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Second edition
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Microbeam analysis — Analytical electron microscopy — Selected area electron diffraction analysis using a transmission electron microscope

Analyse par microfaisceaux — Microscopie électronique analytique — Analyse par diffraction par sélection d'aire au moyen d'un microscope électronique en transmission

Reference number
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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 documents 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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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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 25498:2010), which has been technically revised.

The main changes to the previous edition are as follows:

— the foreword has been revised;

— the introduction has been revised;

— the scope has been revised;

— the figure of Ewald construction has been deleted;

— the terms and definition of terminological entries 3.1, 3.2, 3.10, 3.11, 3.12, 3.13 and 3.14 have been added;

— the subclause 4.1 has been added;

— Clause 5 has been revised;

— Clauses 4, 6, 7, and 10 and subclauses 8.1.5, 8.1.6, 8.2.1, 8.2.2, 8.2.4, 8.2.7, 8.2.8, 8.2.11 and 9.1.2 have been editorially revised;

— the subclause 9.2.5 has been added;

— all formulae have been renumbered;

— Annex A has been revised;

— the subclause B.1 has been revised;

— the figures have been modified;

— the bibliography has been updated.

Introduction

Electron diffraction techniques are widely used in transmission electron microscopy (TEM) studies. Applications include phase identification, determination of the crystallographic lattice type and lattice parameters, crystal orientation and the orientation relationship between two phases, phase transformations, habit planes and defects, twins and interfaces, as well as studies of preferred crystal orientations (texture). While several complementary techniques have been developed, for example microdiffraction, convergent beam diffraction and reflected diffraction, the selected area electron diffraction (SAED) technique is the most frequently employed.

This technique allows direct analysis of small areas on thin specimens from a variety of crystalline substances. It is routinely performed on most of TEM in the world. The SAED is also a supplementary technique for acquisition of high resolution images, microdiffraction or convergent beam diffraction studies. The information generated is widely applied in the studies for the development of new materials, improving structure and/or properties of various materials as well as for inspection and quality control purpose.

The basic principle of the SAED method is described in this document. The experimental procedure for the acquirement of SAED patterns, indexing of the diffraction patterns and determination of the diffraction constant are specified. ISO 25498 is intended for use or reference as technical regulation for transmission electron microscopy.

Microbeam analysis — Analytical electron microscopy — Selected area electron diffraction analysis using a transmission electron microscope

1 Scope

This document specifies the method of selected area electron diffraction (SAED) analysis using a transmission electron microscope (TEM) to analyse thin crystalline specimens. This document applies to test areas of micrometres and sub-micrometres in size. The minimum diameter of the selected area in a specimen which can be analysed by this method is restricted by the spherical aberration coefficient of the objective lens of the microscope and approaches several hundred nanometres for a modern TEM.

When the size of an analysed specimen area is smaller than that restriction, this document can also be used for the analysis procedure. But, because of the effect of spherical aberration, some of the diffraction information in the pattern can be generated from outside of the area defined by the selected area aperture. In such cases, the use of microdiffraction (nano-beam diffraction) or convergent beam electron diffraction, where available, might be preferred.

This document is applicable to the acquisition of SAED patterns from crystalline specimens, indexing the patterns and calibration of the diffraction constant.

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*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
Miller index
notation system for crystallographic planes and directions in crystals, in which a set of lattice planes or directions is described by three axes coordinate

3.2
Miller-Bravais index
notation system for crystallographic planes and directions in hexagonal crystals, in which a set of lattice planes or directions is described by four axes coordinate

3.3
(h k l)
Miller indices (3.1) of a specific set of crystallographic planes

3.4
{*hkl*}
Miller indices (3.1) which denote a family of crystallographic planes

3.5
[*uvw*]
Miller indices (3.1) of a specific crystallographic direction or a zone axis

3.6
interplanar spacing
d_{hkl}
perpendicular distance between consecutive planes of the crystallographic plane set *(h k l)* (3.3)

3.7
(*uvw*)*
notation for a set of planes in the reciprocal lattice

Note 1 to entry: The normal of the reciprocal plane *(uvw)** is parallel to the crystallographic zone axis *[uvw]* (3.5).

3.8
reciprocal vector
g_{hkl}
vector in the reciprocal lattice

Note 1 to entry: The reciprocal vector, $g_{hkl,}$ is normal to the crystallographic plane *(h k l)* (3.3) with its magnitude inversely proportional to interplanar spacing d_{hkl} (3.6).

3.9
***R* vector**
R_{hkl}
vector from centre, *000* (the origin), to the diffraction spot, *hkl*, in a diffraction pattern

Note 1 to entry: See Figure 1.

