INTERNATIONAL STANDARD

ISO 24173

First edition
2009-09-01

Microbeam analysis — Guidelines for orientation measurement using electron backscatter diffraction

Analyse par microfaisceaux — Lignes directrices pour la mesure d'orientation par diffraction d'électrons rétrodiffusés

Reference number
ISO 24173:2009(E)

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

Published in Switzerland

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 24173 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*.

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 identify also 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 on the instrument operating parameters. 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 its use in combination with an accurate serial sectioning technique, such as focussed-ion beam milling [4].

It is strongly recommended that EBSD users 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 produced [5],[6].

Microbeam analysis — Guidelines for orientation measurement using electron backscatter diffraction

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 gives advice 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 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*

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.

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 [7],[8]

NOTE 1 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 Simulations of the atomic arrangement in a small ($4 \times 4 \times 4$ 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 For those unfamiliar with crystallography, it is recommended that a standard textbook be consulted (see for example References [7], [8] and [9]).

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

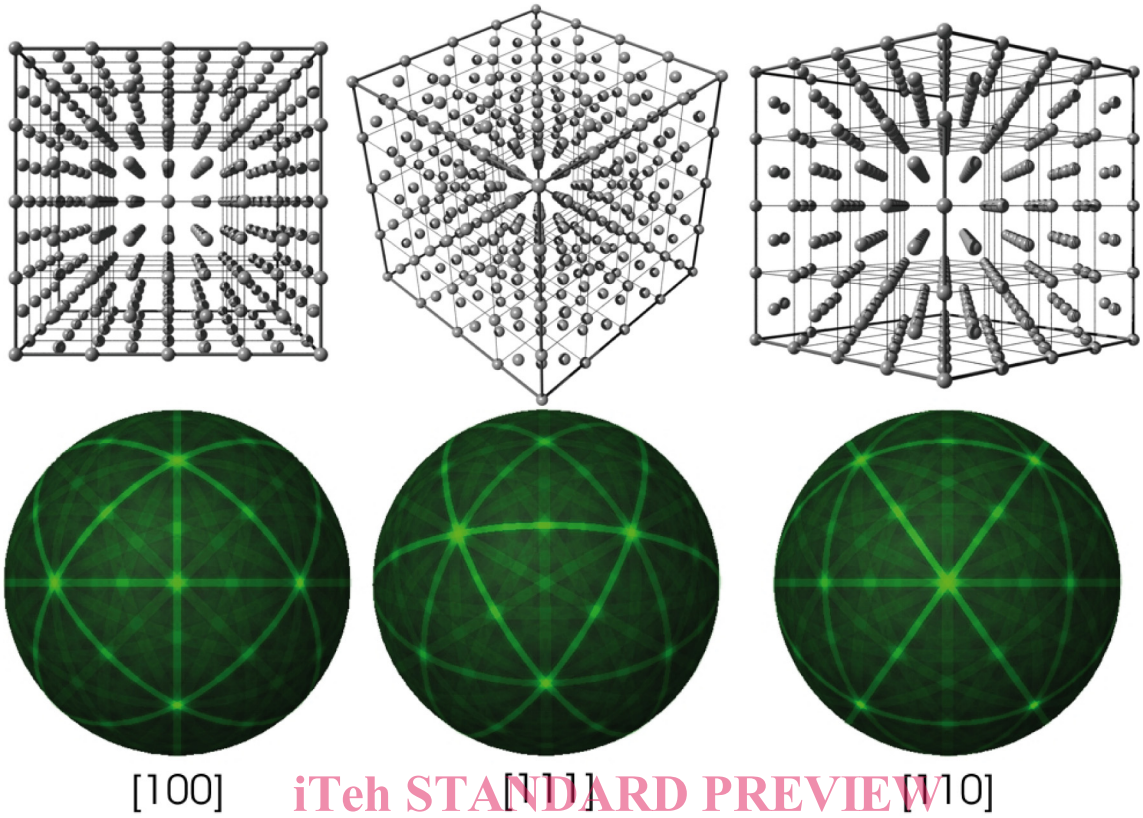


Figure 1 — Simulations of a small aluminium crystal (top) as viewed along the [1 0 0], [1 1 1] and [1 1 0] directions, with their associated spherical Kikuchi patterns (bottom). The symmetry is clearly shown.

3.2
crystal plane
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 integers

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

NOTE 2 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 See Annex C for more information.

