relationship between the pixels and the reference frame

[SOURCE: ISO 24173:2009, 3.17, modified to include reference to examples.]

3.1.7
pattern quality
measure of the sharpness of the diffraction bands or the range of contrast within a diffraction pattern

Note 1 to entry: 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* (3.1.2) derived from the sequential collection of EBSD patterns at each point in a grid [see Figure 1 a)] showing the *pattern quality* (3.1.7) of the individual pixels

Note 1 to entry: 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 to entry: Pattern quality maps can also indicate areas of heavy deformation and inadequate preparation, such as residual scratches.

Note 3 to entry: 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 to entry: 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 to entry: Structures such as high-symmetry tetragonal crystals with an axial ratio, c/a, αpproximately equal to 1 are also likely to exhibit pseudosymmetry in EBSD patterns.

[SOURCE: ISO 24173:2009, 3.22]

3.1.10
misorientation
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, given two crystal o*rientations* (3.1.3)

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 to entry: For most crystal symmetries, there are multiple symmetrically equivalent axes for the disorientation with the smallest misorientation angle.

Note 2 to entry: 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

Note 1 to entry: Other contrast mechanisms such as composition can also affect the contrast obtained.

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

[SOURCE: ISO 10934-1:2002, 2.4.5.1]

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

[SOURCE: ISO 10934-1:2002, 2.4.5.2]

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* (3.1.11) across the line greater than a minimum angle chosen to define the grain boundaries

3.2.2
grain
region of points with similar *orientation* (3.1.3) (within a tolerance), completely enclosed by *grain boundaries* (3.2.1) and greater than the minimum size defined to exclude isolated (often badly *indexed* (3.1.4)) points as small grains

3.2.3
sub-grain boundary
line separating adjacent regions of points in a *grain* (3.2.2) with a difference in *orientation* (3.1.2) across the line smaller than that defining a *grain* (3.2.2) but greater than that defining a *sub-grain* (3.2.4)

Note 1 to entry: 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* (3.2.3) angle

3.2.5
special boundary
boundary between two *grains* (3.2.2) having a special *orientation* (3.1.3) relationship within a tolerance associated with identifying them in *orientation maps* (3.1.6)

3.2.6
twin boundary
particular case of a *special boundary* (3.2.5) 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 1 to entry: 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* (3.2.2) formed by consuming deformed grains through nucleation and growth processes

Note 1 to entry: 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
3D grain size
three-dimensional size of a *grain* (3.2.2) or crystal within a polycrystalline material, measured as a volume

Note 1 to entry: In a strict stereological definition, just the term grain size is sufficient to denote this value, but it is recommended to use the full description 3D grain size to avoid confusion with the *sectional grain size* (3.3.8) which is often shortened to grain size as well.

3.3.2
average grain size
value determined from a two dimensional measurement which is related to the average three dimensional size of a collection of grains or crystals forming a polycrystalline material by stereological relationships[2]. It can be reported as one or more of the following measurements:

a) average area

b) average diameter determined from average area

c) average linear intercept length

3.3.3
line intercept
distance between the points at which a straight line crossing a grain intersects the *grain boundary* (3.2.1) on each side

Note 1 to entry: See ASTM E112[15] for more details.

3.3.4
equivalent circle diameter
D_{circle}
diameter of the circle with an area equivalent to the grain section area, given by:

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

where A is the area of the *grain* (3.2.2)

Note 1 to entry: The ASTM grain size number, G, is given by[15]:

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

where D_{circle} is measured in mm.