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Microbeam analysis — Guidelines for orientation measurement using electron backscatter diffraction

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Foreword

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

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This second edition cancels and replaces the first edition (ISO 24173:2009) which has been technically revised.

The main changes are as follows:

~~— "the electron backscatter pattern" is changed to "the backscatter electron signal". (see 3.6)~~
~~— "Note 2 to entry: See Annex A for more information" is added. (see 3.7)~~
~~— "in the alignment of the coordinate systems of two crystals" is changed to "in the orientation of two crystallites".(see 3.19)~~
~~— "terms of local misorientation (LM), kernel average misorientation (KAM), local average misorientation (LAM), transmission kikuchi diffraction (TKD) are added. (see 3.20, 3.21, 3.22, 3.23)~~
~~— "the electron diffraction pattern" is changed to "the electron signal". (see 3.25)~~
~~— "between the plane of the detector screen" is changed to "between the pattern centre in the detector screen" (see 3.27)~~
~~— "in figure 5, a note is added. (see 3.28)~~
~~— "must be" is changed to "shall be". (see 5.1)~~

~~— "~~— Clause 3 has been updated;

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— "in the working position" is changed to "in the detector position"." (see 6.6 (d)));

— the section of subclause "7.1 prePre-test preparation" in the previous edition is omitted.;

— "— "Annex- B (normative)" is changed to "Annex- B (informative)".)";

— "may" is changed to "can". (see B.2 and B.3)

— changes have been made to align this document with ISO rules.

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.htmlwww.iso.org/members.html.

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Introduction

Electron backscatter diffraction (EBSD) is a technique that is used with a scanning electron microscope (SEM), a combined SEM-FIB (focussed-ion beam) microscope or an electron probe microanalyser (EPMA) to measure and map local crystallography in crystalline specimens[1],[2].

Electron backscatter patterns (EBSPs) are formed when a stationary electron beam strikes the surface of a steeply inclined specimen, which is usually tilted at ≈ 70° from normal to the electron beam. EBSPs are imaged via an EBSD detector, which comprises a scintillator (such as a phosphor screen or a YAG single crystal) and a low-light-level camera (normally a charge-coupled device, CCD). Patterns are occasionally imaged directly on photographic film.

By analysing the EBSPs, it is possible to measure the orientation of the crystal lattice and, in some cases, to also identify the phase of the small volume of crystal under the electron beam. EBSD is a surface diffraction effect where the signal arises from a depth of just a few tens of nanometres, so careful specimen preparation is essential for successful application of the technique[3].

In a conventional SEM with a tungsten filament, a spatial resolution of about 0,25 μm can be achieved; however, with a field-emission gun SEM (FEG-SEM), the resolution limit is 10 nm to 50 nm, although the value is strongly dependent on both the material being examined and the instrument operating parameters. A new method termed as transmission Kikuchi diffraction (TKD)[4] or transmission EBSD (t-EBSD)[5] in SEM has been proved to improve spatial resolutions better than 10 nm and is suited for routine EBSD characterization of both nano-structured and highly deformed samples.

Orientation measurements in test specimens can be carried out with an accuracy of ≈ 0,5°. By scanning the electron beam over a region of the specimen surface whilst simultaneously acquiring and analysing EBSPs, it is possible to produce maps that show the spatial variation of orientation, phase, EBSP quality and other related measures. These data can be used for quantitative microstructural analysis to measure, for example, the average grain size (and in some cases the size distribution), the crystallographic texture (distribution of orientations) or the amount of boundaries with special characteristics (e.g. twin boundaries). EBSD can provide three-dimensional microstructural characterization by combining with an accurate serial sectioning technique, such as focussed-ion beam milling[6].

It is strongly recommended that EBSD users should be well acquainted with both the principles of crystallography and the various methods for representing orientations (both of which are described in the existing literature in this field) in order to make best use of the EBSD technique and the data[7],[8].

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Microbeam analysis — Guidelines for orientation measurement using electron backscatter diffraction

1 Scope

This document gives guidance on how to generate reliable and reproducible crystallographic orientation measurements using electron backscatter diffraction (EBSD). It addresses the requirements for specimen preparation, instrument configuration, instrument calibration and data acquisition.

