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Second edition

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

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Foreword

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

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

The main changes are as follows:

— Clause 3 has been updated;

— “in the working position” is changed to “in the detector position” (see 6.6 (d));

— subclause “7.1 Pre-test preparation” in the previous edition is omitted;

— “Annex B (normative)” is changed to “Annex B (informative)”;

— 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.html.

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]

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

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

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.

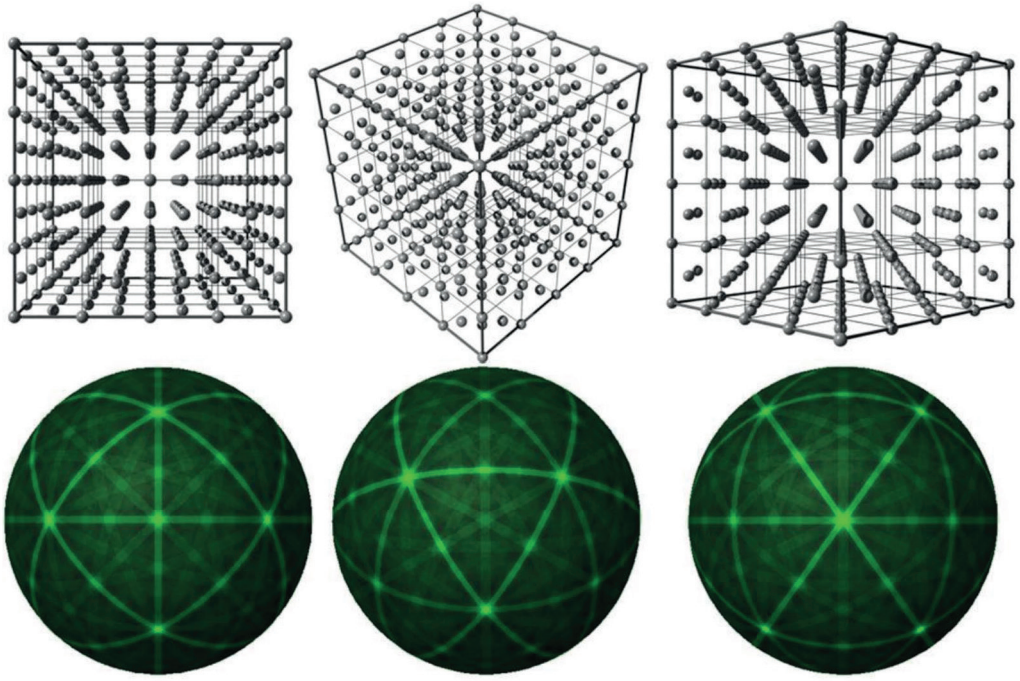


Figure 1 — Simulations of a small aluminium crystal (top) as viewed along the [1 0 0], [1 1 1] and [1 1 0] directions, with their associated spherical Kikuchi patterns (bottom). The symmetry is clearly shown.

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 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
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].

3.5
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)

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”), “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.

3.7
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

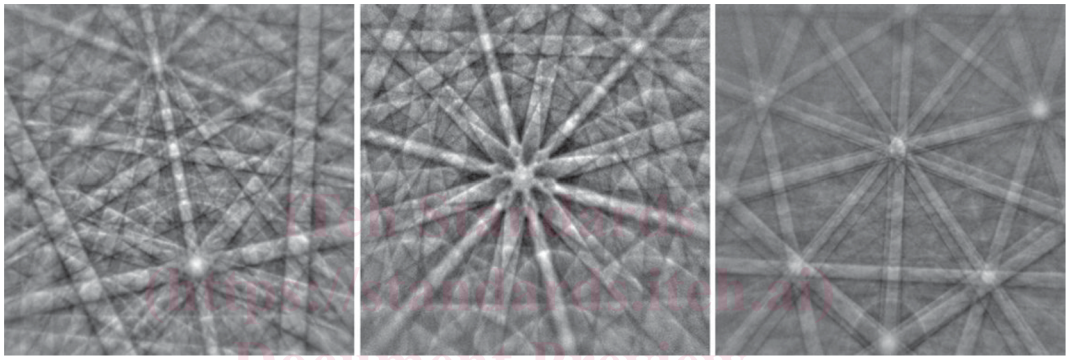


Figure 2 — Examples of EBSPs showing arrays of overlapping Kikuchi bands

3.8
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

3.9
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].

3.10
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.

3.11
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)

3.12
orientation
alignment of a crystal axes relative to a set of specimen axes

Note 1 to entry: 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.

3.13
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.

3.14
misorientation
difference in the orientation of two crystallites, usually expressed as an angle/axis pair

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].

3.15
transmission Kikuchi diffraction
TKD
SEM-based electron-transparent diffraction applies conventional EBSD hardware to a sample

Note 1 to entry: Commonly used alternative terms for TKD are “t-EBSD”[5].

Note 2 to entry: It has been proven to enable spatial resolutions better than 10 nm. This technique is ideal for routine EBSD characterisation of both nanostructured and highly deformed samples.

3.16
phase identification
crystallographic identification of an unknown phase in a specimen by comparing the features of the acquired *EBSP* (3.7) with those simulated or calculated from a set of possible candidate phases [16],[17],[18]

Note 1 to entry: 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* (3.7) best matches the acquired EBSP. In this situation, the procedure is referred as phase discrimination. Alternatively, it can be a manual process in which features of the *EBSP* (3.7), 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.

3.17
phosphor screen
screen used to convert the electron signal to a visible light signal which can be detected with a low-light-level camera

Note 1 to entry: 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* (3.7) signal but is thin enough to be relatively electron-transparent.

