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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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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.

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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]