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Microbeam analysis — Analytical electron microscopy — Method of determination for apparent growth direction of nanocrystals by transmission electron microscopy

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Foreword

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This document was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 3, *Analytical electron microscopy*.

This second edition cancels and replaces the first edition (ISO 19214:2017), which has been technically revised.

The main changes are as follows:

— the title, introduction and scope have been revised;

— Clause 3 has been revised;

— Figures 1 and 2 have been replaced;

— Annex D has been added;

— editorial revisions have been made.

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.

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Introduction

Nanocrystals are a main component in some advanced materials, especially nanomaterials, and also appear in traditional materials, such as needle-shaped precipitates in steels and alloys. Controlling the microstructure of these materials during fabrication is very important for quality control considerations. To control the microstructure and thereby improve the service properties of the relevant materials, the apparent growth direction, or the longest axis of the nanocrystals is one of the essential parameters. This direction of nanocrystals is generally determined by transmission electron microscopy (TEM).

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Microbeam analysis — Analytical electron microscopy — Method of determination for apparent growth direction of nanocrystals by transmission electron microscopy

1 Scope

This document gives a method for determination of the apparent growth direction of nanocrystals by transmission electron microscopy. This method is applicable to all kinds of wire-like crystalline materials synthetized by various methods. This document can also guide in determining an axis direction of the second-phase particles in steels, alloys, or other materials. The applicable diameter or width of the crystals to be tested is in the range of tens to one hundred nanometres, depending on the accelerating voltage of the transmission electron microscope (TEM) and the material itself. Position, which is curved, twisted, and folded, to determine the apparent growth direction, should not be used.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes the 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.

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ISO 15932, *Microbeam analysis — Analytical electron microscopy — Vocabulary*

ISO 25498:2018, *Microbeam analysis — Analytical electron microscopy — Selected area electron diffraction analysis using a transmission electron microscope*

3 Terms and definitions

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For the purposes of this document, the terms and definitions given in ISO 15932 and the following 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
nanocrystal
discrete piece of crystalline material exhibiting a dimensional anisotropy with an axial elongation in one of the three nanocrystalline lattice direction in the nanoscale

3.2
apparent growth direction
crystalline direction which is parallel to the longest dimension of a single crystal

Note 1 to entry: Apparent growth direction does not involve mechanisms of the phase interface migration.

3.3
Miller notation
indexing system for diffraction patterns, which describes a crystal lattice by three axes coordinate

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3.4
Miller-Bravais notation
indexing system for diffraction patterns of hexagonal crystal, which describes the lattice by four axes coordinate

3.5
reciprocal vector
$\boldsymbol{g_{hkl}}$
coordinate vector of *hkl* lattice point in the reciprocal lattice

Note 1 to entry: Reciprocal vector g_{hkl} is perpendicular to the plane (*hkl*) of crystal, its length is inversely proportional to the interplanar spacing d_{hkl}.

[SOURCE: ISO 25498:2018, 3.8, modified — Note 1 to entry has been modified.]

3.6
***R* vector**
$\boldsymbol{R_{hkl}}$
coordinate vector from the central spot *000* to the diffraction spot *hkl* in a diffraction pattern

[SOURCE: ISO 25498:2018, 3.9, modified — Note 1 to entry has been removed.]

3.7
reciprocal space
imaginary space where planes of atoms are represented by reciprocal points and all lengths are the inverse of their length in real space

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4 Specimens

4.1 The sample crystals shall be clean, without contamination or oxidation. They are stable under electron beam irradiation during TEM analysis.

4.2 Powder or extracted powder specimens of the crystals may be analysed. The sample powder shall be well dispersed by a suitable technique so that individual crystals can be observed under the TEM.

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NOTE One of the techniques in common use is ultrasonic dispersion. In this method, the sample powder is immersed in ethanol or pure water and dispersed by ultrasonication for about 0,5 h to 1 h, then dropped onto the supporting film surface of a microgrid. Then, the microgrids are dried at room temperature. The wire-like crystals are usually parallel to the supporting film plane. Other techniques to prepare individual crystal specimens can also be adopted, depending on the physical characteristics of the sample.[1]

4.3 The precipitates or second-phase particles in steels, alloys and the like should be extracted, then treated as powder specimens; see 4.2.

4.4 Thin-foil specimens of various solid substances prepared by suitable methods (focused ion beam, ion beam thinning, etc.) are applicable. The specimen shall be thin enough to transmit the electron beam.[2]

5 Analysis procedure

5.1 Setting the TEM operating condition

5.1.1 Preparation of the TEM

The TEM working condition shall comply with ISO 25498:2018, 8.1.

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5.1.2 Accelerating voltage

The applicable accelerating voltage of the TEM for the analysis mainly depends upon the thickness of the specimen to be studied. Stability of the crystals under electron beam irradiation is also important for the accelerating voltage setting. As long as the structure and/or morphology of the specimen is not altered during the analysis, clear images and sharp diffraction patterns can be obtained on the TEM. The corresponding accelerating voltage or higher may be suitable for the work.

5.1.3 Setting the specimen

Place the specimen to be tested firmly in the double-tilting or tilting-rotation specimen holder, then insert the holder into the specimen chamber. It is recommended to use the cold finger of the TEM before conditioning.

