Diffraction techniques in the Scanning Electron Microscope (SEM)

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Scanning electron microscopy (SEM)
Scattering and diffraction

- Backscattered electrons (EBSD, ECCI, imaging)
- Secondary electrons (Imaging)
- Incident electron beam
- Direct beam (Bright field images)
- Elastic scattered electrons (SAD-patterns, dark field imaging)
- Inelastic scattered electrons (Kikuchi patterns, TKD)
- Auger electrons (surface analysis)
- Back reflection
- X-rays (Chemical analysis)
- Transmission
Electron Diffraction Techniques in the SEM

1. ECCI – Electron channelling contrast images
   Observation of crystal defects (dislocation, stacking faults and grain boundaries)

2. EBSD - Electron Backscatter Diffraction
   Microstructural – crystallographic characterization technique for bulk samples

3. TKD - Transmission Kikuchi Diffraction
   Microstructural – crystallographic characterization technique for thin samples
EBSD
Electron Backscatter Diffraction
Electron backscatter diffraction - history

- 1928 - First observation of BKP by Nishikawa & Kikuchi
- 1973 - Observation of BKP in a SEM by Venables and Harland
- 1984 - Dingley started using TV camera and computer software for orientation determination
- 1993 - Introduction of OIM (Orientation image microscopy) by Adams et al.
Formation of Kikuchi pattern – step 1

The formation of EBSD patterns is a two-step process.

- Electrons strike the specimen

1. They are then inelastically scattered from the point source in all directions.

*Inelastic: some loss of energy*
2. crystalline materials: those electrons (from inside the point source) are diffracted by the crystal lattice planes when the Bragg condition is satisfied.

\[ n\lambda = 2d \sin \theta \]

- \( \lambda \): wavelength of the electrons
- \( d \): spacing of the crystal planes
- \( n \): is an integer
- \( \theta \): angle of incidence
Simplified illustration – one electron and one lattice plane

- Since the scattered electrons are travelling in all direction, the diffracted beam will lie on one of two cones.

Adapted from:
Two electron and two lattice plane

Thin sample

Adapted from:
Simplified illustration – one electron and one lattice plane

Adapted from:
EBSD – tilted sample

Bulk sample

Tilt the sample to approximately 70°
(best compromise between intensity and resolution)
EBSD – tilted sample

Scattering from single lattice planes

View from the SEM

EBSD patterns are:

- **unique** for a specific crystal orientation
- is controlled by the crystal structure: space group symmetry, lattice parameters, *precise* composition

**Position of bands directly linked to the crystallographic orientation**

Diffraction from a specific lattice plane

Intersections of bands = intersections of planes = zone axes

**Angles between bands = angles between planes**

Ref.: Dr. Emmanuelle Boehm Courjault; Introduction to EBSD (Electron BackScatter Diffraction): Principle and Applications
Surface sensitive technique

- Although EBSPs are created by backscattered electrons, the signal does not come from the whole BSE interaction volume.

- Instead, the diffraction signal originates from a "POINT SOURCE" → 5-10nm under the surface.
Surface sensitive technique

- The top layer
  - Free from damage
  - Free from contamination
  - or oxidation layers
  - in case of non conductive samples → the coat must be kept very thin – typically in the range of 2-5 nm.

- Due to high tilt angles (typically 70°),
  - surface topography must be kept to an absolute minimum.

Sample strongly tilted – Resolution y axis is 3x worst

Shadow on the screen
Only a small fraction of the electron arriving at the phosphor screen are diffracted.

Background intensity suppresses both the contrast and sharpness.

Raw pattern

Background

Nb-pattern from S. Zaefferer
Signal is material dependent

Increasing atomic number ($\uparrow Z$):

- Increase the amount of backscatter electrons – **Pattern quality**
- Decreases the interaction volume - improve spatial resolution

![Beryllium (Z=4), 30 kV, exposure time ~ 15 s](image1)
![Magnesium (Z=12), 15 kV, exposure time ~ 6 s](image2)
![TiCr Laves phase (Z=22...24), 15 kV](image3)
Short take home message

• It is not easy to prepare the sample for EBSD
• Once you get patterns, it runs fully automated

Automate data acquisition, pattern indexing and orientation determination

In one automate run you get:
• Grain size,
• texture,
• grain boundary distribution,
• phase distribution,
• …
Automated indexing during the acquisition

Choose the step size and the area

Runs very fast: Now a days up to 3000 patterns per seconds

Orientation map

Grain boundary map (misorientation)

Grains size distribution

Misorientation angles

Grain boundary map

Now a days up to 3000 patterns per seconds
Colourful image

100 plane // to the surface

111 plane // to the surface

110 plane // to the surface
Orientation with respect to the sample coordinate system

EBSD is a materials characterization tool

EBSD paper by subject area

- **MICROSCOPY**: 4%
- **GEOLOGY**: 4%
- **CHEMISTRY**: 7%
- **CRYSTALLOGRAPHY**: 9%
- **MECHANICS**: 11%
- **SCIENCE & TECHNOLOGY**: 13%
- **ENGINEERING**: 22%
- **METALLURGY**: 28%
- **PHYSICS**: 32%
- **MATERIALS SCIENCE**: 64%

Web of Science
Application example -1

Niobium deformed by ECAP and heat treated for 15 min

Local misorientation map

Deformed areas

Recrystallized areas

Orientation bands – related to the type of deformation - **shear**

Niobium deformed by ECAP and heat treated for 15 min

H. Sandim; www.imim.pl