Crystallographic Textures – Measurement

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Measurement of textures
Macrotexture through pole figures by XRD

X-ray diffractometer
Measurement of textures
Macrotexture through pole figures by XRD

Monochromatic radiation required for texture analysis

![Graph showing X-ray absorption and emission wavelengths for various materials.]

**Characteristics of Various X-Ray Tubes and Appropriate Filters**

<table>
<thead>
<tr>
<th>Anode Material</th>
<th>$K\alpha$</th>
<th>$K\beta$</th>
<th>Material</th>
<th>Edge Wavelength (nm)</th>
<th>Thickness for $K\beta/K\alpha = 1/500$ ($\mu$m)</th>
<th>Loss in $K\alpha$ (%)</th>
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</thead>
<tbody>
<tr>
<td>Cr</td>
<td>0.22909</td>
<td>0.20848</td>
<td>V</td>
<td>0.22690</td>
<td>17</td>
<td>51</td>
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<tr>
<td>Fe</td>
<td>0.19373</td>
<td>0.17565</td>
<td>Mn</td>
<td>0.18964</td>
<td>18</td>
<td>53</td>
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<tr>
<td>Co</td>
<td>0.17902</td>
<td>0.16208</td>
<td>Fe</td>
<td>0.17433</td>
<td>19</td>
<td>54</td>
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<tr>
<td>Cu</td>
<td>0.15418</td>
<td>0.13922</td>
<td>Ni</td>
<td>0.14880</td>
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<td>60</td>
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<tr>
<td>Mo</td>
<td>0.07107</td>
<td>0.06323</td>
<td>Zr</td>
<td>0.06888</td>
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<td>71</td>
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<tr>
<td>Ag</td>
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<td>0.04970</td>
<td>Rh</td>
<td>0.05338</td>
<td>92</td>
<td>73</td>
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<td></td>
<td></td>
<td></td>
<td>Pd</td>
<td>0.05092</td>
<td>90</td>
<td>74</td>
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</tbody>
</table>
Measurement of textures

Macrotexture through pole figures by XRD
Measurement of textures

Macrotecture through pole figures by XRD
Measurement of textures

Macrotextrue through pole figures by XRD

- Rotation about an axis perpendicular to the sheet surface (angle $\phi$)
- Rotation about an orthogonal axis (this axis lies in the plane of incident and diffracted beam and is perpendicular to plane normal) by an angle $\chi$
Measurement of textures

Macrotexture through pole figures by XRD
Measurement of textures

Macrotexture through pole figures by XRD
Measurement of textures

Macrotecture through pole figures by XRD

Pole figure scanning
Measurement of textures

Macrotexuture through pole figures by XRD

Iso-intensity contours in experimental pole figures
Measurement of textures

Macrotexture through pole figures by XRD
Measurement of textures

Macrotexuture through pole figures by XRD
Measurement of textures

**Defocusing**

(a) Incident x-ray beam

Sample

Irradiated sample area

(b)

\[ \theta = 90^\circ \quad \theta = 40^\circ \quad \theta = 20^\circ \]

\[ \alpha = 0^\circ \]

\[ \alpha = 45^\circ \]

\[ \alpha = 75^\circ \]
Measurement of textures

Defocusing

Schulz reflection method

Sample view

focusing circle

goniometer circle

detector slit (programmable receiving slit)

source (point focus)

horizontal divergence slit

Sample

Irad. Area
Measurement of textures

Defocusing

Schulz reflection method

- changing statistics
- loss of intensity and peak shift over $\Psi$
- peak broadening and shifting over $2\Theta$
- requires good alignment

Irad. area

Measurement of textures

Defocusing

Further reduced defocusing loss of half of intensity within $65^\circ$, but half of the maximum intensity.

Cu $\{111\}$, $2\theta = 43.45^\circ$
- solid: Cu-CH standard
- open: Cu-WS standard

$\Psi = 0^\circ$
$\Psi = 30^\circ$
$\Psi = 60^\circ$

defocusing function / 1

$\Psi / ^\circ$ (averaged over $\varphi$)

$2\theta / ^\circ$

- red: standard settings
- black: best agreement
- blue: increased focusing
Background correction:

Background error comes because of
(1) Fluorescence in the sample,
(2) Non-coherent scattering in the sample,
(3) Scattering in the path of X-rays by air,
(4) Imperfect monochromatic radiation.

For a pole figure, the background intensity changes with the tilt angle $\alpha$, but usually this does not depend on the sample rotation angle $\beta$.

