Microtexture: Electron Diffraction in the SEM (L16)

Texture, Microstructure & Anisotropy, Fall 2009
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Different types of microtexture techniques for obtaining crystallographic information.
SEM-based Microtexture Techniques
Kossel Technique:

- This technique is based on using electron beam to generate X-rays within the sample under investigation.

- Need high acceleration voltage. About 2-3 times larger than the critical voltage to produce characteristic X-ray Kα radiation.

- The X-rays are subjected to subsequent reflection at the lattice planes of the sample in accordance with Bragg’s law to form cone shapes - Kossel cones.

- These are recorded on an X-ray sensitive film (see Fig. next slide).
(a) Diagram illustrating the formation of Kossel patterns in reflection; (b) Kossel pattern from titanium
• Because the wavelength of X-rays (Kossel technique) is much larger than that of electrons, all Bragg angles $\theta$ between $0^\circ$ and $90^\circ$ may occur.

• Therefore the resulting projection lines on the film plane are strongly curved as seen in Figure 22(b).
Electron Channeling Pattern (ECP):

- Can be used to determine crystallographic orientation, structural type (image contrast), unit cell size, or a measure of the crystal perfection.

- Although we cannot obtain diffraction spot patterns in the SEM, as we can in the TEM, we can make use of electron channeling for diffraction patterns.
• For orientation determination, the electron beam in the SEM is focused on the sample site to be analyzed.

• The beam is tilted at this position by an angle $\alpha$, and rocked on the specimen surface, as shown in Figure, next slide.
Schematic showing the rocking of the beam.
• The resulting pattern is known as selected area channeling pattern (SACP).

• Although there method of formation are not identical, the SACPs are geometrically similar to TEM Kikuchi patterns and EBSD patterns.

• Therefore, they are analyzed in a similar manner.

• They are composed of bright bands of a given thickness representing distinct crystallographic planes (see Figs. next slides).
Schematic showing the resulting pattern.
Electron channel pattern obtained from a gallium phosphide crystal of (111) orientation.
Electron channel pattern of a recrystallized grain in a partially recrystallized Al-Fe-Si sample.
• In general, the spatial resolution of this technique is limited to \( \sim 10 \, \mu m \).

• Also, the technique is very sensitive to lattice defects. Therefore, only recrystallized or recovered microstructures have been successfully analyzed.
• It is also possible to obtain channeling contrast for structural information.

• Channeling contrast is generally much weaker than atomic number contrast. Therefore contrast is low.

• Good electron detector, a carefully prepared specimen and large beam currents are usually required in order to improve the contrast.
Electron Backscattered Diffraction (EBSD):
Can be considered as the most widely used microtexture technique.

Generation of Patterns
• Backscattered electrons are produced when stationary beam (scan coils are turned off) are incident on a specimen (in the SEM) that is tilted to about 70° (see next Figure).
Schematic diagram showing the specimen-beam interaction volume in a specimen tilted for EBSD
The Interaction Volume of Backscattered Electrons

- When Considering the Issue of Interaction Volume\Penetration Depth:
  - Will the beam penetrate through the precipitates?
  - Will there be pattern overlapping from Austenite Matrix?

\[ \delta x \approx 2 \text{ times the spot size} \]
\[ \delta y \approx 2.5 \text{ to } 3 \text{ times the spot size} \]

Penetration depth (\(\delta z\)): 50 – 100nm
• The main effect of tilting the specimen is to reduce the path length of electrons, which have been backscattered by lattice planes.

• Therefore, when compared to flat specimen, more of these electrons undergo diffraction, and escape from the specimen before being absorbed.
• The EBSD pattern results from diffraction of a divergent source of electrons generated within the sample just beneath the point where the primary electron strikes the specimen.

• The electrons that contribute to the pattern are only those:
  – That have lost no more than a few electron volts of energy.
  – Emerge from a depth in the specimen of no more than 30 to 40 nanometers, which is a considerably smaller depth than that reached by the primary beam.
• The lower the primary beam voltage, the better the resolution, because the beam spread is smaller.

• This benefit may be offset, because at lower primary voltages, the probe diameter for a given current is larger.

• The large beam current can be countered by using:
  – Field emission source, or
  – Lower beam currents. However, lower beam current leads to a poorer signal to noise ratio.
Detection of Patterns

• The diffraction pattern can be captured on a recording medium or device placed in front of the tilted specimen.

• Examples of detecting or recording medium are:
  – a phosphor screen, viewed by a low light television camera. The luminosity of a phosphor screen for a beam current of 100 pA at 20 kV is of the order of $10^{-4}$ lux (10 times greater than that detectable in live TV by a SIT camera.
  – CCD camera, which produces about 50 times more luminosity than a phosphor screen.

