Understanding the TSL EBSD Data Collection System

27-750
Texture, Microstructure & Anisotropy
A.D. Rollett

With thanks to: Harry Chien, Lisa Chan, Bassem El-Dasher, Gregory Rohrer, Stefan Zaefferer (Max-Planck, Düsseldorf)

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Overview

• Understanding the diffraction patterns
  – Source of diffraction
  – SEM setup per required data
  – The makeup of a pattern

• Setting up the data collection system
  – Environment variables
  – Phase and reflectors

• Capturing patterns
  – Choosing video settings
  – Background subtraction

• Image Processing
  – Detecting bands: Hough transform
  – Enhancing the transform: Butterfly mask
  – Selecting appropriate Hough settings
  – Origin of Image Quality (I.Q.)
Overview (cont’d)

• Indexing captured patterns
  – Identifying detected bands: Triplet method
  – Determining solution: Voting scheme
  – Origin of Confidence Index (C.I.)
  – Identifying a solution in multi-phase materials

• Calibration
  – Physical meaning
  – Method and need for tuning

• Scanning
  – Choosing appropriate parameters

Questions (1)

• Why do we need to position the specimen at the eucentric point?
• Why does the specimen need to be tilted at a steep angle of incidence (70°) to the electron beam?
• Why is it so important to avoid contact between a specimen and the phosphor screen?
• What is the function of the phosphor screen?
• What is the characteristic appearance of a diffraction pattern in EBSD?
• Why is specimen surface preparation so important?
• What are reflectors and how do you choose them?
• What is the Hough Transform?
• What does “binning” refer to (in connection with Hough Transforms)?
• What is a “sharpening mask”?
• What does “frame averaging” do for acquisition?
Questions (2)

• What does background subtraction do?
• What is image quality?
• What are the coordinates of the image after the Hough transform has been applied?
• Why is the Hough transform effective for detecting lines?
• What are interzonal angles (in the context of an EBSD diffraction pattern)?
• What is the “confidence index” and how is it calculated?
• Why is it important to have a flat surface for the specimen?
SEM Schematic Overview

- All students using this system need to know how to use SEM. It is recommended that all users take SEM courses offered by the MSE department.
Sample Size effect

• All the samples needs to be prepared (polished) before EBSD data collection. As most samples are mounted before polishing, it is recommended to use smaller size mount (1.25 inch preferred)
• It is difficult to work with large mounted samples (with 1.5 inch) in OIM as the edge of the mount may touch either the camera or the SEM emitter after tilting
• It is critically important that the specimen does NOT touch the phosphor screen because this is easily damaged
Diffraction Pattern-Observation Events

• OIM computer asks Microscope Control Computer to place a fixed electron beam on a spot on the sample
• A cone of diffracted electrons is intercepted by a specifically placed phosphor screen
• Incident electrons excite the phosphor, producing photons
• A Charge Coupled Device (CCD) Camera detects and amplifies the photons and sends the signal to the OIM computer for indexing
Electron backscatter diffraction (EBSD)

- Incoherent incoming wave field (produced by inelastic scattering)
- Coherent outgoing wave field (backscatter diffraction)

A typical EBSD pattern (Niobium, 15 kV)
EBSD-based orientation microscopy

- By nature a “quantitative” technique!
- Spatial resolution: lateral ~ 20 ... 80 nm, depth ~ 10 nm
- Angular resolution: conventional 0.5°, special > 0.01°
- Measured area: μm² ... cm²

BSEI of a partially recrystallised IF steel

S. Zaefferer: Introduction to EBSD
Measurement of macroscopic textures using EBSD

**Advantages:**
- Direct measure of orientation distribution
- No corrections for absorption, defocussing etc. required
- No peak overlaps in multiphase materials

