INTERNATIONAL STANDARD

ISO 25498

First edition
2010-06-01

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
ISO 25498:2010(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 25498 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 3, *Analytical electron microscopy*.

Introduction

Electron diffraction techniques are widely used in transmission electron microscopy (TEM) studies. Applications include phase identification, determination of the crystalline 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) etc. While several complementary techniques have been developed, e.g. microdiffraction, convergent beam diffraction and reflected diffraction, selected-area electron diffraction (SAED) is the most frequently employed. Selected-area electron diffraction allows direct analysis of small areas of the sample (fine layers, grains, precipitates, etc.) and is routinely performed on thin specimens of a variety of crystalline materials. The SAED is also a supplementary technique for acquisition of high resolution images, microdiffraction or convergent beam diffraction studies. The information generated is widely used in the studies of structure/property relationships as well as for inspection and quality control purposes.

This International Standard explains the mechanism of the diffraction pattern formation, the practical aspects of SAED operation and the indexing of the patterns.

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

1 Scope

This International Standard specifies the method of selected-area electron diffraction (SAED) analysis using a transmission electron microscope (TEM) to analyse micrometer and sub-micrometer sized areas of thin crystalline specimens. Such specimens can be obtained in the form of thin sections from a variety of metallic and non-metallic materials, as well as fine powders, or alternatively by the use of extraction replicas. The minimum diameter of the selected area in a specimen which can be analysed by this method depends on the spherical aberration coefficient of the objective lens of the microscope and approaches 0,5 µm for a modern TEM.

When the diameter of an analysed specimen area is smaller than 0,5 µm, the analysis procedure can also be referred to this International Standard 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 or convergent beam electron diffraction, where available, might be preferred.

The success of the selected-area electron diffraction method relies on the validity of indexing the diffraction patterns arising, irrespective of which axis in the specimen lies parallel to the incident electron beam. Such analysis is therefore aided by specimen tilt and rotation facilities.

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

2 Normative references

The following referenced documents are indispensable for the application 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, definitions and symbols

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

3.1
$(h\,k\,l)$
Miller indices of a specific set of crystalline planes

3.2
$\{h\,k\,l\}$
Miller indices, which denote a family of crystalline planes

3.3
$[u\,v\,w]$
Miller indices of a specific crystalline direction or a zone axis

3.4
$(u\,v\,w)$
Miller indices, which denote a family of crystalline directions

3.5
interplanar spacing
d_{hkl}
perpendicular distance between consecutive planes of the crystalline plane set $(h\,k\,l)$

3.6
$(u\,v\,w)^*$
indices of a plane in the reciprocal lattice

NOTE The normal of the reciprocal plane $(u\,v\,w)^*$ is parallel to the crystalline zone axis $[u\,v\,w]$.

3.7
reciprocal vector
$\boldsymbol{g_{hkl}}$
vector in the reciprocal lattice

NOTE The reciprocal vector $\boldsymbol{g_{hkl}}$ is normal to the crystalline plane $(h\,k\,l)$ with its magnitude inversely proportional to interplanar spacing d_{hkl}.

3.8
$\boldsymbol{R_{hkl}}$
vector from centre 000 (the origin) to the diffraction spot $h\,k\,l$ in a diffraction pattern

See Figure 1.

3.9
eucentric position
specimen position at which the image exhibits minimal lateral motion resulting from specimen tilting

4 Principle

When an energetic electron beam is incident upon a thin crystalline 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, and displayed on a viewing screen. This pattern can also be displayed on a monitor if the TEM is equipped with a TV or charge-coupled device (CCD) camera system.

