STUDIES ON THE ACCURACY OF ELECTRON BACKSCATTER DIFFRACTION MEASUREMENTS

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ABSTRACT

Automated orientation measurement on a local basis is now widely accepted for characterization of materials. The technique relies upon indexing of electron backscatter diffraction patterns in a scanning electron microscope. In order to exploit the available information, it is important to understand its limitations with respect to accuracy. Experiments were carried out to measure orientation fields from a silicon single crystal. The orientation dispersion was 1°. Disorientation correlation maps revealed anisotropy in the spatial variation in measured orientation.

INTRODUCTION

The practice of Automated Electron Backscatter Diffraction (EBSD) has progressed substantially in recent years. Not only have significant advances been made in the hardware and software used to capture electron backscatter patterns on an automatic basis, but textural analysis of the data has also progressed. It is now possible to perform automated feature recognition on micrographs and combine feature location with orientation determination (Adams et al., 1999). This has permitted novel approaches to the extraction of grain boundary properties to be developed and, for example, grain boundary energy to be mapped as a function of boundary type. In addition to computer-automated acquisition of microstructural information, new methods of interrogating EBSD data sets have been generated. The data is available as a discretized sampling of the orientation field on a section plane, or map, such that each point in the map is associated with a three parameter quantity that represents the orientation at that point. Typically it is differentiated in order to perform – in effect – edge detection, i.e., grain boundaries. Since the boundaries are sharp in the sense that their width is, typically, considerably smaller than the point to point spacing of the EBSD map, the criterion that is used is the height of a one parameter projection of the change in orientation between adjacent points (i.e., the disorientation angle) not the gradient in

orientation. Other interesting information concerning microstructure, however, can be derived by differentiation, such as the dislocation tensor. The dislocation tensor contains information on the geometrically necessary dislocation content of the material (Sun et al., 2000). This requires a differentiation of the orientation field to obtain the dislocation tensor before assigning densities to individual dislocation types. Equally interesting to materials scientists is the organization of orientation over large length scales. Surface roughening, for example, is known to be caused by clusters of similarly oriented grains that are organized into long bands (Valkonen, 1987). A more subtle effect, but equally likely to affect material performance, is ordering of orientation. By analogy to chemical ordering in a binary alloy, where A atoms might alternate with B atoms, one orientation might alternate with another in a regular array. A more useful analogy might be with magnetic ordering for which many types of order exist because the magnetization is a vector quantity. Note that orientations can be represented as vector quantities, as for example the Rodrigues-Frank vector (Frank, 1988) as described in the previous chapter by Rajan. In addition to antiferromagnetism, which resembles ABABA… chemical ordering, helical patterns of magnetic ordering occur, for example, in some materials. One probe of such patterns in orientation fields is the Disorientation Correlation Function (DCF). This is calculated from the EBSD data in an analogous manner to an autocorrelation function where the disorientation between two points takes the place of an intensity (in an image). The DCF is a useful tool for visualizing correlations in orientation at distances larger than the grain size. In aluminum 6022 alloy sheet, for example, it has been used to show that texture banding develops during plane strain deformation (Lee et al., 1998) The spacing of the texture banding was found to be proportional to the periodicity of the surface roughness and was approximately a factor of four smaller (Lee et al., 1999). The validity and utility of any such quantity derived from EBSD data is, therefore, critically dependent on the accuracy with which experimental measurements can be made. This paper presents results of EBSD mapping of single crystals and DCF maps to explore orientation correlation.

DEFINITION OF DISORIENTATION CORRELATION FUNCTION (DCF)

A scalar measure of orientation is needed for constructing correlation maps and it is convenient to use rotation angle between the orientations at two points for this purpose. Among all the symmetrically equivalent misorientations, the smallest in magnitude, i.e., the smallest angle in an axis-angle representation is called the disorientation (Δg), which is generally regarded as the physically meaningful misorientation. The minimum angle of rotation, θ_{dis}, is given by

$$\theta_{dis} = \left|\Delta g_{AB}\right| = \min\left[\cos^{-1}\frac{1}{2}\left(trace\left[S_B^{crystal}\cdot g_B\left(S_A^{crystal}\cdot g_A\right)^{-1}\right]-1\right)\right] \quad (1)$$