3.10
camera length
L
effective distance between the specimen and the plane where diffraction pattern is formed

[SOURCE: ISO 15932:2010, 3.7]

3.11
camera constant
$L\lambda$
product of the wavelength of the incident electron wave and *camera length* (3.10)

Note 1 to entry: Because of the small Bragg angle, the Bragg condition can be given in the first-order approximation, $R_{hkl} \cdot d_{hkl} \cong L\lambda$, where d_{hkl} is the interplanar spacing of plane, *(hkl)*, (3.3) and R_{hkl} (3.9) is the distance of the diffraction spot, *hkl*, from the incident beam.

[SOURCE: ISO 15932:2010, 3.8, modified]

3.12
bright field image
image formed using only the non-scattered beam, selected by observation of the back focal plane of the objective lens and using the objective aperture to cut out all diffracted beams

[SOURCE: ISO 15932:2010, 5.6]

3.13
dark field image
image formed by a diffracted beam only by using the objective aperture for selection or by collecting the diffracted beam with an annular dark-field detector

[SOURCE: ISO 15932:2010, 5.6]

3.14
energy-dispersive X-ray spectroscopy
EDS
analytical technique which enables the elemental analysis or chemical characterization of a specimen by analysing characteristic X-ray emitted by the matter in response to electron irradiation

[SOURCE: ISO 15932:2010, 6.6]

3.15
eucentric position
specimen position at which the image exhibits minimal lateral motion resulting from specimen tilting

4 Principle

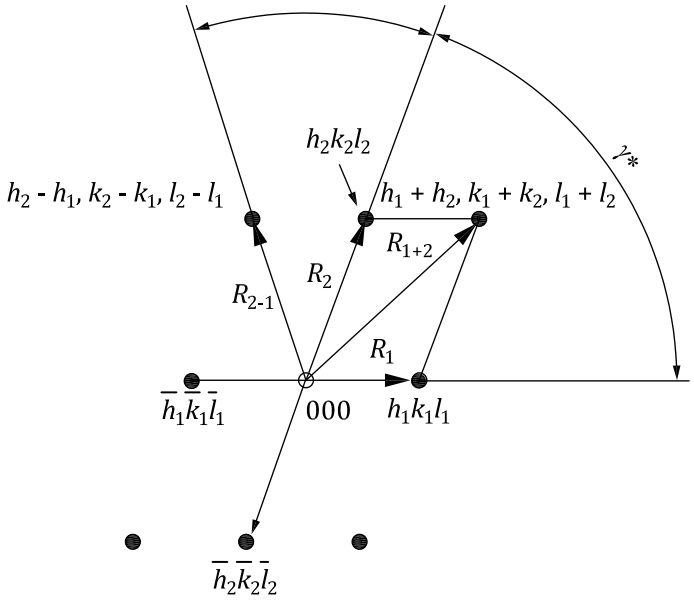
4.1 General

When an energetic electron beam is incident upon a thin crystallographic specimen in a transmission electron microscope, a diffraction pattern will be produced in the back focal plane of the objective lens. This pattern is magnified by the intermediate and projector lenses, then displayed on a viewing screen and recorded (see Reference [3]). This pattern can also be displayed on a monitor if the TEM is equipped with a digital camera system.

4.2 Spot diffraction pattern

The diffraction pattern of a single crystal appears as an array of “spots”, the basic unit of which is characterized by a parallelogram. A schematic illustration of a spot diffraction pattern is shown in Figure 1. Each spot corresponds to diffraction from a specific set of crystal lattice planes in the specimen, denoted by Miller indices (*hkl*). The vector, R_{hkl}, is defined by the position of the diffracted spot, *hkl*, relative to position on the pattern corresponding to the transmitted beam, i.e. the centre-spot, *000*, of the pattern. It is parallel to the normal of the reflecting plane, (*hkl*). The magnitude of R_{hkl} is inversely proportional to the interplanar spacing, d_{hkl}, of the diffracting plane, (*hkl*) (see References [4] to [9]). In the context of this document, vectors $R_{h_1k_1l_1}$, $R_{h_2k_2l_2}$, $\left(R_{h_2k_2l_2} - R_{h_1k_1l_1}\right)$ and $\left(R_{h_2k_2l_2} + R_{h_1k_1l_1}\right)$ are simplified as R_1, R_2, R_{2-1} and R_{1+2}, respectively. The included angle between vectors, R_1 and R_2, is denoted by γ*. The basic parallelogram is defined by R_1 and R_2, where they are the shortest and next shortest in the pattern respectively and not along a common line. The spot, $h_2k_2l_2$, is positioned anticlockwise around the centre spot relative to spot, $h_1k_1l_1$.

