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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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Contents

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.

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 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.

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
$\boldsymbol{g}_{hkl}$
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

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 $[u_1v_1w_1]$), 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 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.

$$L\lambda = R_{hkl} \times d_{hkl} \tag{1}$$

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.

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.

The practical procedure for indexing diffraction patterns may refer to ISO 25498:2018, Clause 9. For the convenience of applying this document, the indexing process is briefly summarized as follows:[3][4][5][6]

a) Select two diffracted spots, $h_1k_1l_1$ and $h_2k_2l_2$, from the diffraction pattern such that these spots are nearest and next-nearest to the central spot, *000*, respectively. Measure the length of correlative vectors $R_{h_1k_1l_1}$ and $R_{h_2k_2l_2}$, which are defined as the vector from the origin, *000*, to the diffraction spot $h_1k_1l_1$ and the spot $h_2k_2l_2$, respectively, in the diffraction pattern. Calculate the corresponding inter-planar spacing $d_{h_1k_1l_1}$ and $d_{h_2k_2l_2}$. Then, assign tentative index values for each spot.

b) Measure the included angle between the vectors $R_{h_1k_1l_1}$ and $R_{h_2k_2l_2}$ as well as the angle between $R_{h_2k_2l_2}$ and $R_{h_2-h_1,\, k_2-k_1,\, l_2-l_1}$ respectively, where $R_{h_2-h_1,\, k_2-k_1,\, l_2-l_1}$ is defined as the *R* vector of the diffraction spot with index h_2-h_1, k_2-k_1, l_2-l_1. Adjust the indices for each spot such that the angle is coincident with the calculated angle by the crystallographic formulation. When the experimental value is consistent with the known value within error, the diffraction spots can be indexed.

c) Calculate the zone axis, *Z*, of the diffraction pattern [*u v w*] by the zone multiplication law; see Formula (2):

$$u:v:w=(k_1l_2-k_2l_1):(l_1h_2-l_2h_1):(h_1k_2-h_2l_1) \tag{2}$$

If the indices *u*, *v* and *w* contain a common integral factor *n*, divide them all by the common factor *n*.

d) Assign consistent indices to the remaining spots on the diffraction pattern by using the vector addition rule.

e) When suitable software is installed with the TEM, measurement of and calculations on the diffraction patterns can be carried out by the computer system.

5.2.5 Non-uniqueness of the indexing result

For a diffraction pattern containing only two-dimensional information (i.e. a planar section of single-crystal diffraction patterns), the indexing of this pattern is not unique. Causes for the non-uniqueness of the indexing may be considered from two factors.

Firstly, the indexing non-uniqueness can result from a multiplicity of crystals with high symmetry. Different variants of a diffracting plane may be equally indexed. Accordingly, the zone axis of a diffraction pattern may have a different index, i.e., variants of the zone axis, but on the stereogram, the poles of these variants are located at different positions.

Secondly, the 180° rotational symmetry of the indices *hkl* of a diffracted spot allows the index to also be described as $\bar{h}\,\bar{k}\,\bar{l}$, similarly, indices [*uvw*] of a direction can also be [$\bar{u}\,\bar{v}\,\bar{w}$]. Therefore, it is necessary to obtain two or more diffraction patterns, Z_1 and Z_2, successively by tilting the specimen. The angle, ψ^*, between the zone axes, Z_1 and Z_2, of the two patterns shall be coincident with the angle between the two specimen holder positions. Also, the indices for all of the patterns shall be consistent with each other.