$S^{crystal}$ is a member of the symmetry subgroup, G, representing crystal symmetry operators (in this paper, the 24 proper rotations comprising the cubic point group 432), g_A is the lattice orientation in matrix form associated with the point A at position (x,y), and g_B is the lattice orientation associated with the point B at (x+a, y+b). Strictly speaking, the disorientation is obtained by finding the particular pair of symmetry operators, with or without an inversion of the misorientation (i.e., B to A, instead of A to B) that places the rotation axis in a specific unit triangle in addition to minimizing the rotation angle, i.e., the fundamental zone. The disorientation correlation function describes the general dependence of the orientation of one position with the orientation at another position. It provides information about the spatial relation and dependence of the orientation data. Hence, it is related to the Auto-Correlation

Function (ACF) or Areal Auto-Correlation Function (AACF) described for analyzing surface topographic data (Stout et al., 1993). AACF and DCF are analogous to each other. AACF uses the product of the residual surface height as a weighting function, whereas the DCF uses the disorientation.

The average disorientation with a fixed relocatable vector corresponds to a modified version of the orientation correlation function (OCF) introduced by Adams et al. (1987), and discussed in a later chapter. The function indicates the misorientation distribution, or orientation dispersion. Intensity in the DCF map, is the average minimum misorientation of two-point pairs separated by a given vector **r**, which can be represented as (*a, b*). When average disorientation is calculated with a small separation vector (*a, b*), most of the pairs of points at the ends of the vector fall in the same grain resulting in a deep well profile around the origin. The well structure at the center of the DCF is detrimental for visualizing the profiles in the high misorientation region. Therefore, the integrals for average disorientation correlation are replaced by sums over the pixelized EBSD data and the result, $f\,(G(a,b))$, is truncated as defined below.

$$f(G)=\begin{cases} p \quad if \quad G<p \\ G(a,b) \quad if \quad G\geq p \end{cases} \tag{2}$$

$$G(a,b)=\frac{\sum_{A=1}^{N-b}\sum_{B=1}^{M-a}\left|\Delta g_{AB}\right|}{(M-a-1)(N-b-1)} \tag{3}$$

$$\text{p = p (clustering, resolution, texture)} \tag{4}$$

In equation (2), p is the minimum average misorientation angle that is chosen to be included in the output and G is the average disorientation between two points related by relocatable separation vector. Thus, the choice of the cutoff p is governed by intensity of the texture, degree of texture clustering and the desired resolution of the DCF profile (as shown in equation (4)), and G is given by equation (3), where N and M are the number of pixels in an input EBSD data file. Note that the average, G, incorporates fewer points as the length of the vector (a,b) increases. For EBSD data from polycrystalline samples, p is typically set equal to 15°, i.e., the criterion for a high angle boundary. For the single crystal scans presented here, $p = 0$ because the average misorientations are very small and no central "well" is present in the DCF map such as occurs for the average grain shape in polycrystalline samples. M and N determine the resolution of the DCF map. Since EBSD data sets contain texture information on a discrete lattice, the equations are also formulated in discrete form. Thus, the intensity in the DCF map, I(a,b), is given by:

$$I(a,b)=f(G(a,b)) \tag{5}$$

where (a,b) are the coordinates of a point in DCF space and also define a vector that separates a pair of points in an EBSD map with the origin of the DCF space at the tail of the vector, and f is the disorientation average. We take advantage of the center of symmetry such that I(-a,-b) = I(a,b), because $|\Delta g_{AB}| = |\Delta g_{BA}|$.

ACCURACY OF ORIENTATION MEASUREMENTS

The development of accurate information on correlations in orientation clearly depends on the availability of high quality data sets. Hence, the accuracy of orientation determination was investigated. Measurements performed with scanning electron microscopes (SEMs) may be inaccurate because of a variety of user errors. Indexing the diffraction pattern at a given point depends upon recognition of a set of low-index zones in the pattern. The user must, therefore, correctly specify the lattice geometry for the material being studied as well as the geometry of the specimen with respect to the electron beam and the camera (used to image the diffraction pattern). Each diffraction pattern is captured in pixelized form, and then image analysis is performed to index the pattern. Therefore, it is necessary for users to have a complete picture of how images in a SEM system depend on changes in instrument settings. We performed a systematic study in order to determine the reliability and repeatability of EBSD measurements, and the limitations of the SEM. Besides the major parameters such as beam voltage and spot size, accurate measurement of the in-plane spread of orientation also depends on variability caused by the mounting and tilting of the specimen. Overall, optimizing these parameters to determine the best result in terms of in plane spread of orientation is an ultimate goal. Similar studies were performed recently and it was shown that the accuracy of measurements could be improved (Humphreys, 1999a; Prior, 1999; Humphreys et al., 1999b)