In practice, the background intensity $I_{BG}(\alpha)$ is measured from pole figure data obtained at an angle away from the diffraction peak angle $\theta$ and integrating over $\beta$. 
Measurement of textures
Corrections to pole figure data

\[ I_{\text{corr}} = I_{\text{meas}}(\alpha, \beta) - BG(\alpha) \]
Defocusing error:

To correct for this *defocusing error*, a correction function $U(\alpha)$ must be applied, which for any value of $\alpha$ normalizes the intensity of a random sample to the values at $\alpha = 0^\circ$:

$$I_{corr} = \frac{I_{meas}(\alpha,\beta) - BG(\alpha)}{U(\alpha)}$$
Absorption correction:
Important in transmission geometry for very thin samples.

When a sample analyzed in transmission geometry is tilted, the path length of the x-rays within the sample increases much more than the increase in the diffracting volume, resulting in a strong decrease in diffracted intensity.

In the case of reflection of x-rays at an “infinitely” thick sample, the increase in absorption is exactly balanced by the increase in diffracting volume, such that the reflected integrated intensity remains constant and a special correction is not necessary.

In the case of very thin samples (thin films), however, the volume increase is dominating and an absorption correction becomes necessary.
Measurement of textures

Corrections to pole figure data

\[
\frac{I(t)}{I_\infty} = 1 - \exp\left(-\frac{2\mu t}{\sin \theta \cos \alpha}\right)
\]
Normalisation:

After pole figure measurement and subsequent correction of the data with respect to background intensity, defocusing error, and, if necessary, absorption, the pole figure data are available as number of counts, or counts per second, per pole figure point \((\alpha,\beta)\).

For representation of the pole figures and for a subsequent evaluation, however, it is necessary to normalize the intensities to standard units that are not dependent on the experimental parameters. The commonly used convention is to express the data in mrd (multiples of random).

\[
I_{\text{norm}}(\alpha,\beta) = \frac{1}{N} \cdot I_{\text{corr}}(\alpha,\beta) \quad \text{where} \quad N = \int_i I_{\text{corr}}(\alpha,\beta) \sin \alpha_i / \int_i \sin \alpha_i.
\]
Measurement of textures

Microtexture in SEM / TEM

Electrons

- SEM-based
  - Kossel
  - ECP
  - EBSD
- TEM-based
  - SADP
  - Kikuchi
Measurement of textures
Microtexture in TEM

• Analysis of selected area diffraction (SAD) spot patterns

• Micro-diffraction and Convergent Beam Electron Diffraction

• High-resolution electron microscopy (HREM)

SAD has been widely used to analyze orientations in a TEM

In contrast to other techniques that yield only orientations of individual crystals, SAD also offers the possibility to measure directly pole figures of small volumes in the TEM
Measurement of textures

Microtexture in TEM

For the determination of individual orientations, Kikuchi patterns, which are obtained by microdiffraction

If highest spatial resolution is required, convergent beam electron diffraction (CBED), is much better suited, as this method combines highest spatial and angular resolutions

Preparation of specimens for TEM examination involves electropolishing for metallic materials and other procedures such as ion beam milling for non-metallic materials

The standard methods used to prepare electron-transparent (i.e., less than approximately 200 nm thick) specimens that are representative of the bulk material are quite exacting, but they are well established
Measurement of textures
Microtexture in TEM

High-Resolution Electron Microscopy

A fascinating technique for determination of local orientations in the very smallest volumes.

The positions of atoms (more precisely, columns of atoms) are imaged by means of an interference method, which enables one to draw directly conclusions on the crystallographic features.

HREM is best suited for determination of mis-orientations as well as for investigation of grain or phase boundaries.
Measurement of textures

Microtexture in TEM

HREM photograph of a 17°/⟨100⟩ grain boundary in gold in which the interfacial structure can be resolved.
Measurement of textures

Microtexture in TEM

For routine orientation measurement, however, HREM is not appropriate for the following reasons:

• Sample preparation is very difficult because HREM requires extremely thin samples (~20 nm).

• Using such thin samples raises the question of whether the orientations determined are actually representative for the sample volume of interest.

• Interpretation of the results is complicated and requires the use of computer simulations.
Measurement of textures

Microtexture in TEM

To utilize diffraction of electrons at the crystal lattice for orientation determination, the volume of interest is irradiated with a parallel electron beam.

To achieve a high resolution, the transmitted volume must be very thin (of the order of the mean free path of the electrons in the material), so that multiple diffraction effects are avoided.

