EBSD Basics

- Quantitative, general microstructural characterization in the SEM
- Orientation measurements, phase identification
- Near-surface technique
  - Using diffraction patterns originating 20 nm - 100 nm below the surface
  - Surface preparation is critical
- Materials analyzed
  - Crystalline materials that survive under the beam
  - Metals, ceramics, minerals
  - Conductors and insulators
  - Pharmaceuticals and polymers generally damage too quickly to work on, although...

EBSP = Electron Backscattered Pattern
**Uses of EBSD**

- **Quantitative microstructural data**
- **Phase identification**
- **Strain analysis**

**How EBSPs are obtained?**

- **Tilt 70° - Spot mode - 30 kV - 10 nA**
- **Phosphor screen + CCD camera**

**EBSD: electron diffraction in the SEM**

- Electrons of the incident beam spread beneath the surface in all directions due to elastic interactions (backscattered electrons):
  - Small divergent source of electron behind (~ 100 nm) the sample surface.
- These electrons are diffracted by crystal planes according to the Bragg condition.
Sample preparation

- **Requirements**

  The backscattering volume (100 nm) below the surface sample must be crystalline and without excessive plastic deformation.

  Problems: → plastic deformation due to mechanical polishing
  → foreign layers (oxide)
  → internal strain

- **Preparations methods**

  ✓ Mirror quality polishing (→ 0.25 μm diamond grade), and:
    - chemical-mechanical polishing (silica or alumina suspension)
    - electro-polishing or chemical polishing/etching
    - ion-milling or plasma etching

  ✓ cleaved surface, growth surface

  ✓ Insulating materials
    → carbon coating (< 100 Å) (degrade pattern quality)
    → low-vacuum SEM (a few Pa)

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Acquisition of EBSPs

EBSD consist of Kikuchi bands corresponding to the various diffracting planes. Intersections of these bands correspond to crystal zone axis. The geometrical arrangement of Kikuchi bands depends on crystal symmetry and crystal orientation.
Diffraction of backscattered electrons

- Incident beam
- Inelastically scattered electrons (forward scattered / back scattered)
- Crystal planes = Bragg diffraction

Formulation of EBSPs

- **Backscattering**: due to slightly inelastic (< 200 eV) interactions (plasmons, phonons) the electron beam is spread in all directions. The emission volume corresponds to a small divergent source of electrons below the sample surface (100 nm).

- **Scattering** by crystallographic planes: 2 diffraction cones

  \[ \text{Bragg} : 2d_{hkl} \sin \theta = n \lambda \]

  \[ 20 \text{ keV} \rightarrow \lambda \approx 7 \times 10^{-3} \text{ nm} \rightarrow \theta \approx 0.5^\circ \]

- **Gnomonic projection** on the screen: 2 hyperbolas (Kikuchi bands). The middle of a band corresponds to the trace of the diffracting plane.

- **Relatives intensities**: structure factors (dynamical effects)
Indexation of EBSPs

Each band = diffraction of a family of planes
Intersections of bands = intersections of planes = zone axes
Angles between bands = angles between planes
Position of bands directly linked to the crystallographic orientation

Hough transform

EBSP : the computer doesn't manage to distinguish between the grey levels
Hough transform : it is easier for the computer to detect the clear spots and dark areas top and bottom
Line positions with a common intersection (zone axis) will lie along a line/sine curve
Hough transform

The transform between the coordinates \((x, y)\) of the diffraction pattern and the coordinates \((\rho, \theta)\) of Hough space is given by (Figure 8):

\[
\rho = x \cos \theta + y \sin \theta
\]  

(3)

A straight line is characterized by \(\rho\), the perpendicular distance from the origin and \(\theta\), the angle made with the \(x\)-axis and so is represented by a single point \((\rho, \theta)\) in Hough space. Kikuchi bands transform to bright regions in Hough space which can be detected.

![Image](image1.png)

Figure 8: The Hough transform converts lines into points in Hough space

Influence of accelerating voltage

![Image](image2.png)

Si [100], same WD and camera length

- The phosphor screen efficiency drops at reduced voltages.
Limited by:

- Accuracy on the localization of Kikuchi bands (Hough, ...).
- The weak signal/noise ratio of the images, and blur of Kikuchi bands.
- Geometrical fluctuations of the conditions of diffraction, and calibration.
- Absolute accuracy: sample position in the chamber.

Spatial and Angular accuracy

- 0.1 - 1° relative
  ≈ 2° absolute
- 20 nm - 1 μm

Limited by the overlapping of diffraction patterns in the vicinity of a boundary

Depend on:

- interaction volume (energy and Z)
- size of the beam spot (probe current, focus, astigmatism)

How to improve resolution:

- FEG SEM: higher brightness, stable and reproducible beam.
Applications of EBSD

EBSD patterns depend mainly on - **crystal structure** (symmetry) - **crystal orientation**

**Micro-crystallography**
Determination of zone-axis symmetries:
- 1, 2, 3, 4, 6, m, 2mm, 3m, 4mm ou 6mm
- \( \Rightarrow \) identification of the crystal point group.
- \( \Rightarrow \) Possible indeterminations: \( 1 / -1, 3 / -3, 4 / -4, 6 / -6 \)

Seldom used

**Orientation measurements**

**Phase identification / discrimination**
- Chemical analysis (EDS, WDS)
- Rough lattice parameter measurement (~5%, depend on calibration)
- Symmetry analysis, or investigation with a crystallographic data base.

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Data analysis and orientation representations
Directional solidification of a Ni-base alloy

Orientation image
The scale is coloured considering the misorientation between the points and the <001> direction oriented along the normal direction (i.e. Z)

Angle (<001>, ND>)
0° 55°

Pole figure
→ Stereographic projection

Phase identification

List of possible phases (determined previously by EDS, XRD...)

Acquire EBSP

Index...

Phase identified
1. Directional solidification of a Ni-base alloy

Angle (<001>, DS)

Cross section at 6 mm

Cross section at 2 mm

Angle (<001>, DS)

2. Identification of chi phase in a duplex steel

Austenite : fcc
Space group : Fm 3 m

Ferrite : bcc
Space group : I m 3 m

Chi phase (Cr$_6$Fe$_{18}$Mo$_5$) : bcc
Space group : I 4 3 m
3. $\text{GdNi}_5$ precipitates in a Ni-based superalloy

Phase map: Ni-alloy (blue), $\text{GdNi}_5$ (red, 7% of area)

4. $\text{GdNi}_5$ precipitates in a Ni-based superalloy

Orientation map (black = grain or phase boundary; red = twins)
$\text{GdNi}_5$ precipitates have all the same orientation