International Standard

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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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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 document 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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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.