5.3 Determination of the crystalline direction

5.3.1 General approach

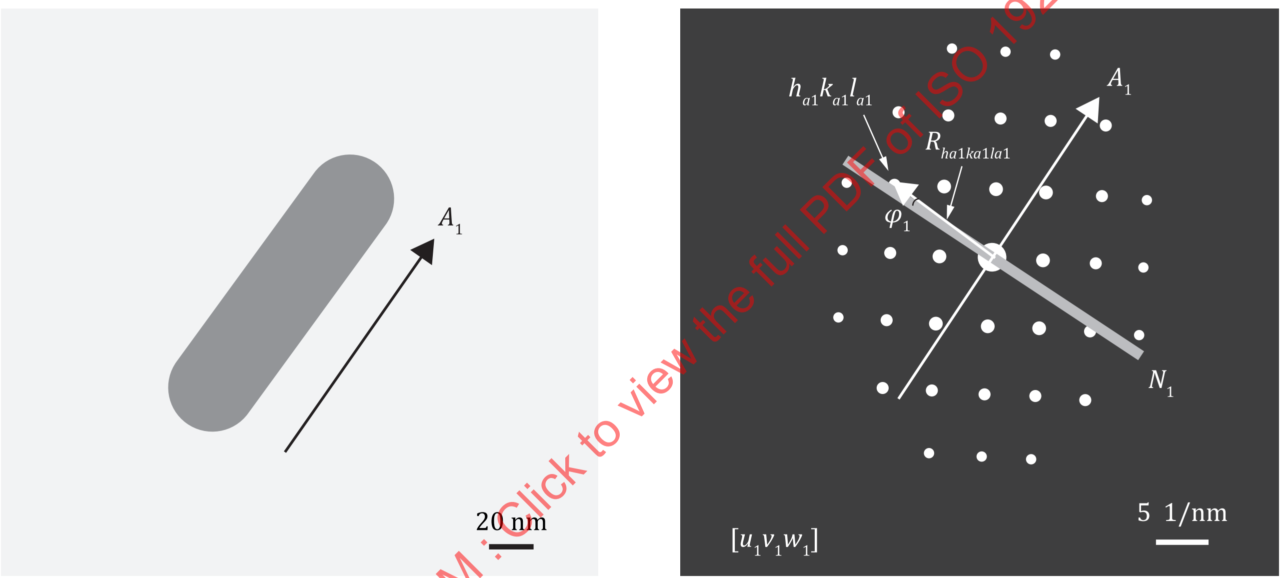
5.3.1.1 Define the projection direction

a) Specify the projection direction, A_1, of the crystal to be determined on its micrograph, M_1. As an example, schematic micrograph M_1 and corresponding diffraction pattern Z_1 of a nanoparticle are given in Figure 1(a) and (b), respectively. Align the diffraction pattern Z_1 with the micrograph M_1 carefully, after compensation of the rotation angle. Draw a line, N_1, perpendicular to the projection direction A_1 of the crystal on the diffraction pattern Z_1. This line, N_1, is the normal of the projection direction, A_1. On diffraction pattern Z_1, identify the diffraction spot with index, $h_{a1}k_{a1}l_{a1}$, which is closest to the line N_1. The vector $R_{h_{a1}k_{a1}l_{a1}}$, from the central spot, *000*, points toward diffraction spot $h_{a1}k_{a1}l_{a1}$, is the normal of plane $(h_{a1}k_{a1}l_{a1})$. Measure the angle, φ_1, between the line N_1 and vector $R_{h_{a1}k_{a1}l_{a1}}$. The projection direction A_1 of the wire-like particle and its normal, N_1, are delineated on the diffraction pattern. The spot indexed as $h_{a1}k_{a1}l_{a1}$ is the closest one to the line N_1. The angle φ_1 between the line N_1 and $R_{h_{a1}k_{a1}l_{a1}}$ is nearly zero in this case.

b) Carry out a similar operation as step a) on the second diffraction pattern, Z_2, and the micrograph M_2. Define the second projection direction, A_2, of the crystal on the diffraction pattern Z_2. Measure the angle, φ_2, between the normal N_2 of projection direction A_2 and the closest direction of vector $R_{h_{a2}k_{a2}l_{a2}}$, namely the normal of plane $(h_{a2}k_{a2}l_{a2})$ on the diffraction pattern Z_2.

c) A similar operation as described in step a) may be performed on more diffraction patterns and their corresponding micrographs. Each pair of diffraction pattern and the corresponding micrograph shall be taken under the same incident beam direction **B** and the same TEM working condition.