Because the centre-spot is often very bright, it is often difficult to determine the exact centre of the pattern. Therefore, a practical procedure is to establish the magnitude of $\left|R_{hkl}\right|$ by measuring the distance between the spots, *hkl* and $\bar{h}\bar{k}\bar{l}$ on the diffraction pattern and dividing by two, i.e. $\left|R_{hkl}\right| = \frac{1}{2}\left(\left|R_{hkl}\right| + \left|R_{\bar{h}\bar{k}\bar{l}}\right|\right)$. Figure 2 shows an example of the SAED pattern where the magnitude of R_1, R_2 and R_{2-1} is obtained from $\frac{1}{2}\left(R_1 + \bar{R}_1\right)$, $\frac{1}{2}\left(R_2 + \bar{R}_2\right)$ and $\frac{1}{2}\left(R_{2-1} + \bar{R}_{2-1}\right)$ respectively.



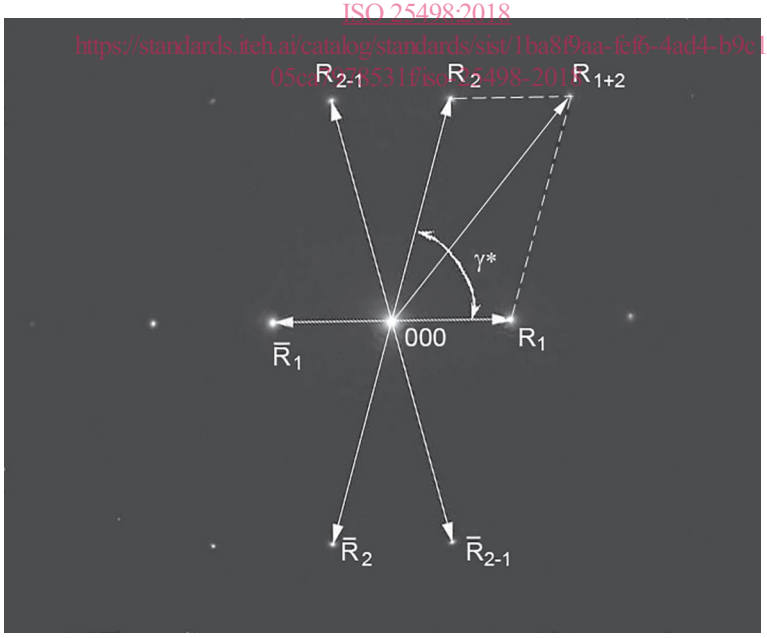
Key

R_1 vector from *000* to spot, $h_1k_1l_1$, the shortest vector in the diffraction pattern

R_2 vector from *000* to spot, $h_2k_2l_2$, the next shortest vector

NOTE The basic parallelogram is constituted by diffraction spots, $h_1k_1l_1$, $h_2k_2l_2$, (h_1+h$_2$, k_1+k_2, l_1+l_2), and central spot, *000*.

Figure 1 — Schematic spot diffraction pattern from a single crystal



Key

R_1 vector from *000* to spot, $h_1k_1l_1$, the shortest vector in the diffraction pattern

R_2 vector from *000* to spot, $h_2k_2l_2$, the next shortest vector

NOTE The basic parallelogram is constituted by R_1 and R_2.

Figure 2 — Example of SAED spot pattern

The relationship between the interplanar spacing, d_{hkl}, and the magnitude, R_{hkl}, for a reflecting plane, (hkl), can be approximately expressed as shown in Formula (1) (see References [7] and [8]):

$$L\lambda = R_{hkl} \times d_{hkl}\left[1-\frac{3}{8}\left(R_{hkl}/L\right)^2\right] = R_{hkl} \times d_{hkl}\left(1-\Delta\right) \tag{1}$$

where

Δ is equal to $\frac{3}{8}\left(R_{hkl}/L\right)^2$;

L is the diffraction camera length and equal to $f_o \times M_i \times M_p$;

where

f_o is the focal length, in millimetres, of the objective lens in the microscope;

M_i is the magnification of the intermediate lens;

M_p is the magnification of the projector lenses;

$L\lambda$ is the camera constant (or diffraction constant) of the transmission electron microscope operating under the particular set of conditions. This parameter can be determined from the diffraction pattern of a crystalline specimen of known lattice parameters (see 8.3);

λ is the wavelength, in nanometres, of the incident electron beam which is dependent upon the accelerating voltage and can be given by Formula (2) (see Reference [4]):

$$\lambda(\text{nm}) = \frac{1{,}226}{\sqrt{V\left(1+0{,}978\,8\times10^{-6}V\right)}} \tag{2}$$

where V is the accelerating voltage, in volts, of the TEM; the factor in parenthesis is the relativistic correction.