3.4
crystal unit cell
cell which is repeated (infinitely) to build up the crystal

NOTE 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 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].

3.6
EBSD detector
detector used to capture the electron backscatter pattern and convert it to an image visible on the display device (computer screen) via a video-camera, which is commonly a high-sensitivity charged-coupled device (CCD)

NOTE See also 3.21.

3.7
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 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).

3.8
electron backscatter pattern
EBSP
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

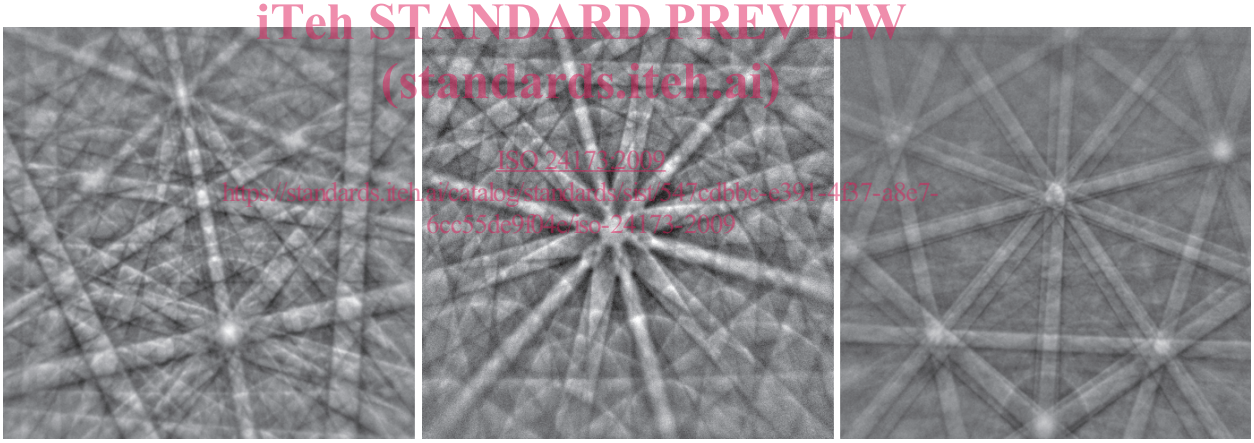


Figure 2 — Examples of EBSPs showing arrays of overlapping Kikuchi bands

3.9
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 [10]

3.10
EBSD spatial resolution
minimum distance between two points in different grains (separated by a sharp boundary) that produces two distinctly different EBSPs that can be correctly indexed using an EBSD system

NOTE 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 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.

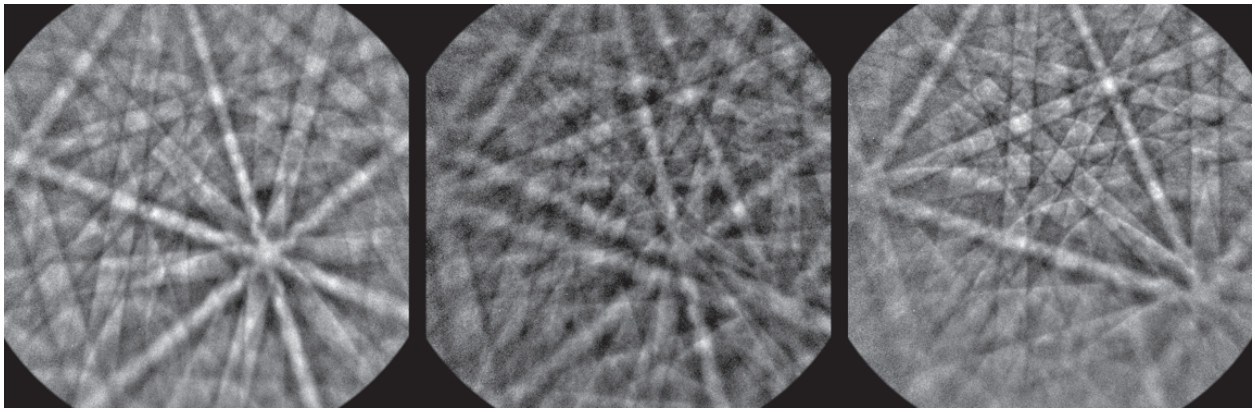


Figure 3 — Examples of EBSPs from either side (far left and far right) and on a grain boundary (centre)
(Note that these images were taken at 30 nm spacings and the centre EBSP is a combination of the other two)

3.11
Euler angles
set of three rotations for representing the orientation of a crystal relative to a set of specimen axes

NOTE 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 [6].