NOTE For hexagonal crystals, the Miller-Bravais notation can also be used to index the diffraction patterns. The relations between the Miller and Miller-Bravais notation are given in Annex A.



a) Bright field micrograph of a nanoparticle **b) Diffraction pattern $[u_1v_1w_1]$**

Figure 1 — Schematic micrograph and corresponding diffraction pattern of a nanoparticle

5.3.1.2 Determining crystallographic direction by stereography

a) Get a standard stereographic projection which corresponds to the crystal structure of the specimen, which can be obtained through crystallography software. On the standard stereographic projection, the poles for the zone axes Z_1, Z_2, and others is revealed (there can be more, depending upon the specimen) (see Figure 2).

b) Draw the great circle, C_1, corresponding to the zone axis Z_1. On this great circle, C_1, mark the pole of the plane $(h_{a1}k_{a1}l_{a1})$. Then mark the pole of the normal N_1 of projection direction A_1 on the great circle C_1 by the angle, φ_1, between the normal N_1 and the normal of plane $(h_{a1}k_{a1}l_{a1})$, which is obtained from the procedure in 5.3.1.1.

c) On the same standard stereographic projection, draw the great circle, C_2, corresponding to the zone axis Z_2. On this great circle, C_2, mark the pole of plane $(h_{a2}k_{a2}l_{a2})$, which is the normal direction of plane $(h_{a2}k_{a2}l_{a2})$. The pole of the normal N_2 of projection direction A_2 is also marked on the great circle C_2 by the angle, φ_2, between the normal N_2 and the normal of plane $(h_{a2}k_{a2}l_{a2})$. When more diffraction patterns and micrographs are acquired, repeat this operation to mark the correlative poles of the normal such as N_3 on the stereographic projection.

d) Draw a great circle, C_N, through the poles N_1, N_2 (and/or more poles of normal N_n of the projection directions obtained from other diffraction patterns and micrographs). The pole of this great circle, C_N, is then the apparent growth direction, A, with index ($H\ K\ L$), which is in terms of the plane normal. So far, all of the normal vectors are indexed in terms of the plane normal and have not been indexed as directions.

e) Convert the index of the plane normal ($H\ K\ L$) to the crystalline direction index [$u\ v\ w$] by using the crystallographic relation; see Formula (3).