**Challenges:**
- Statistical representativeness
- Spatial resolution on highly deformed or sub-μ material

Texture field in a hot-rolled Si-steel
Spatial resolution of EBSD

primary electron energy: 15 kV

length: 80...100 nm

half width: 10...17 nm

depth: 5...10 nm

15kV  7.5kV

D. Steinmetz, S. Zaefferer (2010), Mat. Sci. Tech. 26, 640
A word on spatial resolution of SEM and TEM

see, e.g., Zaefferer, Ultramic. (2007)
An example data set

Cu-Zr HPT-deformed & annealed
420 x 420 x 60 voxels; approx. 40,000 grains
Step Size 50 nm
Courtesy A. Khorashadizadeh, MPIE
Applications of 3D orientation microscopy

- 3D morphology
- 5-parameter grain boundary characterization
- Calculation of GND densities using the Nye-tensor approach
- Coupling with modelling

Interfaces colored according to their miller indices

S. Zaefferer: Introduction to EBSD
Grain Boundaries

3D EBSD: serial sectioning & reconstruction with software QUBE (Konijnenberg, Bruker Nano)

5 rotational parameters: $\omega$ (1), $<uvw>$ (2), $(hkl)_{gb}$ (2)

Grain boundary normal vectors

Tilt boundary

Twist boundary

$53^\circ$ $<323>$

$57^\circ$ $<110>$
Phase and structure determination

FeNdB(-O )Material, Courtesy of T. Woodcock, IfW Dresden

EDS Counts:
O-Red
Fe-Green
Nd-Blue

Phases:
Nd2O3 - Red
NdO - Green
FeNdB - Gray

trigonal
Unknown
cubic
• The Quanta FEG has 3 operating vacuum modes to deal with different sample types:
  – High Vacuum
  – Low Vacuum
  – ESEM (Environmental SEM)

• Low Vacuum and ESEM can use water vapours from a built-in water reservoir which is supplied by the user and connected to a gas inlet provided.

• Observation of outgassing or highly charging materials can be made using one of these modes without the need to metal coat the sample.
Vacuum Status

- **Green**: PUMPED to the desired vacuum mode
- **Orange**: TRANSITION between two vacuum modes (pumping / venting / purging)
- **Grey**: VENTED for sample or detector exchange
The Tool Bar

Surface Positioning detector (automatically detect working Distance)

Image Refreshing rate
Turtle: lower refresh rate (higher resolution)
Rabbit: Higher refresh rate (lower resolution)

Automatic Contrast and Brightness (short key F9)
Eucentric Position

Note that eucentric position only occurs when the working distance is 10.
Diffraction Patterns-Source

- **Electron Backscatter Diffraction Patterns** (EBSPs) are observed when a fixed, focused electron beam is positioned on a tilted specimen.
- The spread in direction is important for generating diffraction at all possible angles that satisfy the Bragg condition.
- To obtain a high yield of backscattered electrons, the specimen is tilted between 55-75°, where 70° is considered **ideal** because it maximizes the yield of backscattered electrons in the direction of the scintillation screen.
- The backscattered electrons escape from 30-40 nm underneath the surface, hence there is a diffracting **volume**.
- Note that \( \delta x \approx 2 \) times spot size and \( \delta y \approx 2.5 \) to 3 times spot size.
Kossel Cones

• As mentioned above, the spread in direction of diffusely scattered electrons is important for generating diffraction at all possible angles.
• The range of locally (elastically) scattered electrons means that a large range of input directions exist that satisfy the Bragg condition.
• At typical energies used in SEMs, the diffraction angle is small, so the Kossel cones intersect the detector as (almost) straight lines.

Diffraction Patterns-Anatomy of a Pattern

• There are two distinct features:
  • Bands
  • Poles

• Bands are intersections of diffraction cones that correspond to a family of crystallographic planes
• The small Bragg angles mean that the lines of intersection of the cones with the scintillation screen are effectively straight lines
• Band widths are proportional to the inverse interplanar spacing
• Intersection of multiple bands (planes) correspond to a pole of those planes (vector)
• Note that while the bands are bright, they are surrounded by thin dark lines on either side
Diffraction Pattern-SEM Settings

- Increasing the Accelerating Voltage increases the energy of the electrons and increases the diffraction pattern intensity.

