Texture and Anisotroy

Part III:

Chapter 6. Kikuchi diffraction pattern

History of Kikuchi Pattern

S. Nishikawa, S. Kikuchi (1928) "The Diffraction of Cathode Rays by Calcite", *Proc. Imperial Academy (of Japan)*, **4**, 475-477.

A gas discharge beam of 50 keV electrons was directed onto a cleavage face of calcite at a grazing incidence of 6°. Patterns were also obtained from cleavage faces of mica, topaz, zincblende and a natural face of quartz. They were called *P-patterns* or *patterns of the fourth kind*. Later names included *reflection Kikuchi patterns* or *backscatter Kikuchi patterns*.



K. Shinohara (1932), R von Meibohm and E Rupp (1933)



Scattering and diffraction: thin film



coherent scattering: electron waves are in phase incoherent scattering: have no phase relationship after interacting

Scattering and diffraction: bulk



Elastic scattering: at low angle 1-10, in the forward direction at high angles >10 incoherent Inelastic scattering: incoherent with low angle <, forward scattering in bulk incoherent backscattering

EBSD Definition

<u>Electron</u> <u>Back</u>scattering <u>D</u>iffraction = EBSD



Electron diffraction techniques

- A Kikuchi diffraction pattern arises by application of the following electron diffraction techniques:
- SAC in SEM
- EBSD in SEM
- Micro-diffraction or convergent beam electron diffraction (CBED) in TEM



Kikuchi patterns in TEM



Formation of Kikuchi patterns in TEM



The formation of Kikuchi lines can be explained in terms of a simplified model which considers only their geometrical aspects.

When an electron beam enters a crystalline solid, it is diffusely scattered in all directions.

There must always be some electrons arriving at the Bragg angle $\theta_{\rm B}$ at every set of lattice plane.

Formation of Kikuchi patterns



The two lines forming a given Kikuchi band in general have different intensities.

Bright: typically the line is closer to the primary beam (θ_e) – excess line

Dark: the other one is faraway from the primary beam (θ_d) – defect line

Formation of Kikuchi pattern in SEM



Tilting the specimen by angles of typically 60-70° allow more electrons to be diffracted and to escape towards the detector.

Kikuchi patterns can also be produced in the SEM by changing the direction of the incident beam.

Formation of Kikuchi bands in SEM



Kikuchi pattern in TEM & EBSD



Differences between TEM and EBSD Kikuchi pattern:

-The capture angle is about five times greater for EBSD pattern than for TEM pattern

- The Kikuchi lines are sharper in TEM pattern than in EBSD pattern



Kikuchi pattern in SAC & EBSD

Differences between SAC and EBSD Kikuchi pattern: -The capture angle is much greater for SAC pattern than for EBSD pattern

- The Kikuchi lines are more fine detail in SAC pattern than in EBSD pattern



FIGURE 6.3

Comparison between (a) SAC and (b) EBSD patterns of a (111) zone axis in TiAl. The EBSD pattern has a much greater capture angle, whereas the SAC pattern contains more fine detail, for example, {110} and {112} superlattice bands. (Courtesy of M.A. Crimp.)

Spherical projection of Kikuchi pattern



Gnomonic projection of Kikuchi pattern: fcc



(a) A single experimental EBSD pattern mapped onto the surface of a sphere (left) and several montaged EBSD patterns (right) shown with black outlines. (Courtesy of A. Day.) (b) Illustration of a Kikuchi pattern as a gnomonic projection, showing the reference sphere (radius *r*) and a projected pole *P*. The projection point is *O* and the origin of the projection (pattern center) *N*.

Gnomonic projection of Kikuchi pattern: bcc



Gnomonic projection of Kikuchi pattern: hcp



Qualitative evaluation of Kikuchi pattern

Information of Kikuchi pattern:

- Lattice strain:

determine lattice strain using pattern diffuseness (Wilkinson 1996; Troost et al. 1993)

- Grain/phase boundaries

distinguish between low-angle and high-angle grain boundaries

- Orientations

Orientation index of Kikuchi pattern in TEM



Angle	(IINI) ₁	$(11 \times 1)_2$
25.2	200	311
29.5	111	311
31.5	220	311
35.1	311	311
35.3	111	220
45.0	200	220
50.5	311	311
54.7	111	200
58.5	111	311
60.0	220	202
63.0	311	131
64.8	220	311
70.5	111	111
72.5	200	131
80.0	111	311
84.8	311	131
90.0	111	220
90.0	200	020
90.0	200	022
90.0	220	113
90.0	220	220

Anala (hkl)

(hk)

Determination of ND



$$\begin{pmatrix} q_1^i \\ q_2^i \\ q_2^i \\ q_2^i \end{pmatrix} \cdot \begin{pmatrix} h \\ k \\ l \end{pmatrix} = \cos \alpha_i \cdot \sqrt{(q_1^i)^2 + (q_2^i)^2 + (q_3^i)^2}$$

$$\alpha_1 = 8.5^{\circ}, \alpha_2 = 11.6^{\circ}, \alpha_3 = 7.2^{\circ}$$

(h,k,l) = (0.486, 0.541, 0.686)