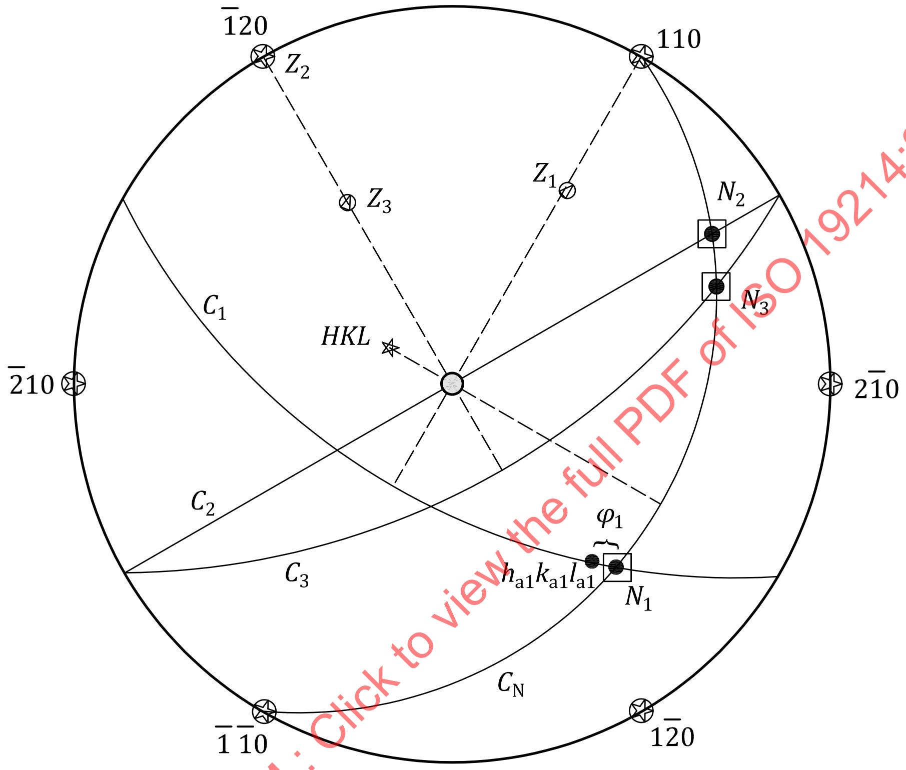


Figure 2 — Standard stereographic projection of the crystal in Miller notation

5.3.2 Convert the crystallographic index

Convert the index [HKL] of the apparent growth direction A, in other words, from the reciprocal space into direction index [uvw] of the real space by crystallographic formulae according to its crystallographic system (see Annex B). This conversion is not required only for cubic system crystals; the direction index [uvw] is equal to the index [HKL].

The normal of a plane (HKL) may be converted into the crystalline direction [uvw] through a calculation using Formula (3):

$$\begin{pmatrix} u \\ v \\ w \end{pmatrix} = G^{-1} \begin{pmatrix} H \\ K \\ L \end{pmatrix} \tag{3}$$

Conversely, a crystalline direction index [uvw] can also be converted into the normal plane (HKL) in reciprocal space by Formula (4):

$$\begin{pmatrix} H \\ K \\ L \end{pmatrix} = G \begin{pmatrix} u \\ v \\ w \end{pmatrix} \quad (4)$$

where matrices G and G^{-1} for the seven crystal systems are given in Annex B.[7]

For cubic system crystals, the index (hkl) of a reciprocal vector g_{hkl} possesses the same value as its corresponding crystalline direction [uvw]; that is, $h = u$, $k = v$, $l = w$. For non-cubic system crystals, it is necessary to convert the index (hkl) into the indices [uvw] of the crystalline direction or zone axis.

5.3.3 Result of the multiplicity factor

Owing to the multiplicity factor, the poles of the growth direction determined from several crystals may coincide with two or more points on the same stereogram. These may be considered as growth directions that are the equivalent direction of a family {HKL}; the indexes of the direction are <uvw>.

5.3.4 Repetition

Select another target crystal and repeat the procedure described in 5.2 and 5.3.

This cycle may be repeated several times depending on the required statistics of the analysis.

The example for determination of long-axis direction from Au nanocrystal is given in Annex D.

6 Uncertainty estimation

Uncertainty of the crystalline direction determination is affected mainly by two processes of image acquisition and measurement results by image analysis.

The uncertainty u_1 caused by process of acquiring micrographs and diffraction patterns involves three factors:

$\sigma_{L\lambda}$ is the uncertainty of the camera constant;

σ_R is the uncertainty of compensation of the rotation angle between image and corresponding diffraction pattern;

σ_ψ is the uncertainty of measurement results of the angle between the two zone axes.

According to Guide 98-3 (GUM:1995),[8] uncertainty is classified into the category of type A and B uncertainty (U_A and U_B). The uncertainty $\sigma_{L\lambda}$ and σ_R are classified into U_B.

In practice, the zone axis of diffraction patterns is basically determined by tilting the specimen holder and reversely parallel to the incident beam direction **B** approximately. Therefore, the uncertainty σ_ψ is classified into U_A and shall be calculated from the results by n times repeated measurements.