- Higher Accelerating Voltage also produces narrower diffraction bands (a vs. b) and is necessary for adequate diffraction from coated samples (c vs. d).

- Larger spot sizes (beam current) may be used to increase diffraction pattern intensity.

- High resolution datasets and non-conductive materials require lower voltage and spot size settings.

- For insulators (most ceramics), consider using a low-vacuum “environmental” SEM.
System setup-Material data

- In order for the system to index diffraction patterns, three material characteristics need to be known:
  - Symmetry
  - Lattice parameters
  - Reflectors
- “Reflector” means a particular set of lattice planes ("hkl" values)
- Information for most materials exist in TSL .mat files
- “Custom” material files can be generated using the ICDD powder diffraction data files; try searching http://www.crystallography.net/new.html to find information. For example, the MTEX software reads CIF files to determine crystal structure and symmetry.
- Symmetry and Lattice parameters can be readily input from the ICDD data
- Reflectors with the highest intensity should be used (4-5 reflectors for high symmetry; up to 12 reflectors for low symmetry)
System setup - Material data

- Enter appropriate material parameters
- Reflectors should be chosen based on:
  - Intensity (higher intensity is better)
  - The number per zone
Pattern capture - Background

Live signal

- The background is the fixed variation in the captured frames due to the spatial variation in intensity of the backscattered electrons.
- Removal is done by averaging 8 frames (SEM in TV scan mode).
- Note the variation of intensity in the images. The brightest point (marked with X) should be close to the center of the captured circle.
- The location of this bright spot can be used to indicate how appropriate the Working Distance is. A low bright spot = WD is too large and vice versa.

Averaged signal
Pattern capture-Background Subtraction

- The background subtraction step is critical as it “brings out” the bands in the pattern
- The “Balance” slider can be used to aid band detection. Usually a slightly lower setting improves indexing *even though it may not appear better to the human eye*
Hough: Accumulator Diagram (1)

- The most basic idea behind the Hough transform is to take each point in the image and “spread it out” along the line defined by the transform in Hough space (angle vs. radius).
Effectively, this transformation converts each pixel of the image space into a sinusoidal curve in the Hough space. The calculated $\rho$ value is rounded to the closest pixel $\rho_j$. The intensity of the pixels $(\theta_j, \rho_j)$ that are part of the sinusoidal curve are augmented by the intensity of the corresponding pixel $(x_i, y_i)$ in the image space. The accumulation of these intensities give rise to peaks in the Hough space which corresponds to the $\theta$ and $\rho$ coordinates of the bands in the image space.

$$\rho = x_i \cos \theta_j + y_i \sin \theta_j$$
“From the definition of the Hough transform, each pixel in the image space is transformed into a sinusoidal curve in the Hough space. The curve represents all the possible uni-dimensional lines that can pass through that pixel in the image space. A few lines are drawn in the figure above with their corresponding position in Hough space represented by circle markers. Only a small fraction of the lines are fully contained in the band, the rest of the lines cross it, but most of their pixels are outside the band. If this geometrical construction is repeated for another pixel, B, of the band L, the same result is obtained. In the figure above, the lines passing by B and their equivalent representation in Hough space using triangular marker. All the lines or curves related to pixel B are drawn as dashed lines. The lines inside of band L and passing by pixel B are the same lines that are also passing by pixel A. In Hough space, these lines end up having the same coordinates $\theta$ and $\rho$, forming a peak. The intersection of the sinusoidal curves therefore corresponds to the lines that are fully inscribed inside the band in the image space. The intensity at this intersection is higher than the background because of two interlinked reasons: a) the sinusoidal curve of the pixels in the band have a higher intensity that the one of the pixels outside of it; and b) the intensity of many sinusoidal curves is added at this intersection.”
Detecting Patterns-Hough Transform

- A modified Hough Transform is used, and transforms the pattern so that it has a reference frame that is akin to polar coordinates.
- Lines in the captured pattern with points \((x_i, y_i)\) are transformed into the length of the orthogonal vector, \(\rho\) and an angle \(\theta\).
- The **average** grayscale of the line \((x_i, y_i)\) in Cartesian space is then assigned to the point \((\rho, \theta)\) in Hough space.