4.1 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 $(h\,k\,l)$. The vector $\boldsymbol{R_{hkl}}$ is defined by the position of the diffracted spot $h\,k\,l$ 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 $(h\,k\,l)$. The magnitude of R_{hkl} is inversely proportional to the interplanar spacing d_{hkl} of the diffracting plane $(h\,k\,l)$ (References [1], [2], [3] and [4] in the Bibliography). In the context of this International Standard, vectors $\boldsymbol{R_{h_1k_1l_1}}$, $\boldsymbol{R_{h_2k_2l_2}}$, $(\boldsymbol{R_{h_2k_2l_2}} - \boldsymbol{R_{h_1k_1l_1}})$ and $(\boldsymbol{R_{h_1k_1l_1}} + \boldsymbol{R_{h_2k_2l_2}})$ are simplified as $\boldsymbol{R_1}$, $\boldsymbol{R_2}$, $\boldsymbol{R_{2-1}}$ and $\boldsymbol{R_{1+2}}$, respectively. The included angle between vectors $\boldsymbol{R_1}$ and $\boldsymbol{R_2}$ is denoted by γ^*.

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 $|R_{hkl}|$ by measuring the distance between the spots $h\,k\,l$ and $\bar{h}\,\bar{k}\,\bar{l}$ on the diffraction pattern and dividing by two, i.e. $|R_{hkl}| = \frac{1}{2}(|R_{hkl}| + |R_{\bar{h}\,\bar{k}\,\bar{l}}|)$. Figure 2 shows an example of the SAED pattern where the magnitude of R_1, R_2 and R_{2-1} is obtained from $\frac{1}{2}(R_1 + \bar{R}_1)$, $\frac{1}{2}(R_2 + \bar{R}_2)$ and $\frac{1}{2}(R_{2-1} + \bar{R}_{2-1})$, respectively.

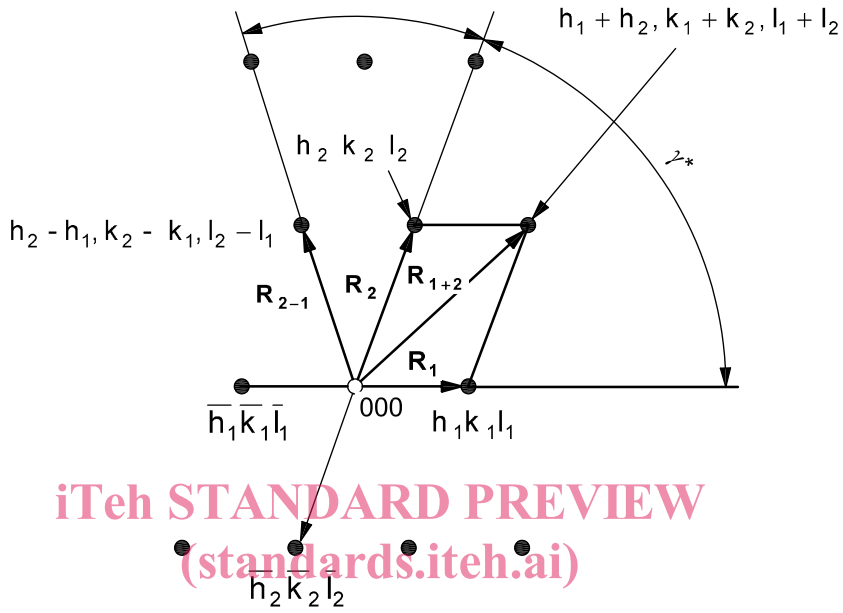


Figure 1 — Schematic spot diffraction pattern from a single crystal, the basic parallelogram constituted by the diffraction spots $h_1k_1l_1$, $h_2k_2l_2$, $(h_1 + h_2, k_1 + k_2, l_1 + l_2)$ and central spot 000

