



**International
Standard**

ISO 19214

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

**Second edition
2024-10**

Reference number
ISO 19214:2024(en)

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Published in Switzerland

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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.

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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.

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).

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 synthesized 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.

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

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

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

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

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

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.

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.

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

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

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 $[u_1 v_1 w_1]$), 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