Introduction to EBSD

EBSD is the abbreviation for Electron Backscatter Diffraction. EBSD is a technique that allows crystallographic orientations, misorientations, texture trends and grain boundary types to be characterized and quantified on a sub-micron scale in the Scanning Electron Microscope. EBSD is a surface sensitive technique with data being acquired from a depth of the order of tens of nanometers. Thus *sample preparation* is important. Using advanced electron optics and in particular FEGSEM, high spatial resolution is possible.

Forward scattering electron imaging greatly assists EBSD investigation by using diffraction contrast to make grains and features in the microstructure more visible.

Electron Backscatter Diffraction Patterns (EBSP) are formed when an electron beam strikes a steeply inclined crystalline sample. <u>Bragg diffraction</u> takes place and the scattered electrons radiate away from the interaction volume in the sample and fall on the EBSD hardware. The steeply inclined collection geometry is used in order to achieve good contrast in the diffraction patterns.

To Summarize:

- Incident electrons scatter between crystal planes to produce cones of diffracted electrons
- These intersect a phosphor screen to produce pairs of lines
- Each pair of lines, known as Kikuchi lines represent a plane in the crystal
- Where lines intersect represent crystal directions or 'zones'
- The EBSP contains the angular relationship between planes, the symmetry of the crystal and orientation information
- · Comparing one pattern to another allows misorientations to be determined

Collection Geometry

Diffracted electrons only escape from a depth in the order of a few tens of nanometers deep from the sample surface. At low tilt angles the total interaction volume close to the surface is very small compared to the interaction volume deep within the material. Consequently, at zero or low tilt, the proportion of diffracted electrons in the overall electron yield may be so low as to be undetectable. Tilting the sample improves the diffracted component to background yield ratio by increasing the volume of near surface material excited. Thus EBSD is generally carried out at approximately 70 degrees tilt.



Distances shown are arbitrary

70.5 degrees is the accepted angle for performing EBSD because:

- This angle produces a good yield of diffracted electrons and thus contrast in the pattern.
- The angle permits a reasonable view of the sample which can viewed either tilted, or be made to appear at zero tilt by using tilt correction in imaging.
- EBSD can be performed at other angles, but reducing the tilt causes a significant fall-off in pattern contrast. This
 may be tolerable if the mean atomic number of the material under investigation is high, i.e., materials with high
 electron scattering factors produce comparatively higher diffracted electron yields and so higher contrast in the
 EBSP.

Forward Scattered Electron Imaging

A high proportion of the electrons scattered during EBSD carry imaging information. Because of the high angle of tilt dictated by the <u>collection geometry</u> required for EBSD, many electrons are scattered forward and down towards the bottom of the phosphor screen. These electrons carry similar information to the conventional backscattered electron signal. Using Forward Scattered Electron (FSE) imaging diffraction contrast is enhanced and the resultant signal makes the presence of individual grains easy to identify. The forward scattered electron signal produced is therefore ideal for EBSD investigations. However, the user may use any electron signal as required for the reference image.



Some examples:



Secondary Electron Image Forward Scattered Electron Image

Sample: Nickel



Secondary Electron Image

Forward Scattered Electron Image

Sample: Austenitic Stainless Steel

About forward-scattered electron detectors

By using extra signals from diodes arranged around the EBSD phosphor panel, the detector can provide orientation contrast images from a wide range of materials.



The top two diodes detect backscatter electrons and normally show atomic number contrast. Only one diode is usually needed. The side and lower diodes detect forward-scattered electrons and show orientation contrast and topography. The side diodes are useful where the detector is not inserted perpendicular to the sample's tilt axis. The detector can produce four or six channels of data. If the detector has four channels, the left-hand top diode and side diode share a connection, and the right-hand top diode and side diode share a connection.

The following examples from a polyphase geological sample show the different signals from each diode. The lower diodes accentuate any surface topography.

The image on the left shows atomic-number contrast, and uses one upper diode. The image on the right shows orientation contrast, and uses two lower diodes.



Understanding shadow effects in a forward-scattered electron image

When looking at images generated by the forward-scatter detection (FSD) diodes, it is important to consider exactly what you are seeing.



The electron image on the left comes directly from the microscope. The image on the right is from two detector diodes, but their slightly different positions create two shadows in a "V" shape in the combined image. This can give a false impression of the real artefacts.

Mapping

EBSD provides point orientation analysis on a sub-micron scale. A natural progression from this is to generate maps that show graphically in some manner the distribution of crystal orientations, localized orientation effects and even the presence and distribution of different phases in a microstructure. This is achieved by the EBSD software taking control of the microscope beam, such that a grid of points at some chosen spatial resolution can be defined. These positions are then visited point by point during mapping acquisition.

An electron image is first acquired as a reference image, and the area to be mapped defined. During mapping acquisition the beam is moved to each position in the grid, halted long enough for a pattern to be acquired and then moved to the next point, when the process is repeated. The pattern acquired at each point is processed in software to find the orientation at that point.