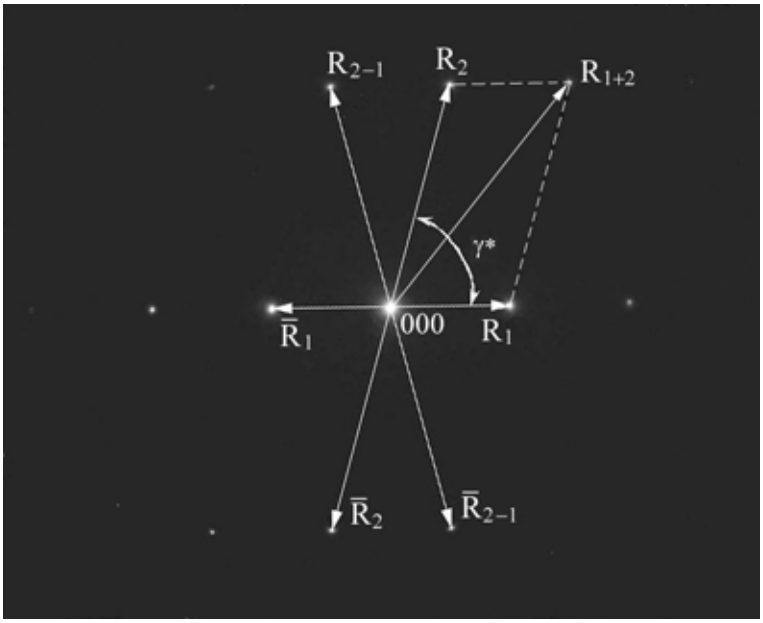


Figure 2 — Example of SAED spot pattern showing the basic parallelogram constituted by R_1 and R_2

The relationship between the interplanar spacing d_{hkl} and the magnitude R_{hkl} for a reflecting plane $(h\,k\,l)$ can be approximately expressed as (Reference [4] in the Bibliography)

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

where

$L = f_o \times M_i \times M_p$ is the diffraction camera length,

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,

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

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

where V is the accelerating voltage, in volts, of the TEM,

$L\lambda$ is the diffraction constant (or camera 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 (refer to 8.3).

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

$$R_{hkl} \times d_{hkl} = L\lambda \tag{3}$$

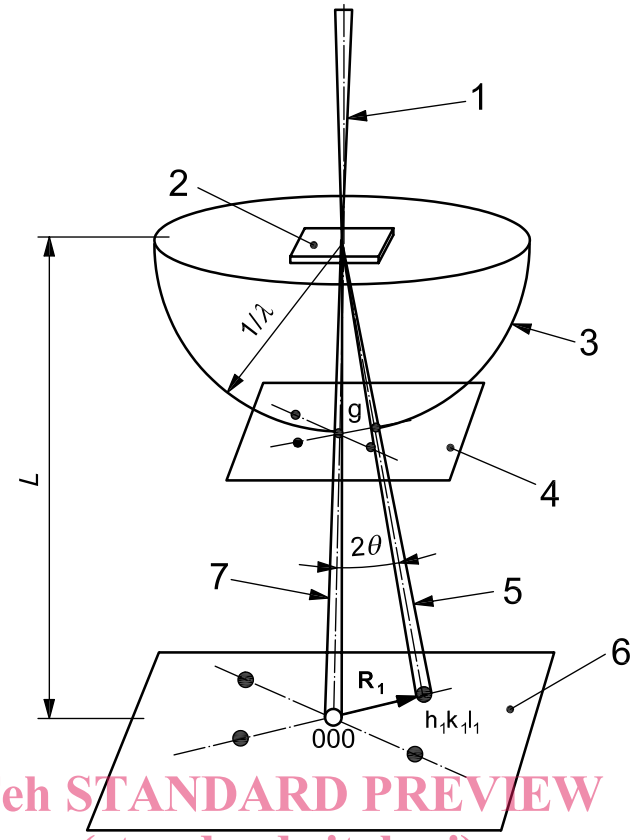
This relation can be understood through the Ewald sphere construction, which is illustrated in Figure 3. For the derivation of the above equation, refer to the textbooks (References [2] to [6] in the Bibliography).

The use of Equation (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, $2\,\boldsymbol{R}_{h_1k_1l_1}$, 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 $2\,\boldsymbol{R}_{h_1k_1l_1}$ is halved and Equation (3) applied.