**Cartesian space**

**Transformed (Hough) space**

\[
\begin{align*}
I: & \ 0 \leq \rho \leq n ; \ 0 \leq \theta \leq \pi/2 \\
II: & \ 0 \leq \rho \leq n ; \ \pi/2 < \theta \leq \pi \\
III: & \ -n \leq \rho < 0 ; \ 0 \leq \theta \leq \pi/2 \\
IV: & \ -n \leq \rho < 0 ; \ \pi/2 < \theta \leq \pi
\end{align*}
\]

\(2n = \text{Hough bin size}\)
Detecting Patterns-The Hough of one band

- Since the patterns are composed of bands, and not lines, the observed peaks in Hough space are a collection of points and not just one discrete point
- Lines that intersect the band in Cartesian space are on average higher than those that do not intersect the band at all
Setting up binning/mask

- Due to the shape of a band in Hough space, a multiplicative mask can be used to intensify the band grayscale.
- Three mask sizes are available: 5 x 5, 9 x 9, 13 x 13. These numbers refer to the pixel size of the mask.

<table>
<thead>
<tr>
<th>-2</th>
<th>-6</th>
<th>-8</th>
<th>-6</th>
<th>-2</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>3</td>
<td>-1</td>
<td>3</td>
<td>0</td>
</tr>
<tr>
<td>1</td>
<td>8</td>
<td>20</td>
<td>8</td>
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<tr>
<td>0</td>
<td>3</td>
<td>-1</td>
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<td>0</td>
</tr>
<tr>
<td>-2</td>
<td>-6</td>
<td>-8</td>
<td>-6</td>
<td>-2</td>
</tr>
</tbody>
</table>

- A 5 x 5 block of pixels is processed at a time.
- The grayscale value of each pixel is multiplied by the corresponding mask value.
- The total value is added to the grayscale value at the center of the mask.
- Note that the sum of the mask elements = zero.
Detecting Patterns-Hough Parameters

Binned Pattern Size = Hough resolution in $\rho$

<table>
<thead>
<tr>
<th>Smaller size</th>
<th>Larger size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Use with broad bands</td>
<td>Use with narrower bands</td>
</tr>
<tr>
<td>Use for faster speed</td>
<td>Use with low symmetry materials</td>
</tr>
</tbody>
</table>

I.Q. = Average grayscale value of detected Hough peaks

<table>
<thead>
<tr>
<th>More peaks</th>
<th>Less peaks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Use with low symmetry</td>
<td>Use with cubic materials</td>
</tr>
<tr>
<td>Increases the number of solutions</td>
<td>Decreases the number of solutions</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Symmetry</th>
<th>Symmetry</th>
</tr>
</thead>
<tbody>
<tr>
<td>More peaks</td>
<td>Less peaks</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Smaller distance</th>
<th>Larger distance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Closely spaced bands</td>
<td>Sparsely distributed bands</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Smaller mag.</th>
<th>Larger mag.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Band intensity is low</td>
<td>Band intensity is high</td>
</tr>
</tbody>
</table>
Indexing Patterns-Identifying Bands

• Procedure:
  – Generate a lookup table from given lattice parameters and chosen reflectors (planes) that contains the inter-planar angles
  – Generate a list of all triplets (sets of three bands) from the detected bands in Hough space
  – Example (cubic): $100^111 = 54.7^\circ$; $111^110 = 35.3^\circ$; $100^110 = 45^\circ$
  – Calculate the inter-planar angles for each triplet set
  – Since there is often more than one possible solution for each triplet, a method that uses all the bands needs to be implemented
Indexing Patterns-Settings

