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International Standard

# ISO/FDIS 25498

# 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*

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# Foreword

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

This third edition cancels and replaces the second edition (ISO 25498:2018), which has been technically revised.

The main changes are as follows:

— Scope has been revised;

— ISO/IEC 17025 has been moved from normative references to bibliography;

— Figure 1 has been replaced;

— Subclause 6.3 has been deleted;

— Subclause 8.3.6 has been deleted, the content of 8.3.6 has been moved to 8.3.2;

— Subclause 9.2.5 has been added and the following subclause has been renumbered;

— Clause 11 has been revised, 11.1,11.2,11.3 and 11.4 have been added;

— Subclauses B.4.1 and B.4.2 have been added;

— Bibliography has been updated and ISO/IEC Guide 98-3 (GUM:1995) has been added.

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

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, nano-diffraction, 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 TEMs 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 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 for 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 hundreds of nanometres for a modern TEM.

When the size of an analysed specimen area is smaller than the spherical aberration coefficient restriction, this document can also be used for the analysis procedure. However, because of the effect of spherical aberration and deviation of the specimen height position, 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 diffraction, where available, can be preferred.

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



## 2 Normative references

There are no normative references in this document.

## 3 Terms, definitions and abbreviations



For the purposes of this document, the following terms and definitions 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 Terms and definitions

**3.1.1**
**Miller notation**
indexing system for crystallographic planes and directions in crystals, in which a set of lattice planes or directions is described by three axes coordinate

**3.1.2**
**Miller-Bravais notation**
indexing system for crystallographic planes and directions in hexagonal crystals, in which a set of lattice planes or directions is described by four axes coordinate

**3.1.3**
**interplanar spacing**
$d_{hkl}$
perpendicular distance between consecutive planes of the crystallographic plane set *(hkl)*

**3.1.4**
**reciprocal vector**
$\boldsymbol{g_{hkl}}$
vector in the reciprocal lattice

Note 1 to entry: The reciprocal vector, $\boldsymbol{g_{hkl}}$, is normal to the crystallographic plane *(h k l)* with its magnitude inversely proportional to *interplanar spacing* $d_{hkl}$ (3.1.3).

**3.1.5**
***R* vector**
$\boldsymbol{R_{hkl}}$
coordinate vector from the direct beam, 000, to a diffraction spot, *hkl*, in a zone diffraction pattern

Note 1 to entry: See Figure 1.

**3.1.6**
**camera length**
*L*
effective distance from the specimen to the screen or recording device in a transmission electron microscope in diffraction mode

**3.1.7**
**camera constant**
diffraction constant
$L\lambda$
product of the wavelength of the incident electron wave and *camera length* (3.1.6)

[SOURCE: ISO 15932:2013, 3.7.1]



**3.1.8**
**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 15932:2013, 5.5]



**3.1.9**
**dark field image**
image formed by a diffracted beam only by using the objective aperture for selection or by collecting the diffracted beams with an annular dark-field detector

[SOURCE: ISO 15932:2013, 5.6]

**3.1.10**
**energy-dispersive X-ray spectrometry**
**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 15932:2013, 6.6]

**3.1.11**
**eucentric position**
specimen position at which the image exhibits minimal lateral motion resulting from specimen tilting

**3.1.12**
**selected area (selector) aperture**
moveable diaphragm that is used to select only radiation scattered from a specific area of the specimen to contribute to the formation of a diffraction pattern

[SOURCE: ISO 15932:2013, 3.2.3.5]

**3.1.13**
**Bragg angle**
$\theta_B$
angle between the incident beam and the atomic planes, at which diffraction takes place

## 3.2 Abbreviated terms and symbols

| | |
|---|---|
| BCC | body-centred cubic structure |
| FCC | face-centred cubic structure |
| HCP | hexagonal close-packed structure |
| SAED | selected area electron diffraction |
| TEM | transmission electron microscope |
| (*hkl*) | *Miller indices* of a specific set of crystallographic planes |
| {*hkl*} | *Miller indices* which denote a family of crystallographic planes |
| [*uvw*] | *Miller indices* of a specific crystallographic direction or a zone axis |
| (*uvw*)* | Notation for a set of planes in the reciprocal lattice |
| | NOTE The normal of the reciprocal plane *(uvw)** is parallel to the crystallographic zone axis *[uvw]* |

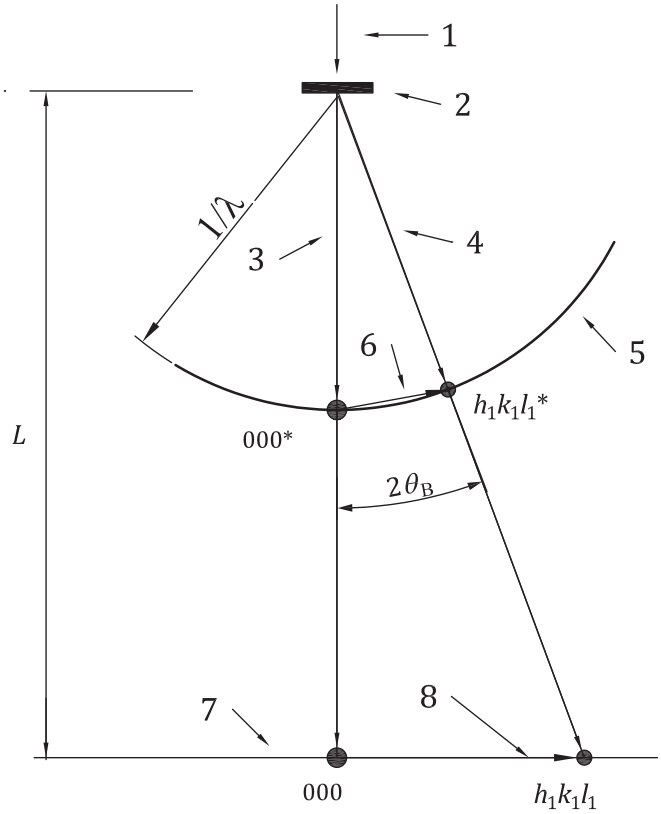


# 4 Principle

## 4.1 General

When an energetic electron beam is incident upon a thin crystal 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], [4], [5]). This pattern can also be displayed on a monitor if the TEM is equipped with a digital camera system.