The uncertainty u_1 is represented as follows in Formula (5):

$$u_1 = \sqrt{\sigma_{L\lambda}^2 + \sigma_R^2 + \left(\sigma_\psi / \sqrt{n}\right)^2} \quad (5)$$

where

$\sigma_{L\lambda}$ is the uncertainty of the camera constant;

σ_R is the uncertainty of compensation of the rotation angle between image and corresponding diffraction pattern;

σ_ψ is the uncertainty of measurement results of the angle between the two zone axes;

n is total number of measurements of the uncertainty σ_ψ .

The uncertainty u_2 caused by measurement results on analysing micrographs contains three factors as follows:

σ_φ is the uncertainty of the angle φ between the coordinate vector $\boldsymbol{R}$ and the normal the projection direction of the crystal measured on the diffraction pattern;

$\sigma_{w\varphi}$ is the uncertainty of the angle measured by Wulff net on the stereographic projection;

σ_w is the uncertainty of the Wulff net as a measuring tool.

The uncertainty σ_φ and $\sigma_{w\varphi}$ are classified into U_A. These uncertainties shall be calculated from the results by m times and l times repeated measurements, respectively. The uncertainty σ_w is classified into U_B.

The uncertainty u_2 is represented by Formula (6):

$$u_2 = \sqrt{\left(\sigma_\varphi / \sqrt{m}\right)^2 + \left(\sigma_{w\varphi} / \sqrt{l}\right)^2 + \sigma_w^{\ 2}} \tag{6}$$

where

σ_φ is the uncertainty of the angle φ between the coordinate vector $\boldsymbol{R}$ and the normal the projection direction of the crystal measured on the diffraction pattern;

m is total number of measurements of the uncertainty σ_φ ;

$\sigma_{w\varphi}$ is the uncertainty of the angle measured by Wulff net on the stereographic projection;

l is total number of measurements of the uncertainty $\sigma_{w\varphi}$;

σ_w is the uncertainty of the Wulff net as a measuring tool.

The combined standard uncertainty u of the crystalline direction determination is represented by Formula (7):

$$u = \sqrt{u_1^{\ 2} + u_2^{\ 2}} \tag{7}$$

The expanded uncertainty U for the determination of crystalline direction is given by Formula (8).

$$U = k \times u \tag{8}$$

where k is the coverage factor.

NOTE For a confidence limit of approximately 95 %, k is set to 2 and for a confidence limit of approximately 99 %, k is set to 3.

The poles of the apparent growth direction experimentally determined from several specimens may be distributed around the pole of $(H\ K\ L)$.

7 Test report

An example of a test report is given in Annex C. The test report shall require information to be given on at least the following aspects of the test:

— type of the sample;

— typical image of the specimen morphology and diffraction patterns with scale bar;

— operating voltage of TEM;

— name of the software;

— crystalline direction determined.

Annex A
(informative)

Relationships of Miller notation and Miller-Bravais notation for hexagonal crystals

Diffraction patterns of a hexagonal crystal may be indexed either by Miller notation or equivalently by Miller-Bravais notation. For the Miller notation, three axes coordinate system are used, i.e. indices (*hkl*) for a plane, [*uvw*] for a direction. However, in the Miller-Bravais notation, the hexagonal system is described by four axes. The indices are (*hkil*) for a plane and are [*UVTW*] for a direction. The two notations may be converted to each other. The relations between the Miller and Miller-Bravais notation are given by Formulae (A.1) and (A.2):[7]

For indices of a plane (*hkl*) and (*hkil*):

$$i = -(h+k) \tag{A.1}$$

For indices of a direction [*uvw*] and [*UVTW*]:

$$\begin{cases} U = \frac{1}{3}(2u-v) \\ V = \frac{1}{3}(2v-u) \\ T = -\frac{1}{3}(u+v) = -(U+V) \\ W = w \end{cases} \tag{A.2}$$

Annex B
(informative)

Matrix G and G^{-1} for the crystal systems

The symbols a, b, and c denote vectors of the lattice parameters of a crystal; the length of each vector is a, b, and c respectively. The angle between b and c is α, the angle between a and c is β, and the angle between a and b is γ. See Table B.1.