3.12
Hough transform
mathematical technique of image processing which allows the automated detection of features of a particular shape within an image

NOTE In EBSD, a linear Hough transform is used to identify the position and orientation of the Kikuchi bands in each EBSP, 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 [11],[12]. See 5.3.7 for more details.

3.13
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)

3.14
microtexture
population of crystallographic orientations whose individual components are linked to their spatial location within the microstructure [13]

3.15
misorientation
difference in the alignment of the coordinate systems of two crystals, usually expressed as an angle/axis pair

NOTE 1 Misorientation is the rotation required to bring one crystal 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 The EBSD software calculates the crystal orientation of a particular point on the specimen surface based on the EBSP acquired at that point. The software can then calculate the misorientation between any two chosen acquisition points (which may or may not be neighbours in the orientation map). [14]

3.16
orientation
alignment of a crystal relative to a set of specimen axes

NOTE It is usually represented by Euler angles (ϕ_1, Φ, 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
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 [15]

NOTE Alternative terms are crystal orientation map (COM), automated crystal orientation map and orientation imaging microscopy map.

3.18
orientation noise
distribution of orientations resulting from a large number of orientation measurements made within a region of a perfect single crystal

NOTE 1 The region must be small enough that electron beam movement over the region does not cause any detectable change in orientation.

NOTE 2 This distribution is a reflection of the statistical nature of the angular resolution of the EBSD technique.

3.19
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

3.20
phase identification
crystallographic identification of an unknown phase in a specimen by comparing the features of the acquired EBSP with those simulated or calculated from a set of possible candidate phases [16],[17],[18]

NOTE This can be an automatic process in which the EBSD software searches a preselected set of crystal phase databases and determines the phase whose simulated EBSP best matches the acquired EBSP. In this situation, the procedure is referred to as phase discrimination. Alternatively, it can be a manual process in which features of the EBSP, such as its symmetry, band widths and HOLZ (higher-order Laue zone) lines are used in the identification procedure. In either case, information about the chemical composition obtained using energy-dispersive X-ray spectrometry (EDX) or wavelength-dispersive X-ray spectrometry (WDX) can be additionally used to reduce the list of possible phases, thereby speeding up the process and providing an increased level of confidence in the results.

3.21
phosphor screen
screen used to convert the electron diffraction pattern to a visible light signal which can be detected with a low-light-level camera

NOTE Most EBSD phosphors are made of a thin layer of phosphor particles, ≈ 4 µm to 10 µm in size, held together with a binder and having a final aluminium coating that both dissipates charge and acts as a mirror to increase the EBSP signal but is thin enough to be relatively electron-transparent.

3.22
pseudosymmetry
potential for an EBSP to be ambiguously indexed in several ways due to internal similarities between the EBSPs for certain crystal orientations

NOTE 1 This is a problem with some minerals, e.g. quartz and olivine, and can also occur with some metallic phases.

NOTE 2 A common example is when the <1 1 1> zone in bcc iron is near the centre of the EBSP, as shown in Figure 4. If only the circular region shown is used for band detection, then it is very difficult to distinguish between these two orientations. The <1 1 1> zone has an apparently 6-fold axis, although it really has only 3-fold symmetry, and only weaker Kikuchi bands near the edges of the region can distinguish the two possible 3-fold axes.

NOTE 3 Pseudosymmetry effects can usually be minimized by decreasing the specimen-to-screen distance, in order to capture more Kikuchi bands, and by using more bands for indexing.