5.1.4 Calibration of the rotation angle

As specified in ISO 25498:2018, 8.1.6, to be able to successfully correlate the axis of interest in an image with the corresponding diffraction pattern, the rotation angle between the micrograph and its corresponding diffraction pattern may need to be calibrated. A molybdenum trioxide crystal specimen may be used as a reference for the rotation angle calibration. The analyst may refer to textbooks such as References [3] and [4] for the experimental procedure for this calibration.

NOTE For some transmission electron microscopes, the rotation angle has been compensated by the manufacturer. In this case, step 5.1.4 can be ignored.

5.2 Data acquisition

5.2.1 Select the target crystal

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On the viewing screen, TV monitor, or computer screen of the TEM, get an overview image of the specimen in low magnification mode. Select an individual crystal which is clean and free from damage or distortion as the target. Under bright-field imaging mode, adjust the magnification to get a clear magnified image of the target crystal. Adjust the specimen height (Z axis) to the eucentric position. Adjust the focal length of the images.

5.2.2 Obtaining diffraction patterns

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5.2.2.1 General

Various electron diffraction techniques may be applicable for the determination of the crystal axis direction. The selected area electron diffraction (SAED) and microbeam diffraction techniques are in common use; however, for the present purpose, the spot diffraction patterns or the patterns formed by the incident beam through a small angle aperture are preferred.

5.2.2.2 Procedure

The procedure for taking diffraction patterns and micrographs of the target crystal is as follows.

a) Select a suitable position of the target crystal in the specimen and select a diffraction mode (SAED, microbeam diffraction, or other suitable mode). Switch to the diffraction mode to get a spot diffraction pattern. Tilt the specimen slightly so that the brightness distribution on the diffraction pattern is symmetrical and a zero-order Laue zone pattern is displayed. Therefore, the zone axis, Z_1 (with index $[u_1v_1w_1]$), of this diffraction pattern is nearly reverse parallel to the incident beam direction, B_1. Record this diffraction pattern, Z_1, and take note of the reading on the X and Y tilting angle of the double tilting specimen stage as X_1 and Y_1, respectively.

   NOTE Refer to the instruction manual provided by the microscope manufacturer for the operation procedure for each diffraction mode.

b) Switch back to the imaging mode without changing the specimen orientation to get a correlative bright field image, M_1, of the target crystal. Check the focus of this image and take a photo or save

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it in the computer system. This image, M_1, is formed under the incident beam direction, B_1, which is approximately reversely parallel to the zone axis, Z_1.

c) Return to the diffraction mode and tilt the specimen to produce a second diffraction pattern with zone axis Z_2. Record this diffraction pattern, Z_2, and take note of the reading on the X and Y tilt angle of the specimen holder as X_2 and Y_2, respectively.

d) Repeat step b) to form the second bright field image, M_2, of the target crystal. This image, M_2, is formed under the incident beam direction, B_2, which is nearly reversely parallel to the zone axis, Z_2, of the specimen.

e) The angle, ψ, between the two specimen holder positions (that is, the angle ψ^* between the zone axis, Z_1, with index $[u_1v_1w_1]$ and Z_2, with index $[u_2v_2w_2]$) can be obtained from the differences between the readings on the X and Y tilting angles at each position (see ISO 25498:2018, 8.2).

5.2.3 Determining the interplanar spacing

To determine the interplanar spacing, d_{hkl}, of the plane (hkl) in crystals, the simplified Bragg law, as shown in Formula (1), shall be followed.

$$L\lambda = R_{hkl} \times d_{hkl} \qquad (1)$$

where

- L is the camera length;
- λ is the wavelength of the incident electron beam;
- R_{hkl} is the distance between the central spot and the diffracted spot of a crystalline plane (hkl) in the diffraction pattern;
- d_{hkl} is the interplanar spacing of the crystalline plane (hkl).

$L\lambda$ is the camera constant. Transmitted spot should be coincident with the optic axis. It is necessary that the central spot is the transmitted spot of used diffraction pattern.

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When the camera constant $L\lambda$ is known, the interplanar spacing d_{hkl} can be found, in principle, using Formula (1) by measuring the distance R_{hkl}. However, in practice, $2R_{hkl}$ (the distance between the spots hkl and $\bar{h}\,\bar{k}\,\bar{l}$) shall be measured, then divided by two to calculate the distance R_{hkl}.

In most cases, the camera constant, $L\lambda$, shall be calibrated for the present work. The practical procedure for camera constant calibration is specified in ISO 25498:2018, 8.3.

Camera constant, $L\lambda$, calibration is usually performed by using a reference specimen such as polycrystalline pure gold or pure aluminium. At a given accelerating voltage, record the ring diffraction pattern of the reference specimen. Index the diffraction rings and measure the diameters $2R_{hkl}$ of the corresponding ring (hkl), respectively. Find the interplanar spacing d_{hkl} for a plane (hkl) of the reference specimen by the crystallographic formulae. The camera constant, $L\lambda$, can then be calculated using Formula (1). In practice, either the $L\lambda \sim D/2$ plot or an average value of the camera constant may be used.

When the crystalline structure and the confident lattice parameters of the specimen are already known, the diffraction constant, $L\lambda$, may be calculated from its diffraction pattern directly. The approximate value of $L\lambda$ can be found on a console readout display of a modern TEM.

5.2.4 Index diffraction patterns

For specimens comprised of crystals in the nanometre size regime, most of the time, only spot diffraction patterns can be observed. Kikuchi patterns seldom appear owing to their small thickness. Therefore, only the procedure for indexing spot diffraction patterns is specified in this document.

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