Table B.1 — Matrix G and G^{-1} of the seven crystal systems[7]

Crystal-line system	G	G^{-1}
Cubic	$\begin{pmatrix} a^2 & 0 & 0 \\ 0 & a^2 & 0 \\ 0 & 0 & a^2 \end{pmatrix}$	$\begin{pmatrix} \frac{1}{a^2} & 0 & 0 \\ 0 & \frac{1}{a^2} & 0 \\ 0 & 0 & \frac{1}{a^2} \end{pmatrix}$
Hexag-onal	$\begin{pmatrix} a^2 & -\frac{a^2}{2} & 0 \\ -\frac{a^2}{2} & a^2 & 0 \\ 0 & 0 & c^2 \end{pmatrix}$	$\begin{pmatrix} \frac{4}{3a^2} & \frac{2}{3a^2} & 0 \\ \frac{2}{3a^2} & \frac{4}{3a^2} & 0 \\ 0 & 0 & \frac{1}{c^2} \end{pmatrix}$
Tetrag-onal	$\begin{pmatrix} a^2 & 0 & 0 \\ 0 & a^2 & 0 \\ 0 & 0 & c^2 \end{pmatrix}$	$\begin{pmatrix} \frac{1}{a^2} & 0 & 0 \\ 0 & \frac{1}{a^2} & 0 \\ 0 & 0 & \frac{1}{c^2} \end{pmatrix}$
Ortho-rhom-bic	$\begin{pmatrix} a^2 & 0 & 0 \\ 0 & b^2 & 0 \\ 0 & 0 & c^2 \end{pmatrix}$	$\begin{pmatrix} \frac{1}{a^2} & 0 & 0 \\ 0 & \frac{1}{b^2} & 0 \\ 0 & 0 & \frac{1}{c^2} \end{pmatrix}$
Rhom-bo-hedral	$\begin{pmatrix} a^2 & a^2\cdot\cos\alpha & a^2\cdot\cos\alpha \\ a^2\cdot\cos\alpha & a^2 & a^2\cdot\cos\alpha \\ a^2\cdot\cos\alpha & a^2\cdot\cos\alpha & a^2 \end{pmatrix}$	$\frac{1}{a^2\cdot B}\begin{pmatrix} \sin^2\alpha & \cos^2\alpha-\cos\alpha & \cos^2\alpha-\cos\alpha \\ \cos^2\alpha-\cos\alpha & \sin^2\alpha & \cos^2\alpha-\cos\alpha \\ \cos^2\alpha-\cos\alpha & \cos^2\alpha-\cos\alpha & \sin^2\alpha \end{pmatrix}$ $B=\sin^2\alpha-2\cos^2\alpha+2\cos^3\alpha$

Table B.1 *(continued)*

Crystal-line system	G	G^{-1}
Mono-clinic	$\begin{pmatrix} a^2 & 0 & a\cdot c\cdot\cos\beta \\ 0 & b^2 & 0 \\ a\cdot c\cdot\cos\beta & 0 & c^2 \end{pmatrix}$	$\begin{pmatrix} \frac{1}{a^2\cdot\sin^2\beta} & 0 & \frac{-\cos\beta}{a\cdot c\cdot\sin^2\beta} \\ 0 & \frac{1}{b^2} & 0 \\ \frac{-\cos\beta}{a\cdot c\cdot\sin^2\beta} & 0 & \frac{1}{c^2\cdot\sin^2\beta} \end{pmatrix}$
Triclin-ic	$\begin{pmatrix} a^2 & a\cdot b\cdot\cos\gamma & a\cdot c\cdot\cos\beta \\ a\cdot b\cdot\cos\gamma & b^2 & b\cdot c\cdot\cos\alpha \\ a\cdot c\cdot\cos\beta & b\cdot c\cdot\cos\alpha & c^2 \end{pmatrix}$	$\frac{1}{A}\begin{pmatrix} \frac{\sin^2\alpha}{a^2} & \frac{\cos\alpha\cdot\cos\beta-\cos\gamma}{ab} & \frac{\cos\alpha\cdot\cos\gamma-\cos\beta}{ac} \\ \frac{\cos\alpha\cdot\cos\beta-\cos\gamma}{ab} & \frac{\sin^2\beta}{b^2} & \frac{\cos\beta\cdot\cos\gamma-\cos\alpha}{bc} \\ \frac{\cos\alpha\cdot\cos\gamma-\cos\beta}{ac} & \frac{\cos\beta\cdot\cos\gamma-\cos\alpha}{bc} & \frac{\sin^2\gamma}{c^2} \end{pmatrix}$ $A=1-\cos^2\alpha-\cos^2\beta-\cos^2\gamma+2\cos\alpha\cdot\cos\beta\cdot\cos\gamma$