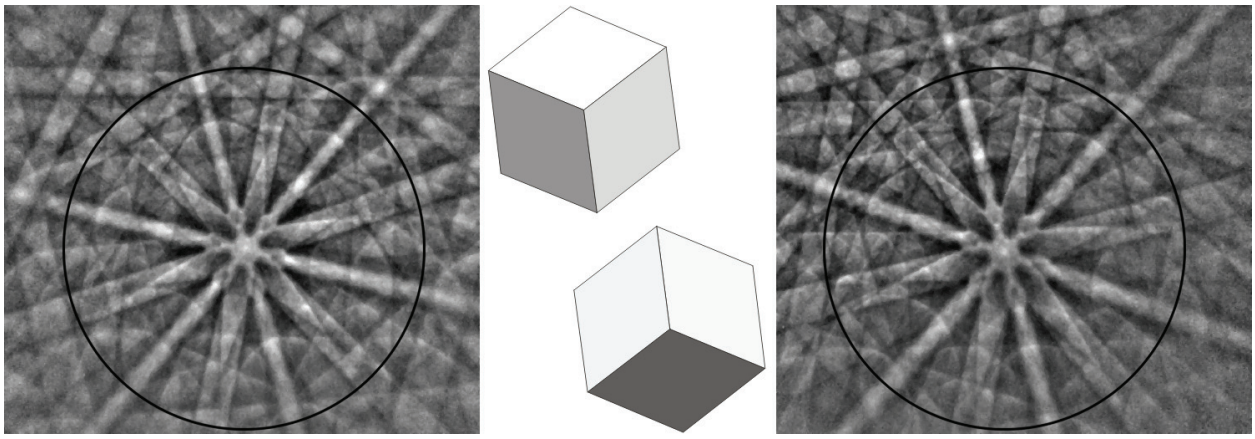


Figure 4 — Pseudosymmetry in bcc iron around a <1 1 1> zone
(If only the stronger Kikuchi bands within the circle are used, then the two EBSPs can be indexed as either of the two orientations shown which are related by a 60° rotation about <1 1 1>)

3.23
specimen-to-screen distance
SSD

distance between the plane of the detector screen and the point where the electron beam strikes the specimen, measured perpendicular to the pattern centre

NOTE If the specimen-to-screen distance decreases, then the EBSP will appear to zoom out about the pattern centre, i.e. more Kikuchi bands will be seen.

3.24
spherical Kikuchi map
SKM

representation of the EBSP diffraction pattern projected on to the surface of a sphere, as shown in Figure 5, the diffracted signal emanating spherically from a point source on the specimen surface

NOTE 1 Spherical Kikuchi maps are useful in that they avoid the distortions associated with the gnomonic projection of the EBSD signal onto the flat phosphor screen used to capture each EBSP.

NOTE 2 The spherical Kikuchi map is centred about the specimen and aligned with the crystallographic directions of the crystal being examined. As the crystal is rotated, the spherical Kikuchi map moves in synchrony.

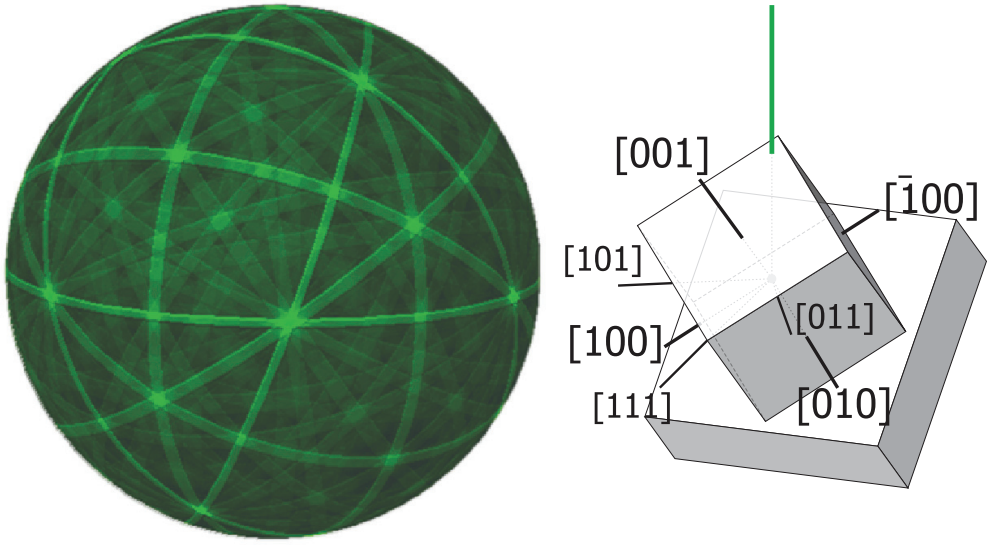


Figure 5 — Schematic diagram showing a silicon unit cell (right) with the main crystal directions labelled and, on the left, a spherical Kikuchi map of silicon at the same orientation
(This orientation is the standard silicon calibration orientation for a 70° tilted specimen; the incident electron beam direction is shown)

3.25
symmetry
property an object is said to have if it looks the same when rotated, translated or mirrored in a certain way

NOTE For further information, see Annex C.

3.26
zone axis
point in an EBSP where the centres of several Kikuchi bands intersect

NOTE It corresponds to a low-index crystal direction in the EBSP.