If the camera constant is known, the interplanar spacing d_{hkl} of plane $(h\,k\,l)$ can be calculated. The included angle between any two vectors $\boldsymbol{R}_{h_1k_1l_1}$ and $\boldsymbol{R}_{h_2k_2l_2}$ can also be measured on the diffraction pattern. This is equal to the angle between the corresponding crystalline 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 the specimen so that the two patterns contain a common spot row (also see 8.2.10). 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.



Key

1 incident beam
2 specimen
3 Ewald sphere
4 reciprocal plane
5 diffracted beam
6 diffraction pattern
7 transmitted beam

Figure 3 — Ewald sphere construction illustrating the diffraction geometry in TEM

4.2 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 planes. 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 $\boldsymbol{R_{hkl}}$ from the corresponding crystalline plane (hkl). An example of a Kikuchi pattern is given in Figure 4, 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 Equation (4).

$$D_{K\text{-}hkl} \times d_{hkl} = L\lambda \tag{4}$$

The angles between different Kikuchi line pairs are also equal to the angles between the relevant crystalline planes. The Kikuchi patterns present the real crystal symmetry of the specimen. They are a very useful aid in tilting the specimen from one zone axis to another and also in establishing crystal orientation with very high accuracy (References [6] and [7] in the Bibliography).

Generally, when Kikuchi lines are visible, the specimen-tilting method is preferred to acquire diffraction patterns with different zone axes from the same area.

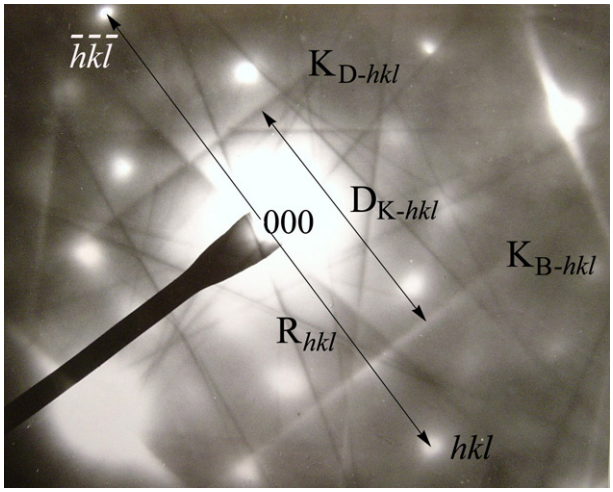


Figure 4 — Kikuchi pattern from a steel specimen, D_{K-hkl}: the distance between the line pair K_{B-hkl} (bright line) and K_{D-hkl} (dark line)

4.3 Polycrystalline specimen

The diffraction pattern from a polycrystalline specimen is comprised of a series of concentric rings centred on the transmitted spot 000. An example of the pattern from a polycrystalline gold (Au) specimen is given in Figure 5. Each diffracted ring arises from the diffraction beams from differently oriented crystalline planes of the form $\{h\,k\,l\}$; 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 Equation (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 (Reference [8] in the Bibliography).

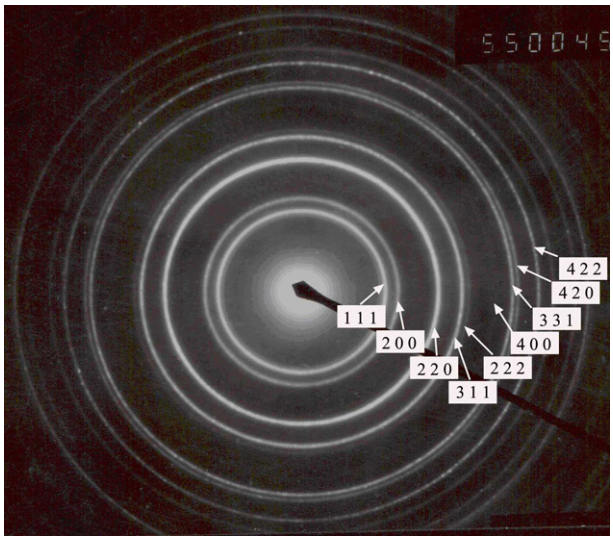


Figure 5 — Diffraction ring pattern with indices from a polycrystalline Au specimen