Electron Backscatter Diffraction (EBSD)

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Outline

- History
- Introduction
- Applications driven look at what EBSD can do (Advantages)
- Performance and Limitations
- Summary
P-Patterns


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)
Wide Angle Kikuchi Patterns

H. Boersch (1937) “About bands in electron diffraction”, *Physikalische Zeitschrift*, 38, 1000-1004

20 kV electrons using cylindrical photographic plates on cleaved, polished and etched surfaces of NaCl, KCl, PbS, CaCO₃, CaF₂, quartz, mica, diamond, Cu and Fe.
High Angle Kikuchi Patterns


Introduced the technique to the SEM and coined the term Electron Backscatter Diffractions Patterns (EBSP)
On-line Indexing – Zone Axes


User required to identify the (hkl)’s and the locations of three zone axes in a pattern.

1984 - EBSP in the SEM recorded on film
1987 - Film replaced by TV camera
Full Automation – EBSD Mapping


What is Microstructure

Conventional Measures of Microstructure
- Grain Size
  - Optical Metallography
- Grain Shape
  - Optical Metallography
- Chemistry
  - EDS
- Phases
  - Phase Contrast

What is missing?
- Orientation & Misorientation
Why does crystallographic orientation matter?

Because materials properties are “anisotropic”. We all know that the strength of wood varies with the direction of the grain. But the properties of a single crystal also vary with orientation.
Isn’t the anisotropy of the individual crystals averaged out in the bulk?

→ Not necessarily. It is true if the grains all have random orientations, but if the grains have similar orientations then the bulk material will exhibit anisotropy similar to the constituent crystal lattices.

Neutron Diffraction:  X-Ray Diffraction:  Electron Diffraction:
Volume Sampling  Surface Area Sampling  Point Specific Sampling
Electron Backscatter Diffraction
Bragg’s law: \( n\lambda = 2d_{hkl} \sin \theta \)
In an OIM scan the beam is stepped across the sample surface in a regular grid. At each point the EBSP is captured and automatically indexed and the orientation and other information recorded (such as the pattern quality of the EBSP, an indexing reliability factor, the secondary detector intensity and EDS data.)
EBSD Mapping Results
Orientation Imaging Microscopy
An image or pattern quality map can often reveal more structure than an SE image.
Aluminum Thin Films

Orientations can also be represented by color or by crystal
The acquired orientation information is three dimensional
Aluminum Thin Films

Similar orientations can be grouped together as grains
Aluminum Thin Films

This makes grain size analysis simple and fast. This data was collected in less than 2 minutes.
The MTF for an interconnect line stressed under electromigration conditions, as a function of crystallite morphology, is given by:

$$MTF = K(S/s^2) \log \left[ \frac{I_{111}}{I_{200}} \right]^3$$

where $S$ is the mean grain size and $s$ is the standard deviation of the log normal grain size distribution. $I_{111}$ and $I_{200}$ are the intensities at the centers of the 111 and 200 pole figures.

Texture – Grain Size – Mean Time to Failure (MTF)

High MTF Al Film ($I_{111} = 127$)

Low MTF Al Film ($I_{111} = 14$)

Texture – Grain Size – Mean Time to Failure (MTF)
Gold Wire Bonding

Wire bonding is when melted wire is attached to a bond pad using applied pressure, heat, and ultrasonic energy.
Gold Wire Bonding

The resultant grain structure significantly influences the bond strength. Here the grains and grain size are mapped.
In addition localized deformation and recrystallization can also be examined to optimize the bonding parameters.
In addition to grain size the grain shape can also be analyzed.
Titanium Grain Structure

The microstructure can be easily partitioned into differently shaped regions
Titanium Grain Structure

Once the grain shape is known, the orientation of the major axis can also be examined to understand growth mechanisms.
The microstructure of an extruded aluminum bar obtained from the top edge and center sections. A difference in machinability and performance was observed.
Grain maps show differing structure, but the number average grain sizes are similar: 5.86 microns for the top edge and 6.04 microns for the center.
Extruded Aluminum for Automotive Manufacturing

Changing the grain grouping tolerance from 5 degrees to 1 degree lowers the grain size: 4.15 microns for the top edge and 3.66 microns for the center.
Extruded Aluminum for Automotive Manufacturing

EBSD mapping makes it very easy to calculate grain sizes with differing tolerance angles.

This allows for a better understanding of the grain boundary mechanisms that are active.

It also helps for better predictive behavior with Hall-Petch type equations.
Consider a cubic crystal in a rolled sheet sample with "laboratory" or "sample" axes as shown below.

The Pole Figure plots the orientation of a given plane normal (pole) with respect to the sample reference frame. The example below is a (001) pole figure. Note the three points shown in the pole figure are for three symmetrically equivalent planes in the crystal.
Extruded Aluminum for Automotive Manufacturing

The pole figures for both sections are similar.
But the spatial distributions of orientations are very different. EBSD allows for statistical measurements of spatially specific orientation rapidly and accurately.
EBSD mapping can sometimes raise more questions than it answers. This technique has allowed new insight on exactly what is a grain.
Grain Boundaries

Having specific “special” grain boundaries can also improve material performance.
Grain Boundary Energy

Low angle boundaries have a lower energy than high angle boundaries. High angle boundaries cannot be produced through dislocation lines. Instead there is a transition zone where atomic bonds are not satisfied.
Special Grain Boundaries

However for some specific grain boundaries, the associated energy is lower due to some degree of order at the boundary. This lower energy makes this boundary act differently.
Through processing, you can increase the number of special boundaries and improve performance.
The grain size difference when included or excluding twin boundaries is very pronounced for copper films.