3.27
Bravais lattice
three-dimensional geometric arrangement of the atoms or molecules or ions making up a crystal

4 Equipment for EBSD

4.1 SEM, EPMA or FIB instrument, fitted with an electron column and including controls for beam position, stage, focus and magnification (see Figure 6).

4.2 Accessories, for detecting and indexing electron backscatter diffraction patterns, including:

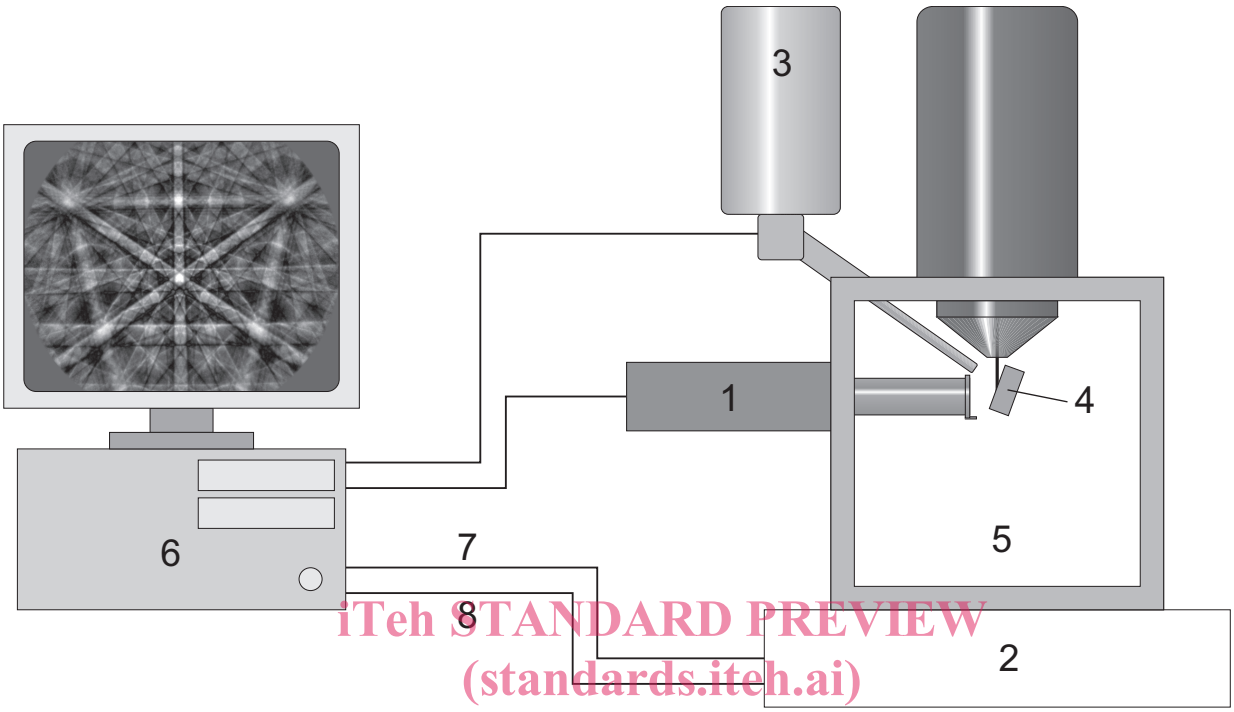
4.2.1 Phosphorescent ("phosphor") screen, which is fluoresced by electrons from the specimen to form the diffraction pattern.

4.2.2 Video camera, with low light sensitivity, for viewing the diffraction pattern produced on the screen.

4.2.3 Computer, with image processing, computer-aided pattern indexing, data storage and data processing, and SEM beam (or stage) control to allow mapping.

NOTE 1 Modern systems generally use charge-coupled devices (CCDs).

NOTE 2 Some systems incorporate detector(s) mounted around the phosphor screen to detect electrons scattered in the forward direction from the specimen; the detectors are usually silicon diodes, similar to those used in solid-state backscatter detectors. The images (orientation and atomic number contrast) give a rapid overview of the specimen microstructure [19].



Key

1	EBSD instrument	5	chamber
2	SEM	6	EBSD computer
3	EDX (energy-dispersive spectrometer) (optional)	7	beam control
4	tilted specimen	8	SEM and stage control

Figure 6 — Diagram of an experimental EBSD arrangement

4.3 If specimens need to be prepared for EBSD, the following equipment might be required (depending on the types of specimen to be prepared — see Annex B): cutting and mounting equipment, mechanical grinding and polishing equipment, electrolytic polisher, ultrasonic cleaner, ion-sputtering equipment and coating equipment.