For analysis of small sampled regions, the area of view is reduced by inserting an appropriate aperture in the plane of the first magnified image that has a magnification of typically $\times 25$. 
Measurement of textures

Microtexture in TEM
Investigation of diffraction from single-crystal volumes yields characteristic patterns that are composed of a regular arrangement of individual diffraction spots, which can be evaluated for orientation determination.

SAD pattern of an aluminum crystal with a zone axis near $\langle 110 \rangle$
The diffraction spots are formed by coherent elastic scattering of the electrons at the crystal lattice.

Because of the very short wavelength of the electron radiation, the diffraction angles between the reflecting lattice planes and the primary beam are very small as well—at the most approximately 2°.

This means that all the reflecting planes are situated almost parallel to the primary beam or, in other words, the primary beam is a zone axis of the reflecting planes.
Measurement of textures

Microtexture in TEM

\[ n\lambda = 2d \sin \theta \approx 2d\theta = \frac{2dR_{hkl}}{2L} \]

\[ \lambda L = R_{hkl}d \] or \[ R_{hkl} = \frac{\lambda L}{d} \]
Schematic illustration of formation of SAD ring patterns in polycrystalline assemblies. (b) SAD diffraction pattern of evaporated Al with random texture. (c) SAD diffraction pattern of cold-rolled aluminum with strong texture.
Formation of SAD pole figures in a TEM; (b) and (c) coverage of the pole figure as the angle $\alpha$ is gradually increased.
Measurement of textures

Microtexture in TEM

Orientation: (101) <1-1-1>
Measurement of textures

Kikuchi lines in TEM

![Diagram of Kikuchi lines in TEM](image)
Simulated diffraction patterns for a [100] axis showing both SAD spots and Kikuchi lines for (a) un-tilted, that is, exact [100] orientation and (b) 2° tilted orientation. These patterns show that Kikuchi lines have much greater sensitivity to crystal orientation than SAD spots (simulation program TOCA by Zaefferer, 2002).
Measurement of textures

Kikuchi lines in TEM

Micro-diffraction patterns from [111]-oriented crystals obtained with increasing convergence of the electron beam. (a) Kikuchi lines and HOLZ lines in the zero-order Laue zone (ZOLZ) (Nimonic PE16).
Measurement of textures

Kikuchi lines in TEM

\[ p_1 d_1 = p_2 d_2 = p_3 d_3 = \text{constant} \]

AC = 3.8 cm
BC = 2.2 cm
AB = 2.65 cm
OA = 2.5 cm
OB = 1.35 cm
OC = 2.7 cm

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Measurement of textures
Kikuchi lines in TEM

(a)

(b)
Measurement of textures

Kikuchi lines in SEM

- Each band: diffraction of a family of planes
- Intersections of bands: intersections of planes = zone axes
- Angles between bands: angles between planes
- Band widths: proportional to d(hkl) related to lattice parameters Middle line of a kikuchi band represents plane

Zone axis
Measurement of textures

Kikuchi lines in SEM
Measurement of textures

- The origin of the gnomonic projection, labeled $N$ referred to as the pattern center, $PC$
- The radius of the reference sphere, labeled $ON$ and referred to as the specimen-to-screen distance, $Z_{SSD}$
Measurement of textures

Automatic indexing in EBSD

- Diffraction pattern capture, digitization
- Average $n$ frames ($1 \leq n \leq 16$)
- Background subtraction
- Hough transformation (lines $\rightarrow$ points)
- Evaluate angles between zones
- Compare sets of known interzonal angles with measured set
- Select best fit
- Calculate Euler angles
Measurement of textures

Hough Transform

\[ \rho_i = x_j \cos \theta_i + y_j \sin \theta_i \]
Measurement of textures

Automatic indexing in EBSD
Automatic Indexing

• Automated indexing of the Kikuchi patterns is accomplished by making lists of inter-zonal angles, i.e. the angle between each pair of (strong) bands.

• These angles must correspond to the fixed angles between zones in the crystal structure known to be present in the specimen, e.g. the angle between [001] and [111] is 54.7°.

• Once the zones have been identified, the geometry of the system permits the orientation of the crystal to be related to that of the pattern.
The unit vector \( n_1 = \frac{r_1 \times r_2}{|r_1 \times r_2|} \) is perpendicular to the crystal plane generating the first Kikuchi band and the vector \( n_2 = \frac{r_3 \times r_4}{|r_3 \times r_4|} \) is perpendicular to the crystal plane generating the second Kikuchi band.