The grain map on the left shows the microstructure including twin boundaries, while the map on the right excludes the twins. There is nearly an order of magnitude difference in average grain size.
Twins and Grain Size in Copper Interconnect Lines

This same twin removal analysis can be used with interconnect lines. Notice that with this copper sample, the resultant bamboo microstructure after twin removal.
Grain Boundaries

Remember that a high angle grain boundary can be described as a rotation angle about a specific crystal axis.

Small misorientation or low angle grain boundaries can be thought of as dislocation arrays.
YBCO Superconductors

\( J_c \) decreases exponentially with increasing \( \theta \)

\[ J (MA/cm^2) \]

\[ \theta \text{ (GB angle)} \]

[001] tilt bicrystals thin PLD films

Verebelyi et al.
YBCO grain structure depends strongly on the deposition method.

**In situ**
- YBCO forms as material is deposited
  - Pulsed Laser Deposition (PLD)

**Ex situ**
- Precursor is deposited first, then converted to YBCO
  - Metalorganic deposition (MOD)

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**Columnar grain structure**

**Laminar grain structure**

- BaF₂-based precursor
- Conversion

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YBCO

substrate
Pulsed Laser Deposition (PLD) results in excellent epitaxy of the YBCO

Improved out-of-plane alignment!

Angle between RD/ND and nearest crystal axis or (011) direction for YSZ
Strong correlation between out-of-plane tilts of the YSZ and Ni-W

- EBSD allows grain-to-grain comparison of texture

  - Similar results using X-ray micro-diffraction
  - Improved grain alignment attributed to growth by ledge propagation
Pulsed Laser Deposition (PLD) results in excellent epitaxy of the YBCO

Angle between RD/ND and nearest crystal axis or (011) direction for YSZ

Improved out-of-plane alignment!

Excellent epitaxy!
Metalorganic Deposition (MOD) results in improved YBCO alignment

Out-of-plane

In-plane

Angle between RD/ND and nearest crystal axis or (011) direction for YSZ

10°
0°

100 µm

100 µm
In-Situ Recrystallization of Copper

Deformed structures have a larger number of dislocations and low angle grain boundaries. Recrystallization is the formation of new dislocation free grains at higher temperatures. In-situ observation allows direct measurement of the orientation changes that occur.
Recrystallization Kinetics of Copper

\[ f = 1 - \exp(-Bt^n) \]

JMAK Equation parameters can be determined to help understand recrystallization kinetics

<table>
<thead>
<tr>
<th>Temp</th>
<th>B</th>
<th>n</th>
</tr>
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<tbody>
<tr>
<td>165</td>
<td>9.0 \times 10^{-5}</td>
<td>1.29</td>
</tr>
<tr>
<td>170</td>
<td>9.2 \times 10^{-5}</td>
<td>1.38</td>
</tr>
<tr>
<td>175</td>
<td>9.4 \times 10^{-5}</td>
<td>1.48</td>
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Small Misorientations in Ferritic Steel

Low angle grain boundaries introduced during plastic deformation as imaged with both EBSD image quality and orientation maps.
Small Misorientations in Ferritic Steel

Orientation gradients are present within the ferrite grains. The constant grain rotation can be observed and measured with EBSD mapping.
Small Misorientations and Orientation Contrast in Ferritic Steel

Secondary electron (SE) image does not show the orientation contrast, while the forward scatter detector (FSD) shows it strongly.
The FSD intensity and the Mean Intensity IQ appear to be very sensitive to subtle variations in local orientation whereas the Hough IQ is very sensitive to the actual boundaries (or subboundaries) themselves.
Forward Scatter Detector (FSD) Contrast

High Angle Boundary ~50°

Small variations in orientation produce as much (if not more) contrast than large changes in orientation, e.g. at high angle grain boundaries.
The contrast mechanism varies with position from topographic to orientation to phase contrast. The image collected at 20 mm retraction has contrast similar to the reference BSE image.
Simultaneous EBSD and EDS Mapping

At each point in a prescribed grid collect crystallographic information using EBSD and chemical information using EDS.
Skutterudites are materials that have potential for improved thermoelectrical properties. EBSD helps understand the crystallographic growth mechanisms while EDS shows chemical changes that inhibit crystallization.
Phase Identification

EBSD

Fe₂TiO₄

EDS Spectrum

Fe₂TiO₄, Cubic, a=11.297
Fe-Ti-O, Cubic, a=11.31
Fe₄(TiO₄)₃, Tetragonal, a=9.3, c=9.5
FeTiO₃, Trigonal, a=5.0884, c=14.093
Fe₂TiO₄, Cubic, a=8.5352
Fe₃Ti₃O₁₀, Orthorhombic, a=7.789, b=10.008, c=3.74162
Fe₂TiO₃, Orthorhombic, a=5.026, b=5.174, c=7.245
Fe₂Ti₃O₉, Hexagonal, a=2.8667, b=4.5985