The geometric relationship of the parameters for selected area electron diffraction (SAED) technique can be understood through the Ewald sphere construction, which is illustrated in Figure 1.




**Key**

1 incident beam
2 specimen
3 direct beam
4 diffracted beam
5 Ewald sphere
6 reciprocal vector $\boldsymbol{g}_{hkl}$
7 diffraction pattern
8 $\boldsymbol{R}_{hkl}$ vector
$L$ is the diffraction camera length;
$\theta_B$ is Bragg angle;
λ is the wavelength of the incident electron beam.



**Figure 1 — Ewald sphere construction illustrating the diffraction geometry in TEM**

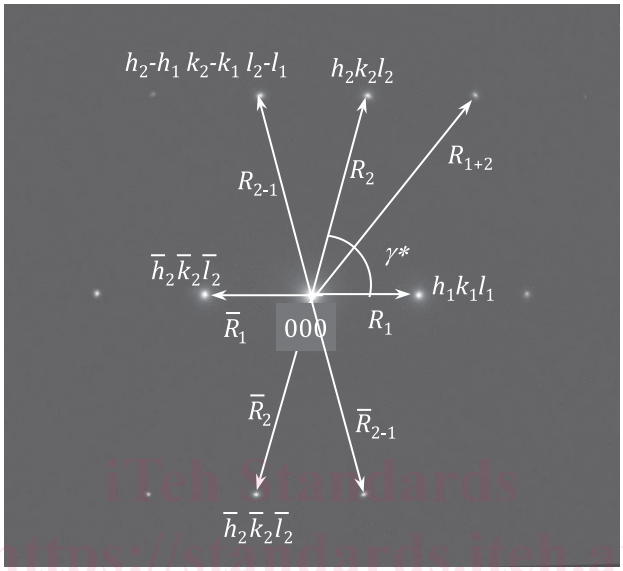
## 4.2 Spot diffraction pattern

The diffraction pattern of a single crystal appears as an array of "spots", the basic unit of which is characterized by a parallelogram. An example of the spot diffraction pattern is shown in Figure 2. Each spot corresponds to diffraction from a specific set of crystal lattice planes in the specimen, denoted by Miller indices (*hkl*). The vector, $\boldsymbol{R}_{hkl}$, is defined by the position of the diffracted spot, *hkl*, relative to position on the pattern corresponding to the direct 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 $\boldsymbol{R}_{h_1k_1l_1}$, $\boldsymbol{R}_{h_2k_2l_2}$, $\left(\boldsymbol{R}_{h_2k_2l_2} - \boldsymbol{R}_{h_1k_1l_1}\right)$ and $\left(\boldsymbol{R}_{h_1k_1l_1} + \boldsymbol{R}_{h_2k_2l_2}\right)$ 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*. The basic parallelogram is

defined by $\boldsymbol{R}_1$ and $\boldsymbol{R}_2$, 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 $|\boldsymbol{R}_{hkl}|$ by measuring the distance between the spots, *hkl* and $\overline{hkl}$ on the diffraction pattern and dividing by two, i.e.

$|R_{hkl}|=\frac{1}{2}\left(|R_{hkl}|+|R_{\overline{hkl}}|\right)$. On the example pattern shown in Figure 2, the magnitude of $\boldsymbol{R}_1$, $\boldsymbol{R}_2$ and $\boldsymbol{R}_{2-1}$ 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**

$\boldsymbol{R}_1$ is the vector from 000 to spot, $h_1k_1l_1$, the shortest vector in the diffraction pattern

$\boldsymbol{R}_2$ is the vector from 000 to spot, $h_2k_2l_2$, the next shortest vector

NOTE The basic parallelogram is constituted by $\boldsymbol{R}_1$ and $\boldsymbol{R}_2$.



**Figure 2 — Example of the spot diffraction pattern from a single crystal**

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

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

where

$\Delta$ 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);

$\lambda$ 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]):

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

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:

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

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 (Section 4.4 and Figure 4). 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, $\boldsymbol{R}_{h_1k_1l_1}$ and $\boldsymbol{R}_{h_2k_2l_2}$, can also be measured on the diffraction pattern. This is equal to the angle between the corresponding 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 8.2.11 and Figure 5). Index the diffracted spots, and then select three non-coplanar 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, $\boldsymbol{R}_{hkl}$, from the corresponding crystallographic plane *(hkl)*. Namely they are perpendicular to the reciprocal vector, $\boldsymbol{g}_{hkl}$, of the plane, ($hkl$).

An example of the Kikuchi patterns is given in Figure 3, where the bright line, $K_{B-hkl}$, and dark line, $K_{D-hkl}$, pairs are superimposed on the spot pattern. The perpendicular distance, $D_{K-h_1k_1l_1}$, between the line pair,