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

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

— IEC Electropedia: available at ~~https://www.electropedia.org/~~https://www.electropedia.org/

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3.1
crystal
entity consisting of a regular, repeated arrangement of atoms in space and usually described by a space group, a crystal system, unit cell parameters (including the lengths and angles between the unit cell axes) and the positions of the atoms inside the unit cell[9],[10]

Note 1 to entry: For example, an aluminium crystal can be represented by a cube (unit cell) of length 0,404 94 nm along each edge and with atoms at the corners and centres of the cube faces.

Note 2 to entry: Simulations of the atomic arrangement in a small (4 × 4 × 4 unit cells) aluminium crystal, as viewed along the [1 0 0], [1 1 1] and [1 1 0] directions, are shown in Figure 1, together with the associated spherical Kikuchi patterns for each crystal orientation. The 4-fold, 3-fold and 2-fold crystal symmetries are easily seen, as are the mirror planes.

Note 3 to entry: For those unfamiliar with crystallography, it is recommended that a standard textbook be consulted (see for example References [9], [10] and [11]).

Note 4 to entry: Annex C contains a brief introduction to crystallography and a guide to the indexing of EBSPs for materials with cubic crystal symmetry.

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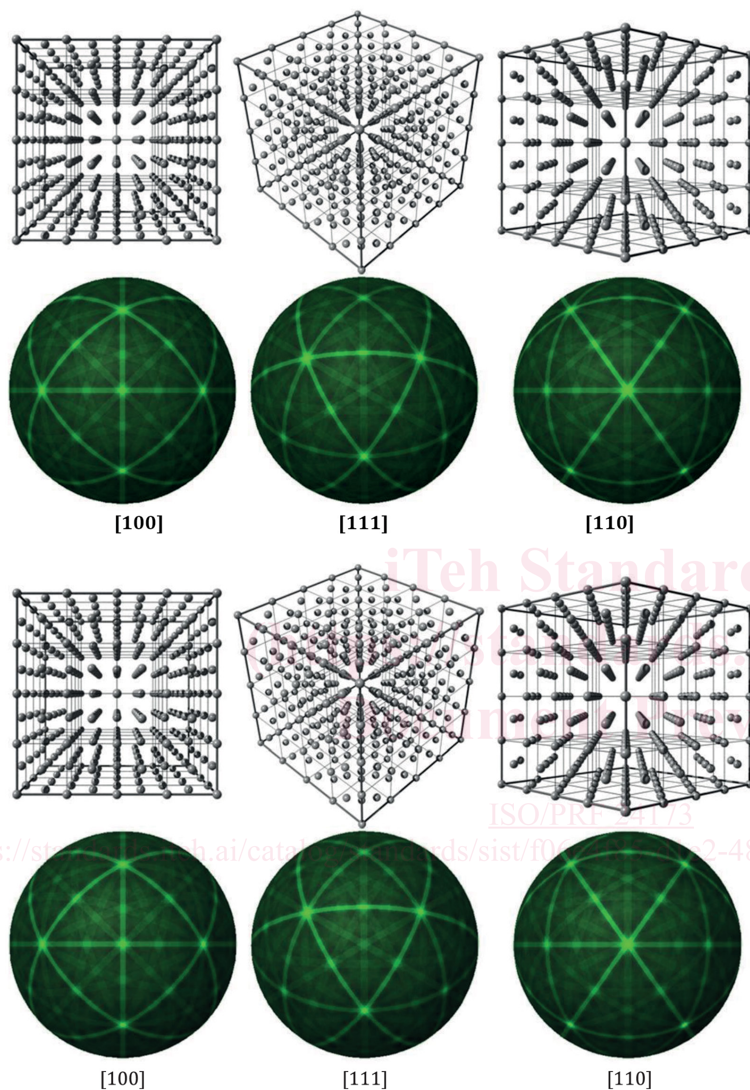

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Figure 1 — Simulations of a small aluminium crystal (top) as viewed along the [1 0 0], [1 1 1] and [1 1 0] directions, with their associated spherical Kikuchi patterns (bottom). The symmetry is clearly shown.

3.2
crystal plane

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plane, usually denoted as (*h k l*), representing the intersection of a plane with the a-, b- and c-axes of the unit cell at distances of 1/*h*, 1/*k* and 1/*l*, where *h*, *k*, and *l* are the minimum mutual integers

Note 1 to entry: The integers *h*, *k*, and *l* are usually referred to as the Miller indices of a crystal plane.

Note 2 to entry: See Annex C for more information.

3.3
crystal direction
direction, usually denoted as [***u v w***], representing a vector direction in multiples of the basis vectors describing the *a*, *b* and *c* crystal axes

Note 1 to entry: See Annex C for more information.