The angle between the planes forming the two Kikuchi bands is \( \cos^{-1}(n_1 \cdot n_2) \).
Measurement of textures

- Indexing of the planes and zone axis
- Assigning the orientation / calculation of Euler angles
**Pattern Centre Calibration**

- Crystal of known orientation, Si [001]

\[
\cos \phi = \frac{\begin{pmatrix} x_1 - x_{PC} \\ y_1 - y_{PC} \\ Z_{SSD} \end{pmatrix} \cdot \begin{pmatrix} x_2 - x_{PC} \\ y_2 - y_{PC} \\ Z_{SSD} \end{pmatrix}}{\sqrt{(x_1 - x_{PC})^2 + (y_1 - y_{PC})^2 + Z_{SSD}^2} \cdot \sqrt{(x_2 - x_{PC})^2 + (y_2 - y_{PC})^2 + Z_{SSD}^2}}
\]
Pattern Centre Calibration

Iterative Pattern Fitting

- Calibration via iterative pattern fitting requires only an EBSD pattern of reasonable quality in which at least three bands or zone axes can be identified.

- Equation 1 can then be formulated \( n(n - 1)/2 \) times, where \( n \) is the number of identified zone axes, substituting in the known indices of two of the poles in turn. These equations have to be solved numerically to obtain \( x_{PC} \), \( y_{PC} \), and \( Z_{SSD} \).

- The numerical method needs a “first guess” for the values of the \( PC \) coordinates and \( Z_{SSD} \). If the first guess is reasonably good, less iterations will be required to converge toward the right solution.
Resolution and Operational Parameters

- Microscope parameters (SEM / FEGSEM)
- Material (atomic number)
- Specimen/microscope geometry (working distance, tilt angle)
- Accelerating voltage (interaction volume)
- Probe current
  - (pattern quality)
- Pattern quality
  - (defects in the crystal)
Spatial resolution of EBSD in nickel as a function of accelerating voltage
(a) The effect of probe current on effective resolution for several aluminum specimens. The minima in the plots are caused by the reduced pattern-solving accuracy at low probe currents. (b) Effective EBSD spatial resolution for various metals in tungsten filament and FEG SEMs. (c) Misorientation measurements between adjacent points on a single-crystal silicon specimen for four different probe currents (in amperes). The highest-probe current provides the most accurate result.
Original and Fourier-transformed EBSD patterns with different quality to derive the image quality $IQ$. (a) Original poor-quality EBSD pattern; (b) original high-quality EBSD pattern; (c) Fourier spectrum of (a) ($IQ = 0.29$); (d) Fourier spectrum of (b) ($IQ = 0.43$).
Sample Preparation for EBSD

- Specimen preparation is straightforward, often similar to that for optical microscopy.

- The specimen preparation objective for EBSD can be stated very simply: the top 10–50 nm of the specimen should be representative of the region from which crystallographic information is sought.

- The specimen surface must not be obscured in any way—by mechanical damage (e.g., grinding), surface layers (e.g., oxides and most coatings), or contamination.

- The standard metallographic preparation route for most specimens, especially metals and alloys, is mounting, grinding, and polishing.
Sample Preparation for EBSD

Illustration of the penetration depth of the electron beam in a silicon EBSD specimen. (a) No coating, 40 kV accelerating voltage; (b) no coating, 10 kV accelerating voltage; (c) coating with 5 nm of nickel, 40 kV accelerating voltage; (d) coating with 5 nm nickel, 10 kV accelerating voltage. There is less beam penetration at 10 kV since the underlying silicon pattern is indistinct.
Sample Preparation for EBSD

• Mounting the specimens in a conducting medium is clearly advantageous for SEM work; otherwise, electrical contact with the specimen can be established by using silver or carbon paint or conductive tape, or simply by cutting the specimen from the mount after the preliminary preparation stages.

• It is the final preparation step that ensures suitability for EBSD. Diamond polishing is not an appropriate final stage because of the remnant mechanical damage entailed.

• A highly recommended method for preparing a variety of specimens for EBSD is final polishing in colloidal silica, since this medium does not introduce the harsh mechanical damage associated with diamond polishing.
Measurement of textures

Pattern Quality map

EBSD map of hot-deformed and partly recrystallized Al–Mg alloy (AA5182). (a) Pattern quality map in which the high quality patterns appear brighter
Grains of the main texture components coloured—Red = *Cube* {100}001, Green = *Goss* {011}100, Blue = *Brass* {011}211
Measurement of textures

Orientation mapping

- Strongly cube textured material i.e., strong \{100\} <001> orientation
Measurement of textures
Mesotexture analysis

- Analysis of grain boundary network and the misorientation distribution permits quantitative description of microstructure.