• Next Figure shows a schematic of the specimen and the detecting medium.
Schematic of a tilted specimen and a detecting medium.

THE KIKUCHI DIFFRACTION PATTERN

incident electron beam

sample

phosphor screen

(x^S, y^S, z^S)

r_i

(x^PC, y^PC)

(x^*, y^*)
THE KIKUCHI DIFFRACTION PATTERN

TEM
• Image processing involves **integration of successive patterns and background subtraction**, which are used to improve the signal to noise ratio.

• The above reduces the dynamic range to between gray level 90 and gray level 120. Sharper and higher order lines are usually lost in this process.

• Further image processing is performed to reduce the number of pixels in the image to approximately 10,000. This enables the computer analysis of the pattern, namely the Hough transform, to be executed in a fraction of a second.
Schematic of the components of an EBSD system.
Timeline of EBSD development.

1930s-1950s....

First observations of high-angle Kikuchi patterns.

1970s....

Electron back-scattering patterns recorded in the SEM

Early 1980s....

Computer routines for interactive EBSD pattern evaluation
Late 1980s....

Automated EBSD pattern analysis

1990s....

Orientation mapping
**EBSD Specimen Preparation:**

- A carefully prepared specimen, with no deformed surface (top 10-50 nm) layer.

- Specimen preparation is supposed to be uncomplicated, and similar to that for optical microscopy.

- Since the diffraction zone is shallow, the specimen surface must no be obscured by:
  - mechanical damage due to grinding,
  - surface layers due to thick coatings,
  - surface undulation due to protruding particles, or
  - contamination
• Minimization of diamond polishing is necessary in order to avoid surface damage.

• It may be necessary to avoid some common electropolishes or etchants, which may deposit films on the surface. A useful guide is to use TEM electrolytes.

• A combination of etching and mechanical polishing may be used, especially if OIM is required.

• Final polish in colloidal silica is highly recommended.
• Inadequate or inappropriate specimen preparation could give rise to erroneous data interpretation.

• Acceleration voltage and coating of samples can affect the quality of diffraction pattern obtained, as can be seen in the next Figure (a) - (c).
Effect of acceleration voltage on the quality of diffraction pattern of sample with no coating (a) 10 kV accelerating voltage and (b) 40 kV accelerating voltage.
Effect of acceleration voltage on the quality of diffraction pattern of a coated (5 nm Ni) sample (c) 10 kV accelerating voltage and (d) 40 kV accelerating voltage.
Some Specimen Preparation Methods

• Heavily Deformed OFHC Copper
  – Mechanical polish
  – Light diamond polish
  – Silica on Vibromet polish
  – Etch in 50% Ammonium Hydroxide and 50% Peroxide
  – Silica on Vibromet polish
• Commercially pure aluminum, titanium alloys
  – Mechanical polish
  – Light diamond polish
  – Electropolish in 5% perchloric acid in ethanol at -25°C

• Aluminum-lithium alloys
  – Mechanical polish
  – Light diamond polish
  – Immerse for several seconds in Keller’s reagent, slightly warmed.
• Mild Steel
  – Mechanical polish
  – Light diamond polish
  – Swab with 2% nital for several seconds
Limitations of EBSD Technique:

• Materials with very fine grain/subgrain size.  
  e.g. Nanocrystalline material - can be resolved by employing the TEM-based microtexture technique.

• Materials that have undergone extensive deformation.  
  e.g. heavily cold worked material by wire drawing - may resolve through novel polishing technique.
Resolution and Operation Parameters:

• The absolute spatial resolution of EBSD technique depends on the interaction volume of the electron beam and the specimen, which in turn depends on the spot size. Quoted values are:
  – average spatial resolution is about 200 to 500 nm
  – accuracy is about 1°

• The above parameters are influenced by:
  – Material
  – Specimen/microscope geometry
  – Accelerating voltage
  – Probe current
  – Pattern clarity
• **Material**
  - Backscattered signal increases with atomic number. Therefore, better patterns are generally obtained from materials with high atomic number elements.

• **Specimen/Microscope geometry**
  - specimen tilt angle must be about 70°
  - Maintain short working distance, and this will depend on the geometry of the microscope.
  - Specimen to screen distance. This is fixed for each microscope - camera pair.
• Accelerating voltage
  – There is a linear relationship between accelerating voltage and interaction volume for every element. One needs to find an optimum accelerating voltage, and this can always be compensated/complemented with probe current.

• Probe current
  – This is usually selected in accordance with the light sensitivity of the camera.
Orientation Imaging Microscopy (OIM):

- An Orientation Imaging Micrograph is produced by successively collecting and indexing Electron Backscattered Diffraction (EBSD) patterns at point spaced over a specimen surface in a regular grid.

- The technique is based on electron backscattered diffraction in the scanning Electron microscope.