 $(h,k,l) \approx (5,6,7)$

Orientation determination of RD in TEM



 γ between the (0-22) band normal and RD is 41.5°

$$RD = [uvw] = [-0.616, -0.345, 0.707]$$

$$\approx [\overline{212}]$$

$$TD = [qrs] = [0.620, -0.766, 0.165]$$

$$\approx [4\overline{51}]$$

Orientation determination of ND in TEM



Orientation determination of EBSD



2D screen coordinates (x_i^*, y_i^*) to 3D sample coordinates

$$r_{i} = \begin{pmatrix} x_{i}^{s} \\ y_{i}^{s} \\ z_{i}^{s} \end{pmatrix} = A \cdot \begin{pmatrix} x_{i} \\ y_{i} \\ z_{i} \end{pmatrix} = A \cdot \begin{pmatrix} x_{i}^{*} - x_{PC} \\ y_{i}^{*} - y_{PC} \\ Z_{SSD} \end{pmatrix}$$

Coordinate transformation

Transform the specimen coordinates into the crystal coordinates by:

$$r_i^c = g \cdot r_i^s$$

For each vector r_i (axis or band) in the sample frame:

$$r_i^s = \begin{pmatrix} x_i^s & y_i^s & z_i^s \end{pmatrix}$$

For each vector r_i (axis or band) in the crystal frame:

$$r_i^c = \begin{pmatrix} x_i^c & y_i^c & z_i^c \end{pmatrix}$$

Definition orientation matrix g

Definition of orientation matrix g is given:

$$\begin{pmatrix} x_1^c & x_2^c & x_3^c \\ y_1^c & y_2^c & y_3^c \\ z_1^c & z_2^c & z_3^c \end{pmatrix} = g \cdot \begin{pmatrix} x_1^s & x_2^s & x_3^s \\ y_1^s & y_2^s & y_3^s \\ z_1^s & z_2^s & z_3^s \end{pmatrix}$$

$$g = \begin{pmatrix} x_1^c & x_2^c & x_3^c \\ y_1^c & y_2^c & y_3^c \\ z_1^c & z_2^c & z_3^c \end{pmatrix} \cdot \begin{pmatrix} x_1^s & x_2^s & x_3^s \\ y_1^s & y_2^s & y_3^s \\ z_1^s & z_2^s & z_3^s \end{pmatrix}^{-1}$$

Automated evaluation of EBSP: Hough transformation

 $\rho = x_i \cdot \cos\theta + y_i \cdot \sin\theta$



Schematic representation of the Hough transform. (a) Line with three points in the original image; (b) all the corresponding three Hough-transformed curves from (a) meet in one point in Hough space whose coordinates ρ and θ then specify the original line.

Hough images



Gray-tone weighted Hough transform, or Radon transform, of two bands of distinct width in a gray-tone image. (a) Original image; (b) Radon-transformed image, showing the development of the characteristic butterfly shape of transformed EBSD bands.

Indexed bands in EBSD pattern





FIGURE 6.9

Gray-tone weighted Hough transform, or Radon transform, of an EBSD pattern of aluminum with overlaid bands. (a) Original EBSD pattern; (b) pattern after the Radon transform. (Courtesy of A. Gholinia.)

Problems in TEM automation evaluation



For TEM Kikuchi pattern, problems should be overcome:

- much higher dynamic range in TEM Kikuchi patterns due to variations in background intensity

- inhomogenous intensity of bright and dark Kikuchi lines in TEM Kikuchi patterns
- Unambigious indexing of TEM Kikuchi patterns is more complicated

Dynamic range

The contrast of TEM Kikuchi patterns is much larger than that of EBSD.

The TEM Kikuchi patterns are superimposed by high-intensity SAD diffraction spots and by primary beam.

bright and dark Kikuchi lines

TEM Kikuchi patterns consist of pairs of bright and dark lines. Therefore, the defect Kikuchi lines with intensities below that the background are not removed.

Low-index lines in TEM are associated with parallel high-order diffraction lines.

Unambiguous indexing

Owing to the smaller steric angle, but larger number of high-index bands, unambiguous indexing is more complicated for TEM that for EBSD.

In EBSD patterns of fcc materials only bands up to (311), whereas in TEM Kikuchi patterns bands likes (244) and higher are obtained.

Pattern quality

Kunze et al.: A quality parameter IQ, "Image quality", is defined as the sum over the peak sharpness of all detected peaks.

Lassen et al.: using Fourier transformation

$$F(u,v) = \frac{1}{n^2} \sum_{x=0}^{n-1} \sum_{y=0}^{n-1} I(x,y) \exp\left(-2\pi i \frac{ux+vy}{n}\right)$$

$$IQ = 1 - \frac{I}{I_{\text{max}}}$$

Image quality



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).