Annex C
(informative)

Example test report

A number of the testing report:

General information
Name of the testing laboratory:
Name of the client:
Address of the client:
Telephone number of the client:
Name of the TEM manufacturer and model:
TEM working condition
Operating voltage: (kV)
Image formation mode
Magnification of the micrograph field width
Diffraction mode:
Name and identification of the software:
International Standard reference:
Testing results
Name and serial number of the specimen:
Type of the specimen:
Typical micrograph of the specimen morphology (with scale bar):
Diffraction patterns:
Crystalline direction determined:
Authority
Operator name:
Testing date:
Name of person(s) authorizing:
Signature of person(s) authorizing:

Annex D
(informative)

Example for determination of long-axis direction from Au nanocrystal

D.1 General

The following example is given for the convenience of users to understand correctly the processing procedure indicated in this method.

D.2 Acquisition of micrographs and diffraction patterns from target crystal

Bright-field micrographs of an Au nanocrystal (target crystal) and corresponding diffraction patterns along three directions of the incident electron beam, respectively, are recorded digitized images by a digital camera and the photographic film by an image scanner. They are shown in Figure D.1 and Figure D.2, respectively. Information on Figures D.1 and D.2 is as follows.

— Specimen: Au nanocrystals, length 60 nm, width 17 nm.

— Crystal structure: fcc

— lattice parameter: a= 0,408 nm

— Observation apparatus: TEM equipped with a CCD camera (1k×1k pixels)

— Accelerating voltages: 200 kV

— Type of electron sources: lanthanum hexaboride gun (LaB_6)

— Camera length: L= 40 cm

— Information on diffraction pattern:

  — Tilting indicator provided by the goniometer to record the diffraction pattern Z_1: X_1=6,1, Y_1=10,7;

  — Tilting indicator provided by the goniometer to record the diffraction pattern Z_2: X_2=-19,0, Y_2=15,4;

  — Tilting indicator provided by the goniometer to record the diffraction pattern Z_3: X_3=-6,0, Y_3=-11,0.