5 Operating conditions

5.1 Specimen preparation

The volume of material sampled by the electron beam during EBSD analysis must be crystalline. The crystal features (e.g. grain size, deformation state) of this volume shall be representative of the bulk specimen or of the part of the specimen about which the nature of the microstructure will be inferred in the case of segmented microstructures (e.g. layered thin films or heat-affected/non-heat-affected zones near welds). Since the EBSP is generated by electron diffraction within a few tens of nanometres of the specimen surface, very good preparation of the specimen surface is required to ensure this and to prevent the EBSD data from being deleteriously affected by inadequate preparation. The top layer under investigation shall be free from deformation due to specimen preparation and flat. Poor specimen preparation can leave deformation at, or just below, the surface or can leave contaminants, oxides or reaction product layers on the specimen surface. Due to the high tilt of the specimen surface (typically 70°) with respect to the electron beam, minimizing

surface relief is also an important part of good specimen preparation. Guidelines on specimen preparation for EBSD are given in Annex B.

5.2 Specimen alignment

Accurate calibration (see Clause 6) and measurement using EBSD requires careful specification of the alignment between the coordinate systems of the specimen, the SEM scanning coils, the stage and the EBSD detector. The specimen shall be aligned in the microscope such that the normal to the acquisition surface is at a chosen tilt angle (typically ≈ 70°) to the electron beam and such that a reference direction on the acquisition surface, often a specimen edge, is parallel to both the stage tilt axis and, in the case of beam scanning, to one axis of the beam-scanning system. Accurate alignment can be achieved more easily when the specimen is mounted on a stage that allows rotation of the specimen within the tilted acquisition plane, since fine adjustment can be performed with the specimen inside the microscope. First, the specimen reference direction shall be aligned with the stage tilt axis. This alignment can be verified by moving the stage back and forth along the tilt axis and checking in the electron image that the specimen reference direction moves back and forth through a fixed point on the display, such as a particular intersection point on a grid overlay. The long axis of the beam scan can then be aligned with the tilt direction by adjusting the scan rotation until these two directions appear aligned in the electron image. If a pre-tilted specimen holder is being used (or the stage does not allow rotation within the acquisition plane), then it is critical that the specimen be mounted with the specimen reference direction as close as possible to one of the orthogonal SEM stage axes.

With a manual-tilt stage, a mechanical end-stop at the desired tilt angle is recommended so that the stage can be tilted to the desired tilt angle with better reproducibility.

5.3 Common steps in collecting an EBSP

5.3.1 Setting the microscope operating conditions

5.3.1.1 Accelerating voltage

To contribute to the formation of the pattern, the electrons must have sufficient energy so that, when backscattered, they retain enough energy to cause scintillation in the phosphor screen. This also increases the number of electrons falling on the screen and thus the brightness of the diffraction pattern. This allows the integration time of the camera to be reduced but will make the spatial resolution poorer by increasing the electron beam size. Note, however, that this reduced resolution is typically only a small effect. An accelerating voltage ranging between 15 kV and 30 kV is recommended for most applications. Increasing the accelerating voltage reduces the electron wavelength and hence reduces the width of the EBSD bands in the diffraction pattern. Higher accelerating voltages within this range are beneficial for analysing the material below a very thin (up to approximately 10 nm) conducting coating or very thin layer of surface deformation. Lower accelerating voltages might be required for analysing thin (10 nm to 50 nm) surface films or to reduce charging on specimens with poor conductivity.

5.3.1.2 Probe current

Increasing the probe current will increase the number of electrons contributing to the diffraction pattern and so allow the camera integration time to be reduced, allowing faster mapping. However, this advantage must be balanced against the associated loss of spatial resolution because increasing the probe current results in the EBSD signal being generated from a larger volume in the specimen and also increases problems due to both charging and contamination effects.

The electron beam shall be focussed on the specimen surface and dynamic focussing used, if available, to compensate for the tilted specimen.

5.3.2 Detector and working distances

For general use, the ideal working distance for EBSD is the working distance at which the brightest region of the raw EBSP (i.e. without background correction) is in the centre of the phosphor screen. Other experiments can dictate a different position. Pattern intensity can be increased by increasing the camera gain but at the