RESULTS

Experimental measurements

EBSD scans were performed in beam control on a silicon single crystal wafer with a [001] normal on a XL-40 Philips FEG SEM. The aim was to analyze the spread of orientation in the scan plane. Scans were performed with two different resolutions and scan areas using an accelerating voltage of 15kV with the specimen at a 70° tilt angle. The large scan has an area of 500x500 μm^2 with a ten μm step size and the smaller one has an area of 100x100 μm^2 with a two μm step size. Theoretically, the surface should have an orientation of (315°,0°,90°), expressed in terms of Bunge's Euler angles, with a mosaic spread of no more than 0.1°. As a result of measurement errors, however, there is an apparent variation of orientation in the plane of scan. This is demonstrated by converting the measured Bunge Euler angles to axis-angle pairs and plotting the absolute value of angle distribution within the (001) plane with respect to an arbitrary reference frame. In other words, the mean value of the angle is subtracted so that the histogram plot has a zero mean. Mathematically this can be expressed as $\omega' = \omega - \langle\omega\rangle$, where ω is the orientation angle. From Figure 1 we can see that there is approximately a ***1 degree*** spread in orientation within the plane based on the full width half maximum of the distribution. In addition, Figure 2 shows the dependencies of the scatter in orientation on beam voltage and spot size. There is a little variation in orientation spread with voltage. Some variations are observed with spot size and the intermediate spot size gives the smallest orientation spread.

In order to analyze the effect of orientation spread within the (001) plane systematically, different scan sizes were chosen between 100x100μm^2 and 1x1mm^2. In addition, the in-plane rotation dependence of orientation spread was of interest. Therefore, we scanned each sample in two different configurations. One configuration is defined as the reference position and the other configuration was obtained by a 45-degree rotation about the (001) plane normal. Standard deviations and mean values of these scans are shown in Table 1, which were obtained over the whole area of the scans. As shown in Figure 3, the standard deviation of the orientation spread decreases with decreasing scan size. The high deviation observed for

the 250x250 μm scan on the 45° rotated sample was probably an anomalous result, although it illustrates the ease with which additional errors can be introduced.

In order to show that no strong spatial correlations exist within the sample plane, pole figures are plotted in Figure 4 for two different randomly chosen regions within a 750x750 μm^2 scan area. The two regions have identical pole figures. We applied the same calculations to different regions with similar results. Therefore, it can be deduced that these different areas within the same sample plane are statistically independent from each other.

Table 1. Orientation spread for different scan areas.

	Reference Position		45 Degree Rotation	
Scan Size (μm^2)	Mean	Standard Dev.	Mean	Standard Dev.
1000x1000	117.77	0.9236	70.03	0.7959
750x750	117.50	0.6748	70.09	0.6613
500x500	117.54	0.3083	74.46	0.2624
250x250	117.49	0.2177	74.26	0.6327
100x100	117.54	0.1577	74.47	0.1205

DCF Calculations

The calculations of DCF profiles presented here were performed at a lower resolution than the EBSD data sets used as input because of computational limitations. Typical EBSD data sets have upwards of 40,000 points which would allow M=N≤200. The number of calculations required for a DCF profile, however, scales as M^2*N^2, i.e., the fourth power of the chosen resolution. Thus, the DCF profiles presented here used M=50 and N=50. Calculating the coordinates of each point and associating the orientation of the nearest point in the EBSD data set determine each point used for the DCF calculation. This interpolation method is adequate for these examples in which the (linear) density of EBSD points is ofg order four times that of the corresponding DCF. It is important to be aware of the spatial sampling frequency implicit in the choice of resolution: if the DCF cell size (e.g., max(a)/M) is too large compared to the grain size, then the DCF will not reveal the expected information on grain shape and misorientation in the vicinity of the average grain. Since we performed our analysis on a silicon single crystal wafer, grain size and shape discussions are not relevant. For other details of the experimental approach, see Lee et al. (1999). In order to increase the computation speed, the quaternion formalism is used instead of axis/angle pairs. Euler angles (ϕ_1,Φ,ϕ_2), which are obtained from EBSD measurements, are converted to quaternions (Morawiec and Pospiech, 1989; Sutton and Balluffi, 1995).