Color Key for Crystal Orientation Maps (COMs)

The *Inverse Pole Figure* can be conveniently used as a color key for *Crystal Orientation Maps* (COMs). This is because the figure has three corners, and individual orientation measurements relate to a single point plotted in an Inverse Pole Figure. By assigning colors such as red, blue and green to each of the corners of the figure, with smooth gradation of colors in between, any given measurement can be related to a distinct color in the COM:



Inverse Pole Figure Color Key

Thus it is a simple matter to visualize for a cubic material that:

- The face of the crystal {100} lies parallel to the surface when red is shown in the Sample Normal Map
- The edge of the crystal {110} lies parallel to the surface when green is shown in the Sample Normal Map
- The corner of the crystal {111} lies parallel to the surface when blue is shown in the Sample Normal Map

For instance:



of Sample Normal COM

It is simple to visualize which crystal planes lie parallel to the surface of the sample. This type of display does not show in a single view how crystals are rotated with respect to the surface. Therefore, it is usual to show at least two COMs, or even all three views, i.e., Sample Normal, Transverse Direction and Rolling or Longitudinal Directions. In this way, grain rotations can be visualized. Further, this can be helpful in visualizing <u>texture</u>.

Electron Backscatter Diffraction Patterns (EBSP's

Pattern Formation

EBSD occurs due to Bragg diffraction

Bragg's law: nl = 2d sinq



where n is the order of reflection, I is the electron wavelength, d is the lattice spacing, and q is the Bragg angle, the angle of incidence of the electron.

During EBSD, electron scattering takes place between the crystal planes such that Bragg diffraction can occur from a high proportion of the crystal planes present. In this manner diffraction occurs from a multiplicity of crystal planes. This effect was observed in the SEM and described by Kikuchi. Thus lines/bands in the EBSP are described as Kikuchi lines or Kikuchi bands.



Even though diffraction occurs throughout the electron interaction volume in the sample, which leads to the generation of other electrons, i.e. Primary and secondary electrons, the resultant flux of diffracted electrons is an extremely surface sensitive effect. Diffracted electrons undergo deflection and collisions within the bulk of the material and therefore only those generated near the surface carry diffraction information. Dependent on the atomic number of the material under investigation, the diffraction information is obtained from a region of the order of a few tens of nanometers deep.

For this reason <u>sample preparation</u> is important in obtaining good diffraction patterns. Any residual strain induced in the surface by preparation methods will act to reduce the <u>pattern quality</u>, or completely suppress it altogether. Further, the <u>collection geometry</u> used contributes to the contrast available in the pattern

Because most of the diffracted electrons generated do not escape, the proportion of diffracted electrons compared to the overall yield of electrons from the sample surface is very small. Thus the majority of electrons that fall on the phosphor screen contain no diffraction information, and cause the screen to fluoresce with a background distribution of electrons. The diffraction pattern is therefore superimposed on the background, and consequently often very faint or indistinct, depending on the atomic number of the material. Heavy atomic number materials produce more contrast diffraction patterns due to higher scattering factors, compared with lighter atomic number materials.

EBSD is a comparatively weak effect which requires enhancement to make the patterns more visible. This can be simply achieved in practice by performing background subtraction on the patterns.

Hough transform

The EBSP contains many Kikuchi bands at different angles and positions. These relate to the orientation of the crystal that formed the pattern. In order to be able to index the pattern and obtain the orientation of the crystal, it is first necessary to detect the band positions using a 'Hough Transform'.

The Hough Transform provides a suitable technique for deriving the parameters of a straight line and thus the band positions in an EBSP.

A line can be specified by the angle of its normal and the distance from an origin. Every point on that line can be transformed to a single point in Hough Space, using the equation:



Schematic Representation.

Note that three points are indicated on the line in the image diagram, which could represent three pixels in the digitized image of an EBSP. Using the equation, each point is transformed into a sinusoidal curve. These curves in Hough space intersect at a point. This single point in Hough space describes the position of the line in the image. This can be shown with an actual example:



Hough Transform of a single line.

Thus the same process can be applied to an EBSP:



Hough Transform of Germanium

Peak detection is used to find the maxima of the Hough Transform, which are then related back to real space. The distances between the spots are computed to give the angles between the zones in the pattern and thus *index* the EBSP.

What Information does the EBSP Contain?

The EBSP contains the angular relationship between planes, the symmetry of the crystal and orientation information. Each Kikuchi band in the EBSP relates to a physical plane in the crystal under the electron beam. Where the bands meet in the pattern is indicative of a crystal <u>'zone'</u> or <u>crystal direction</u>. Using the <u>Hough Transform</u> the pattern can be <u>'indexed'</u> to find the orientation of the crystal under the beam.



Bright 'Kikuchi' bands correspond to planes in the crystal lattice. The width of a band is dependent upon the electron wavelength and lattice plane spacing.