For most work using a TEM, the value of Δ in Formula (1) is usually smaller than 0,1 % and, hence, a more simplified Formula (3) may be used, namely

$$R_{hkl} \cdot d_{hkl} \cong L\lambda \tag{3}$$

For the derivation of the above equation, refer to the textbooks (see References [4] to [9]).

The use of Formula (3) requires measuring the length of R_{hkl}. Since, as mentioned earlier, the location of the pattern centre may not be easily determined; it is recommended that the distance measurement taken, $2R_{h_1k_1l_1}$, be from the $h_1k_1l_1$ diffracted spot to the $\bar{h}_1\bar{k}_1\bar{l}_1$ spot on the pattern. This is equivalent to a diameter measurement on the ring pattern from a polycrystalline specimen. To obtain the interplanar information, the measured distance, $2R_{h_1k_1l_1}$, is halved and Formula (3) applied.

If the camera constant is known, the interplanar spacing, d_{hkl}, of plane, (hkl), can be calculated. The included angle between any two vectors, $R_{h_1k_1l_1}$ and $R_{h_2k_2l_2}$, can also be measured on the diffraction pattern. This is equal to the angle between the corresponding crystallographic planes, $(h_1k_1l_1)$ and $(h_2k_2l_2)$.

Since diffraction data from a single pattern will provide information on a limited number of the possible diffracting planes in a specimen area, it is necessary to acquire additional diffraction patterns from the same area (or from different grains/particles of the same phase). This requires either the tilting of the specimen or the availability of differently oriented grains or particles of the same phase.

Acquire a second diffraction pattern from another zone axis from the same area by tilting (or tilting and rotating) the specimen so that the two patterns contain a common spot row (see 8.2.10 and Figure 5).

Index the diffracted spots, and then select three non-planar spots in the two patterns to constitute a reciprocal lattice, which, if the spots correspond to low values of Miller indices, may define the primitive unit cell of the crystal lattice. Therefore, crystal lattice parameters can be determined and the orientation of the grain or particle in the thin specimen can also be calculated.

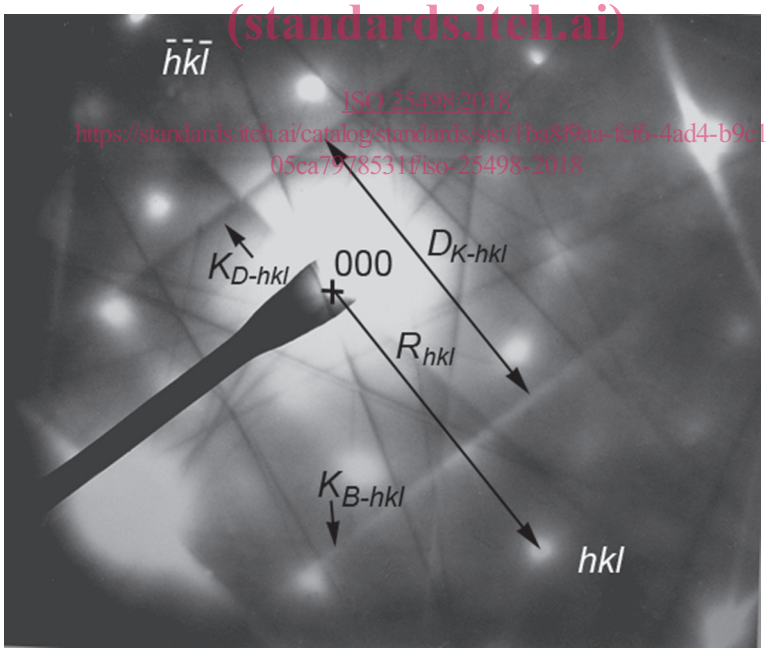
4.3 Kikuchi pattern

When a specimen area is nearly perfect but not thin enough, Kikuchi lines may occur. They arise from electrons scattered inelastically through a small angle and suffering only a very small energy loss being scattered again, this time elastically. This process leads to local variations of the background intensity in the diffraction pattern and the appearance of Kikuchi lines.