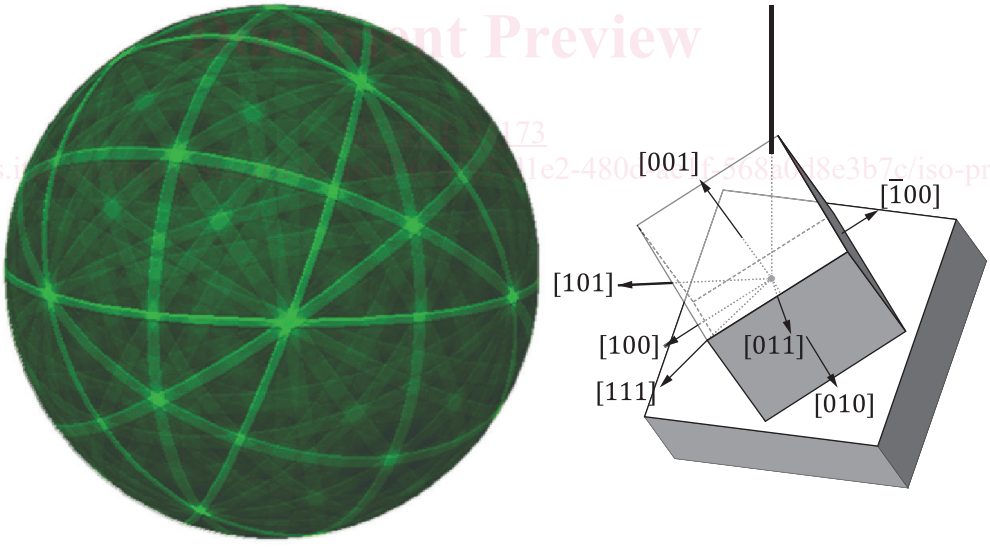
3.18
specimen-to-screen distance
SSD
distance between the pattern centre in the detector screen and the point where the electron beam strikes the specimen

Note 1 to entry: If the specimen-to-screen distance decreases, then the *EBSP* (3.7) will appear to zoom out about the pattern centre, i.e. more Kikuchi bands will be seen.

3.19
spherical Kikuchi map
SKM
representation of the *EBSP* (3.7) diffraction pattern projected on to the surface of a sphere, as shown in Figure 3, the diffracted signal emanating spherically from a point source on the specimen surface

Note 1 to entry: 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* (3.7).

Note 2 to entry: 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.



NOTE This orientation is the standard silicon calibration orientation for a 70° tilted specimen; the incident electron beam direction is shown.

Figure 3 — 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

3.20
symmetry
property an object has if it looks the same when rotated with a certain angle, translated or mirrored in a certain way

Note 1 to entry: For further information, see Annex C.

3.21
zone axis
point in an *EBSP* (3.7) where the centres of several Kikuchi bands intersect

Note 1 to entry: It corresponds to a low-index crystal direction in the *EBSP* (3.7).

3.22
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 4).

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

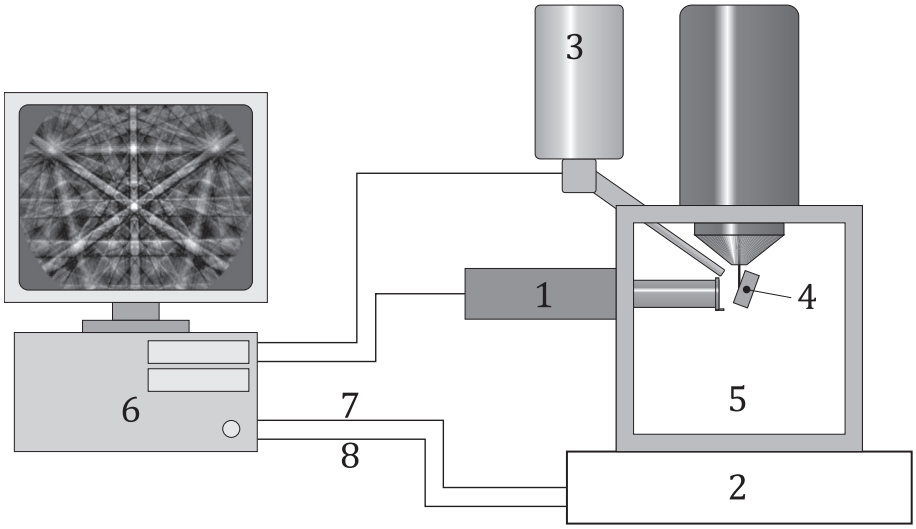
4.2.1 Phosphorescent ("phosphor") screen, which is fluoresced by electrons from the specimen to form the diffraction pattern.

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) or complementary metal-oxide-semiconductors (CMOS).

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 4 — Diagram of an experimental EBSD arrangement

4.3 If specimens need to be prepared for EBSD, the following equipment can 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 shall be crystalline. The crystal features (e.g. grain size, deformation state) of this volume should be representative of the bulk specimen or 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 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 Kikuchi bands in the diffraction pattern. Lower 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.

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 shall 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 expense of increasing noise levels. Short working distances will generally improve the spatial resolution of EBSD measurements, although additional care has to be taken to avoid collisions between the specimen and the SEM pole-piece or the backscatter detector (if present).

The ideal detector (specimen-to-screen) distance for EBSD depends on the size of the phosphor screen and on the nature of the analysis being conducted. For a typical EBSD investigation, the phosphor screen is placed approximately 15 mm to 25 mm from the intersection point between the electron beam and the specimen. With a smaller specimen-to-screen distance, more bands are captured in each EBSP, which can be useful for improving the indexing of low-symmetry phases and for improving discrimination between phases or of orientations with similar (pseudosymmetric) EBSPs. With a larger specimen-to-screen distance, a smaller region of diffraction space is imaged on the phosphor screen,