The relationship is given by the Bragg equation: I = 2d sinq

Data Presentation for EBSD

EBSD data can be presented using the standard methods developed and popularized by the x-ray diffraction community for visualizing orientation and misorientation information.

Crystallography predominantly deals with information which is three dimensional, and thus many of the plots employed were developed to render that information in a two dimensional form which can be presented on paper. No single plot developed for x-ray diffraction can render all of the information that could be of possible interest at once, in a convenient form. Thus a variety of plots have been developed over the years, some being widely adopted, with others being less popular.

Different plots present the same raw data either processed in different ways, or with respect to different axes such that the data can be inspected for different aspects: texture trends, alignments, special grain boundary types, etc. With the advent of EBSD, new approaches of visualizing the data is possible.

It is common to use the specimen axes as the reference axes:



The following types of data presentation are in common use:

Pole Figures

Pole Figures

A Pole figure is a 'Stereographic Projection' in which orientations are plotted as two dimensional projections. In a Pole figure, the poles, i.e. the normal to a lattice plane for a chosen family of planes, are plotted relevant to the sample reference axes. The sample reference axes are the Longitudinal or Rolling direction, the Transverse direction and the 'Normal' direction.



In order to visualize the process employed, it is convenient to consider a reference sphere which is intersected by an 'equatorial plane'. An individual orientation or grain can be considered to reside within the sample, placed at the center of the equatorial or 'projection' plane. The axes of the sample are superimposed on the projection plane.

The plot can be made for different families of poles. For instance it is common to plot Pole Figures for the family of 100 poles, the 110 family of poles, or the 111 family of poles. For example, it would be common to refer to a '100 Pole Figure'. It is possible to plot Pole Figures for any chosen poles.

Considering the poles that have been chosen to display (for this example the <100> poles have been chosen), it is possible to project lines corresponding to the chosen poles out to the hemisphere above the projection plane. Where these poles meet the reference sphere, lines are projected to the bottom of the figure. Where these projected lines intersect the equatorial plane, a point is plotted.



Thus for a 100 Pole Figure, each measurement will result in multiple points in the equatorial or projection plane:



For each measurement on the sample, the different orientations alter the projected positions of the poles and hence different positions are plotted for the corresponding points in the projection plane. Thus a highly populated Pole Figure (one with many different measurements plotted) is useful for evaluating the orientation distribution:



The Sample Normal Direction is represented by the center of the pole figure.

To Summarize:

- Grain orientation data is plotted with respect to the sample axes in a Pole Figure.
- Different Poles can be chosen and plotted.
- This type of plot can indicate whether there is a preferred crystallographic orientation present.
- Multiple points are plotted for each measurement.

The same data can be plotted in an *Inverse Pole Figure*, which uses crystal directions as the reference axes.

Inverse Pole Figures

An Inverse Pole Figure is a stereographic projection, similar to a Pole Figure. However, the principal difference is that the orientations are plotted relative to crystal axes (and not the sample axes as is the case in a Pole Figure). An Inverse Pole Figure is plotted for a specific, chosen sample direction, which must be stated for the plot to be meaningful.

The diagram below shows the approach used to plot an Inverse Pole Figure, which is similar to that used for a Pole Figure. Because the symmetry of the crystal is repeated in the circular projection plane of an Inverse Pole Figure, it is common to extract and display a reduced portion of the whole plot, as shown.

The resultant shape displayed contains all of the orientation information without any repetition of the data, which would otherwise be confusing.



Because of the way the data is processed in an Inverse Pole Figure, each orientation is displayed as a single point, rather than a number of points as is the case with a Pole Figure. Inverse Pole Figures are commonly plotted for the Sample Normal Direction (ND), Rolling or Longitudinal Direction (RD/LD) and the Transverse Direction (TD).



Considering the above Inverse Pole Figure plotted for the Sample Normal, the plot displays the orientation of the crystal directions with respect to the sample normal, i.e., that the observed orientation has the [001] direction close to the sample normal. For a cubic material, that implies that the (001) plane for that particular measurement lies close to parallel to the sample surface.



This example shows an Inverse Pole Figure containing a number of measurements for a cubic material.

The distribution of points above show a significant tendency for some grains to be aligned with the [101] crystal direction parallel, or close to parallel, to the sample normal, i.e. the closer that the points are to the [101] corner of the figure, the closer the [101] directions in the grains measured are aligned with respect to the sample normal.

Similarly, it indicates that a significant proportion of (101) planes favour alignment parallel, or close to parallel, to the surface of the material.

This observation may be very significant with regard the processing or properties of a material.

To Summarize:

- An Inverse Pole Figure shows orientations plotted with crystal directions as the axes for the figure.
- The plot is specific to a chosen sample direction, which must be stated.

- A single point relates to each measurement.
- This type of plot is useful for visualizing the alignment/trends of planes/directions with respect to chosen sample directions.
- No rotational information is shown (A Pole Figure shows grain rotations).