The Kikuchi patterns consist of pairs of parallel bright and dark lines, which are parallel to the projection of the corresponding reflecting plane, (*hkl*). The bright (excess) line and dark (defect) line in the Kikuchi pattern are denoted by K_{B-hkl} and K_{D-hkl}, respectively. Therefore, the line pair, K_{B-hkl} and K_{D-hkl}, will be perpendicular to the vector, R_{hkl} from the corresponding crystallographic plane *(hkl)*. Namely they are perpendicular to the reciprocal vector, g_{hkl}, of the plane, (*hkl*).

An example of a Kikuchi pattern is given in Figure 3, where the bright line, K_{B-hkl}, and dark line, K_{D-hkl}, pair is superimposed on the spot pattern. The perpendicular distance, D_{K-hkl}, between the line pair, K_{B-hkl} and K_{D-hkl}, is related to the interplanar spacing, d_{hkl}, and camera constant, $L\lambda$, by Formula (4).

$$D_{K-hkl} \cdot d_{hkl} \cong L\lambda \tag{4}$$



Key

K_{B-hkl}	bright line of Kikuchi pair
K_{D-hkl}	dark line of Kikuchi pair
D_{K-hkl}	distance between the line pair K_{B-hkl} and K_{D-hkl}
+	centre of the direct beam

Figure 3 — Kikuchi pattern from a steel specimen

The distance between the two Kikuchi lines equals to the distance between the diffraction spot, *hkl*, and the central spot, *000*. The angles between intersecting Kikuchi pairs are the same as the angles

between their corresponding diffraction spots, and can be measured accurately. These angles are also equal to the angles between the relevant crystallographic planes.

When the specimen is tilted, the diffraction spots only gradually change the brightness, faint or increase the intensity, but their positions are almost at the same place. Instead, Kikuchi lines are sensitive to the tilting. Their movement is significant on the viewing screen. Hence, specimen tilting can be guided by Kikuchi map from one zone axes to another one. The Kikuchi patterns present the real crystal symmetry of the specimen. They can also be used in establishing crystal orientation with a very high accuracy (see References [5] and [9]).

The problem is that Kikuchi patterns cannot always be observed in all of the specimens. In most cases, the SAED studies rely mainly on the spot patterns, though they are not as accurate as Kikuchi patterns.

4.4 Polycrystalline specimen

For randomly oriented aggregates of polycrystals, the diffraction pattern is comprised of a series of concentric rings centred on the spot, *000,* of the direct beam. An example of the pattern from polycrystalline gold (Au) specimen is given in Figure 4. Each diffracted ring arises from the diffraction beams from differently oriented crystallographic planes of the form, {*hkl*}; each of these having an identical interplanar spacing. From the diameter of each diffraction ring, the corresponding interplanar spacing, d_{hkl}, can be calculated using Formula (3). Indices of the diffraction rings can be ascribed and then the lattice parameters can also be determined. For the method of indexing ring patterns, refer to that used in X-ray powder diffraction (see Reference [9]).

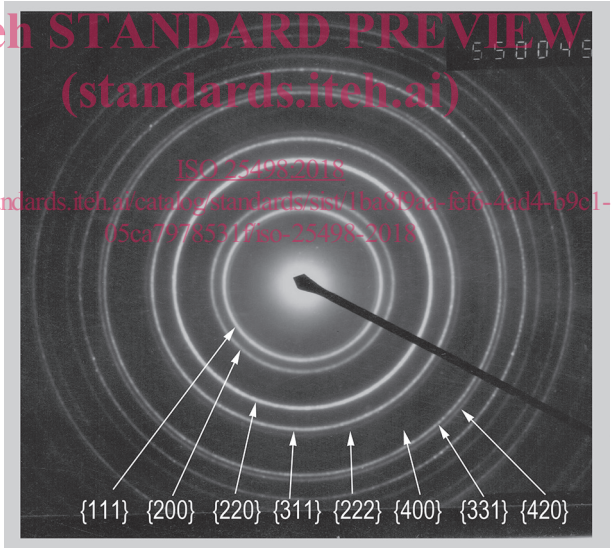


Figure 4 — Diffraction ring pattern with indices from a polycrystalline Au specimen

5 Reference materials

A reference specimen is required for determining the diffraction constant, $L\lambda$, of the microscope in electron diffraction studies. In principle, any thin crystalline foil or powder could be considered as the reference specimen, provided its crystalline structure and lattice parameters have been acquired accurately and they are certified and stable under irradiation of the electron beam. It should be ensured that the reference material, which is as thin as electrons can penetrate through it, has the same crystallographic properties as the bulk material. In addition, a number of sharp diffraction rings or spots with known indices can be observed. The thickness of the crystal foil or powder grain size should be consistent with the beam energy and the quality of the diffraction pattern so that clear diffraction patterns can be observed (when it is too thick, the pattern will lack sharpness).