Tolerance = How much angular deviation a plane is allowed while being a candidate

<table>
<thead>
<tr>
<th>Lower Tolerance</th>
<th>Larger Tolerance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Use if many bands are tightly bunched</td>
<td>Use with poorer patterns</td>
</tr>
<tr>
<td>Higher speed (eliminates possible solutions)</td>
<td>Lower speed (more possible solutions)</td>
</tr>
</tbody>
</table>

Band widths: check if the *theoretical* width of bands should be considered during indexing

If multi-phase indexing is being used, a “best” solution for *each* phase will be calculated. These values assign a weight to each possible factor:
- Votes: based on total votes for the solution/largest number of votes for all phases
- CI: ratio of CI/largest CI for all phases
- Fit: fit for the solution/best (smallest) fit between all phases

The indexing solution of the phase with the largest Rank value is chosen as the solution for the pattern
Indexing Patterns-Voting Scheme

- Consider an example where there exist:
  - Only 10 band triplets (i.e. 5 detected bands)
  - Many possible solutions to consider, where each possible solution assigns an $hkl$ to each band. Only 11 solutions are shown for illustration.
- Triplets are illustrated as 3 colored lines.
- If a solution yields inter-planar angles within tolerance, a vote or an “x” is marked in the solution column.
- The solution chosen is that with most number of votes.
- Confidence index (CI) is calculated as:

$$CI = \frac{\# \text{ votes of } S1 - \# \text{ votes of } S2}{\text{number of band triplets}} = \frac{10 - 4}{10} = 0.6$$

- Once the solution is chosen, it is compared to the Hough and the angular deviation is calculated as the fit.

<table>
<thead>
<tr>
<th>Solution #</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>11</th>
</tr>
</thead>
<tbody>
<tr>
<td>Band triplets</td>
<td>S1</td>
<td>S1</td>
<td>S1</td>
<td>S1</td>
<td>S1</td>
<td>S1</td>
<td>S1</td>
<td>S1</td>
<td>S1</td>
<td>S1</td>
<td>S1</td>
</tr>
<tr>
<td>S1 (solution w/most votes)</td>
<td>S2 (solution w/ 2$^{nd}$most votes)</td>
<td># votes</td>
<td># votes</td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
</tbody>
</table>

Confidence index $= 0.6$
Scanning

- The selection of scanning parameters depends on some factors:
  - Time allotted
  - Desired area of coverage (scan size)
  - Desired detail (step size)

- To determine if the scan settings are acceptable time-wise you must:
  - Start the scan
  - Use a watch and note how many patterns are solved per minute (n)
  - Divide the total number of points by n to get the total time

- To decide if the step size is appropriate for your SEM settings, use the following rough guide:

<table>
<thead>
<tr>
<th>Spot Size Selection</th>
<th>Approximate Spot Size</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>5 nm</td>
</tr>
<tr>
<td>2</td>
<td>10 nm</td>
</tr>
<tr>
<td>3</td>
<td>20 nm</td>
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<tr>
<td>4</td>
<td>50 nm</td>
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<tr>
<td>5</td>
<td>100 nm</td>
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<tr>
<td>6</td>
<td>200 nm</td>
</tr>
<tr>
<td>7</td>
<td>500 nm</td>
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Supplemental
**Cubics:**

interplanar angles

<table>
<thead>
<tr>
<th>{h_2k_2l_2}</th>
<th>{h_1k_1l_1}</th>
<th>100</th>
<th>110</th>
<th>111</th>
<th>210</th>
<th>211</th>
<th>221</th>
<th>310</th>
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</thead>
<tbody>
<tr>
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<td>0</td>
<td>90</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>110</td>
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<td>60</td>
<td>90</td>
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<tr>
<td>111</td>
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<tr>
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