Candidate Phases

Database
Multi-Phase EBSD Mapping
Examination of EBSD patterns and EDS spectrum indicate that there are 7 primary phases in the region of interest:

Nickeline, Pyrrhotite, Chalcopyrite, Cobalite, Pentlandite, Biotite, and Carbon
Notice that while some phases are easily identified, there are regions where accurate phase differentiation has not occurred. This is due to crystallographically similar candidate phases.
Pattern Analysis of a Phase

Two of the candidate phases are cubic, and the third is pseudo-cubic.

Three of the candidate phases match.
This mineral, chalcopyrite, is tetragonal. When looking at the unit cell however, it consists of two stacked near-cubic unit cells with the same atomic positions, but different occupancy. Only the band intensities between a pseudo-cubic and tetragonal unit cell will differ, and are difficult to measure. Similar problems can occur when a cubic and hexagonal unit cell can be fitted to the same atomic lattice.
When chemical composition information is used however it is easy to identify the correct candidate phase.

<table>
<thead>
<tr>
<th>Phase Candidates</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickeline</td>
</tr>
<tr>
<td>Pyrrhotite</td>
</tr>
<tr>
<td>Chalcopyrite</td>
</tr>
<tr>
<td>Cobalite</td>
</tr>
<tr>
<td><strong>Pentlandite</strong></td>
</tr>
<tr>
<td>Biotite</td>
</tr>
<tr>
<td>Carbon</td>
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</tbody>
</table>

Fe, Ni, S
To ambiguously analyze an multi-phase sample using chemical filtering, EDS count limits are determined for each candidate phase.
Collect EDS & EBSD data

Set up an elemental filter for each phase present

Assign each scan point a crystallographic phase(s) based on the chemical filter

Index EBSPs at each scan point using the phase information assigned to each scan point

Multi-Phase EBSD-EDS Mapping
Here the results on combined EBSD + EDS analysis show improved phase differentiation.
Multi-Phase EBSD-EDS Mapping

This allows for accurate orientation determination and inter- and intra-phase orientation relationship analysis.

It also saves time through less processing.
Multi-Phase EBSD-EDS Mapping

When using chemistry it no longer matters how many phases are in your material.
Angular Resolution

1° is the value generally stated in the community, but our experience would put it closer to 0.3°.
OIM of Pd, smallest grains are 20 nm wide
RESOLUTION FACTORS

Sample:
Deformed vs. Non Deformed, Atomic Number, Sample Preparation

Microscope:
Gun type, acceleration voltage

Camera:
CCD, sensitivity, speed

It should be noted while grains as small as 8nm have been imaged, these grains are at the tail end of a distribution with an average grain size of approximately 50nm
Large Area Capability – Nanometers to Centimeters
Limitations – Sample Tilt

SEM vacuum chamber

Sample

Limitations – Sample Tilt
Limitations – Sample Tilt

70 nAmps, 25kV, $\gamma = 0$

- Exposure
- Image Quality

Exposure (seconds)

Image Quality

Specimen Tilt (degrees) $\beta$
Region of Contrast Reversal

Nickel
\( \beta = 30^\circ, \gamma = 0^\circ, 25 \text{ kV}, \sim 70 \text{ nAmps} \)

\( \alpha \sim 40^\circ \)
Limitations – Material State

Deformed materials produce more diffuse patterns. As the degree of order decreases within the diffraction interaction volume decreases so does pattern quality. Obviously amorphous materials will not produce EBSD patterns.
Limitations – Sample Preparation

You can take something like a quarter from your pocket and click around and find EBSD patterns.
Limitations – Sample Preparation

As you search around the surface for EBSD patterns, a wide range of emotions are experienced.

This is easier than EDS

Is that one pattern or two?

I should have bought a Coke

Is this some secret amorphous alloy to prevent counterfeiting?

There is often a lot of searching though to find a decent pattern for analysis.
However when trying to collect orientation mapping data from this surface, not many points could be analyzed.
Limitations – Sample Preparation

After proper preparation though, it is quite easy to analyze this type of materials.
While EBSD image formation is not completely understood, it is strongly dependent on crystal lattice quality at the specimen surface.

Typically EBSD patterns come from the top 20-50 nm of material.

This distance will vary with SEM acceleration voltage and specimen atomic number.

Sample preparation is often the most important factor in maximizing the performance on an EBSD system.
Summary

- EBSD has become a well established microstructural analytical technique.
- EBSD mapping can be used to explore the processing – microstructure – properties relationships.
- EBSD can be used on most crystalline materials
- Grain size, sample tilt, and sample preparation are the primary limitations of the technique.
Acknowledgements

- Stuart Wright, John Carpenter, Andy Fisher, Rene de Kloe – EDAX
- David Field – Washington State University
- Matthew Feldman – University of Wisconsin
- Thanks for listening