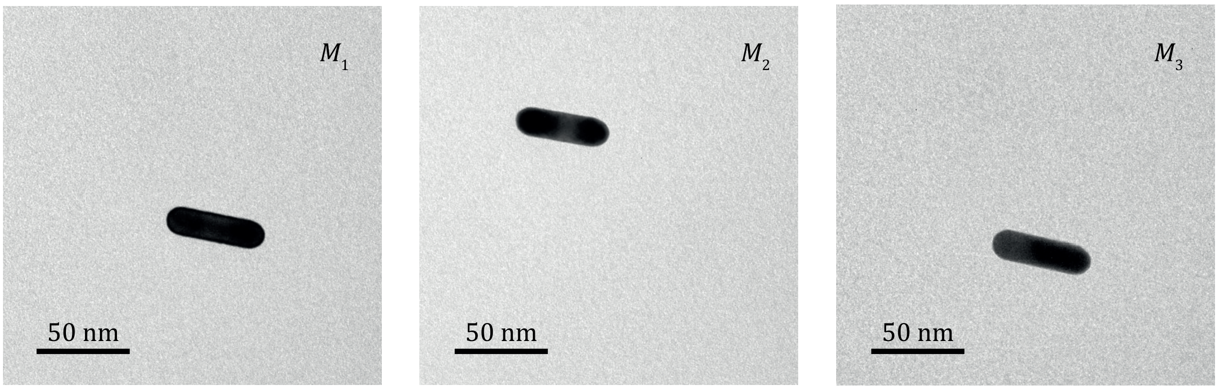


Figure D.1 — Bright field micrographs of Au nanocrystal along incident electron beam directions Z_1, Z_2 and Z_3, respectively

D.3 Indexing the diffraction patterns

The practical procedure for indexing diffraction patterns may refer to ISO 25498:2018, Clause 9. The indexing result of the diffraction patterns Z_1, Z_2 and Z_3 are also shown in Figure D.2, respectively. The obtained angle between two incident electron beam directions by tilting the specimen holder is compared with the theoretical value to check the angular relationship between the two zone axes. The angle between [310] and $[21\bar{1}]$ is 25,52°. The angle between $[21\bar{1}]$ and [110] is 28,80°. The theoretical value of the angle between [310] and $[21\bar{1}]$ is 25,35°, and the angle between $[21\bar{1}]$ and [110] is 30° for cubic system crystals. Therefore, the experimental values are consistent with the theoretical values within error. The indexing of the diffraction patterns has been confirmed.

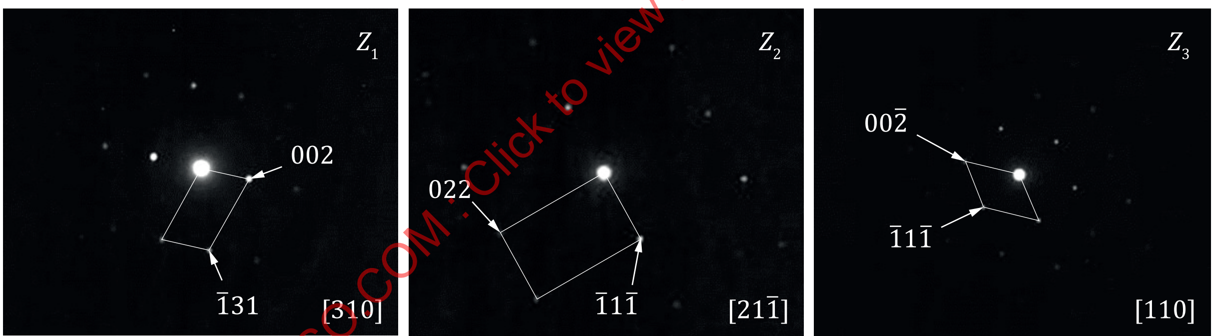


Figure D.2 — Indexed diffraction patterns from the Au nanocrystal corresponding micrograph M_1, M_2 and M_3, respectively, in Figure D.1

D.4 Defining the projection direction

After calibration of the rotation angle between the bright field micrograph and its corresponding diffraction pattern, align the diffraction pattern with its bright field micrograph carefully. Draw the projection direction A_1, A_2 and A_3 on the diffraction patterns and their bright field micrographs, respectively. Then draw the normal line of the projection direction N_1, N_2, and N_3 on the diffraction patterns, respectively, as shown in Figure D.3.

Mark the indices of the diffracted spot, which is closest to the normal N_1 (that is $\bar{2}60$) on the diffraction pattern Z_1. Measure the angle φ_1 (that is 0) between the normal N_1 and the coordinate vector of diffraction spot $R_{\bar{2}60}$ (equivalent to $R_{\bar{1}30}$), in the present case angle φ_1 is nearly zero. A similar operation as described in previous step, mark the indices of diffracted spot, which is closest to the normal N_2 (that is $\bar{2}40$, equivalent to $\bar{1}20$) on the diffraction pattern Z_2. Mark the indices of diffracted spot, which is closest to the normal N_3