3.4
~~**crystal unit cell**~~
~~cell which is the smallest repeating unit to build up the crystal~~

~~Note 1 to entry: It is usually defined by three lengths, *a*, *b* and *c*, and three angles, *α*, *β* and *γ*. The lengths are usually given in ångströms or nanometres and the angles in degrees.~~

~~**3.5**~~
crystallographic orientation
alignment of the crystal coordinate system (for example, [1 0 0], [0 1 0], [0 0 1] for a cubic crystal) in relation to the specimen coordinate system

Note 1 to entry: The specimen coordinate system can be denoted as X, Y, Z. When EBSD is applied to the study of rolled materials, it is often denoted as RD, TD, ND [RD = reference (or rolling) direction, TD = transverse direction and ND = normal direction].

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3.~~6~~5
EBSD detector
detector used to capture the backscatter electron signal and convert it to an visible image on the display device (computer screen) via a video-camera, which is commonly a high-sensitivity charged-coupled device (CCD) ,or complementary metal-oxide-semiconductors (CMOS)

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~~Note 1 to entry: See also 3.21.~~

~~**3.7**~~
3.6
electron backscatter diffraction
EBSD
diffraction process that arises between the backscattered electrons and the atomic planes of a highly tilted crystalline specimen when illuminated by a stationary incident electron beam

Note 1 to entry: Commonly used alternative terms for EBSD are “EBSP” (or more usually the “EBSP technique~~”) (see 3.8),~~”), “BKD” (backscattered Kikuchi diffraction), “BKED” (backscattered Kikuchi electron diffraction) and “BKDP” (backscattered Kikuchi diffraction pattern).

Note 2 to entry: See Annex A for more information.

3.~~8~~7
electron backscatter pattern
EBSP

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intersecting array of quasi-linear features, known as Kikuchi bands (see Figure 2), produced by electron backscatter diffraction and recorded using a suitable detector, for example observed on a phosphorescent screen or, less commonly, on photographic film

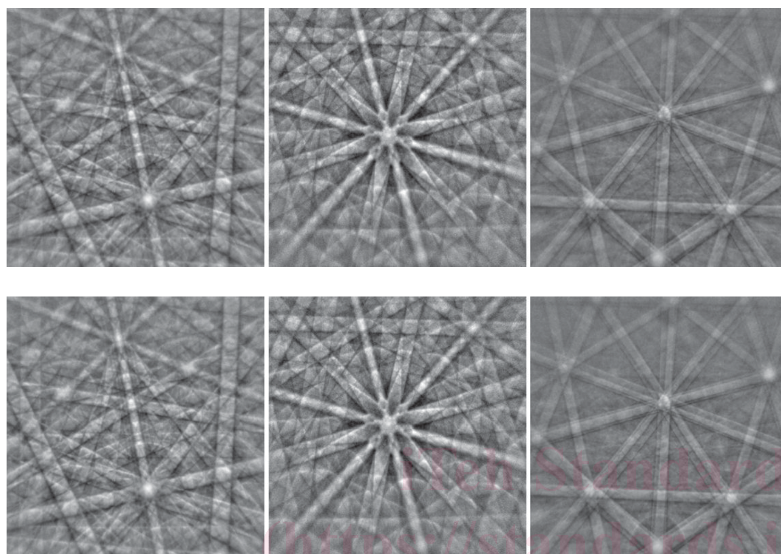

Figure 2 — Examples of EBSPs showing arrays of overlapping Kikuchi bands

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3.98
pattern centre
PC
point in the plane of the detector screen on a line normal to the plane of the screen and passing through the point where the electron beam strikes the specimen

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3.109
EBSD grain
region, with similar orientation, delineated by boundaries at which the misorientation between neighbouring measurement points is greater than a defined critical value which depends on the application[12].

3.11
EBSD spatial resolution
minimum distance between two points in different grains (separated by a sharp boundary) that produces two distinctly different *EBSPs* (3.8) that can be correctly indexed using an EBSD system

Note 1 to entry: An example is shown in Figure 3 where the electron beam has been passed over a boundary in a meteorite specimen. Two distinct and different EBSP orientations can be seen in the far-left and far-right images, but the central *EBSP* (3.8) is a mixture of the two. Modern indexing algorithms frequently allow solution of such overlapping patterns, which leads to an effective improvement in the EBSD spatial resolution.

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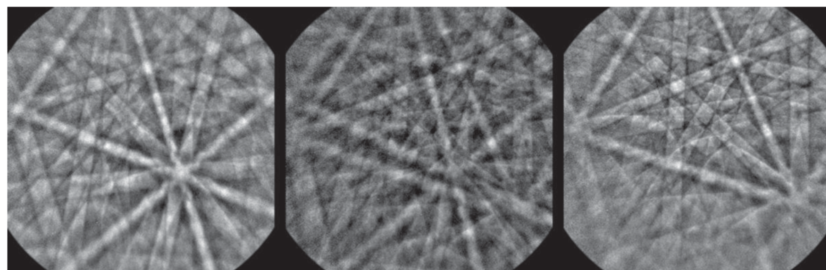

NOTE These images were taken at 30 nm spacings and the centre *EBSP* (3.8) is a combination of the other two.

Figure 3 — Examples of *EBSPs* (3.8) from either side (far left and far right) and on a grain boundary (centre)

3.12

Euler angles

set of three rotations for representing the orientation of a crystal relative to a set of specimen axes

Note 1 to entry: The Bunge convention (rotations about the Z, X'and Z"directions) is most commonly used for describing EBSD data. The Euler angles give the rotation needed to bring the specimen coordinate system into coincidence with the crystal coordinate system. It should be noted that there are equivalent sets of Euler angles, depending on crystal symmetry[8].

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3.1310

Hough transform

mathematical technique of image processing which allows the automated detection of features of a particular shape within an image

Note 1 to entry: In EBSD, a linear Hough transform is used to identify the position and orientation of the Kikuchi bands in each *EBSP* (3.8), which enables the EBSP to be indexed. Each Kikuchi band is identified as a maximum in Hough space. The Hough transform is essentially a special case of the Radon transform. Generally, the Hough transform is for binary images, and the Radon transform is for grey-level images.[13],[14]. See 5.3.7 for more details.

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3.1411

indexing

process of identifying the crystallographic orientation corresponding to the features in a given EBSP, for example, determining which crystal planes correspond to the detected Kikuchi bands or which crystal directions match the Kikuchi band intersections (zone axes) and thereby determining the orientation (and phase)

3.1512

microtexture

population of crystallographic orientations whose individual components are linked to their spatial location within the microstructure[15]

3.16

orientation

alignment of a crystal axes relative to a set of specimen axes

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Note 1 to entry: It is usually represented by Euler angles ~~(ϕ_1~~(ϕ_1, Φ, ~~ϕ_2~~ϕ_2) or a 3 × 3 orientation matrix of direction cosines between the crystal and specimen axes and/or a Rodrigues-Frank vector.

3.~~17~~13
orientation map
OM
map-like display of crystal orientation data derived from the sequential measurement of the crystal orientation at each point in a grid[16]

Note 1 to entry: Alternative terms are crystal orientation map (COM), automated crystal orientation map and orientation imaging microscopy map.

3.~~18~~14
~~**orientation noise**~~
~~distribution of orientations resulting from a large number of orientation measurements made within a region of a perfect single crystal~~

~~Note 1 to entry: The region shall be small enough that electron beam movement over the region does not cause any detectable change in orientation.~~

~~Note 2 to entry: This distribution is a reflection of the statistical nature of the angular resolution of the EBSD technique.~~

~~**3.19**~~
misorientation
difference in the orientation of two crystallites, usually expressed as an angle/axis pair

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Note 1 to entry: Misorientation is the rotation required to bring one crystal grain into coincidence with another. It can be described by a rotation matrix, a set of Euler angles, an axis/angle pair or a Rodriguez vector. The axis/angle pair is most common, but the smallest angle description is generally used.

Note 2 to entry: The EBSD software calculates the crystal orientation of a particular point on the specimen surface based on the *EBSP* (3.8) acquired at that point. The software can then calculate the misorientation between any two chosen acquisition pixels (which can or cannot be neighbours in the orientation map)[17].

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3.~~20~~15
~~**local misorientation**~~
~~**LM**~~
~~assigned to the average value of the misorientations between each pixel and nearest neighbours~~

~~**3.21**~~
~~**kernel average misorientation**~~
~~**KAM**~~
~~calculation value of the average misorientation between each pixel and its nearest neighbours~~

~~**3.22**~~
~~**local average misorientation**~~
~~**LAM**~~
~~assigned to the average value of the misorientations between each pixel and the average orientation of the kernel~~

~~**3.23**~~
transmission Kikuchi diffraction
TKD

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