$$\begin{pmatrix} q_1 \\ q_2 \\ q_3 \\ q_4 \end{pmatrix} = \begin{bmatrix} \sin\frac{\Phi}{2}\cos\left[\frac{(\phi_1-\phi_2)}{2}\right] \\ \sin\frac{\Phi}{2}\sin\left[\frac{(\phi_1-\phi_2)}{2}\right] \\ \cos\frac{\Phi}{2}\sin\left[\frac{(\phi_1+\phi_2)}{2}\right] \\ \cos\frac{\Phi}{2}\sin\left[\frac{(\phi_1+\phi_2)}{2}\right] \end{bmatrix} \tag{6}$$

$$\cos\Phi = \left(q_4^2+q_3^2\right)-\left(q_1^2+q_2^2\right)$$
$$\chi = \sin\Phi/2 \tag{7}$$

$$\sin\phi_1 = (q_4 q_2 + q_1 q_3)/\chi$$
$$\sin\phi_2 = (q_1 q_3 - q_4 q_2)/\chi \tag{8}$$

$$\cos\phi_1 = (q_4 q_1 - q_2 q_3)/\chi$$
$$\cos\phi_2 = (q_4 q_1 + q_2 q_3)/\chi \tag{9}$$

where q is the quaternion. Adoption of the quaternion formalism in place of matrix representation of orientations accelerated the speed of misorientation calculations because it permits the disorientation angle to be determined with fewer floating point operations than with other representations of orientation. The reason for this is as follows. Symmetry operations are straightforward with quaternions and they are shown in Table 2 for the cubic crystal system.

Table 2. Symmetry operations with quaternions in the cubic crystal system.

Axes	Directions
six diads about <100>	±(1,0,0,0), ±(0,1,0,0), ±(0,0,1,0)
three four-fold about <100>	±1/√2(±1,0,0,1), ±1/√2 (0, ±1,0,1), ±1/√2(0,0, ±1,1)
six diads about <110>	±1/√2 (±1,1,0,0), ±1/√2 (0,1, ±1,0), ±1/√2 (±1,0,1,0)
four triads about <111>	±1/2 (±1,1,1,1), ±1/2 (1,-1, 1,1), ±1/2 (1,1,-1,1), ±1/2 (-1,-1, 1,1), ±1/2 (-1,1,-1,1), ±1/2 (1,-1,-1,1), ±1/2 (-1,-1,-1,1)

Sutton and Balluffi (1995) quote the 24 equivalent representations in the 432 point group. Two quaternions, q_A and q_B combine to form a third quaternion, q_C as follows, where q_B follows q_A:

$$\begin{aligned} q_C &= q_A q_B \\ q_{C1} &= q_{A1}q_{B4} + q_{A4}q_{B1} - q_{A2}q_{B3} + q_{A3}q_{B2} \\ q_{C2} &= q_{A2}q_{B4} + q_{A4}q_{B2} - q_{A3}q_{B1} + q_{A1}q_{B3} \\ q_{C3} &= q_{A3}q_{B4} + q_{A4}q_{B3} - q_{A1}q_{B2} + q_{A2}q_{B1} \\ q_{C4} &= q_{A4}q_{B4} - q_{A1}q_{B1} - q_{A2}q_{B2} - q_{A3}q_{B3} \end{aligned} \tag{10}$$

Symmetry arguments allow for a very simple procedure for finding the disorientation based on quaternions. The objective is to find the quaternion that places the axis in a specified unit triangle (e.g. 0<u<v<w) with the minimum rotation angle (maximum fourth component). If one considers the action of the diads on <100>, the result is obtained that $\pm(q_1,q_2,q_3,q_4)$ is equivalent to $\pm(q_4,q_3,-q_2,-q_1)$, which is also equivalent to $\pm(-q_3,q_4,q_1,-q_2)$, which is also equivalent to $\pm(q_2,-q_1,q_4,-q_3)$. This means that one can place the fourth component in any other position in the quaternion. Since the first three components correspond to the rotation axis, [uvw], we know that we can interchange any of the components q_1, q_2 and q_3, and we can change the sign of any of the components. These rules taken together allow us to interchange the order and the sign of all four components of the quaternion. If this is done so as to have $q_4 > q_3 > q_2 > q_1 \geq 0$, i.e., all four components positive and arranged in increasing order, then the only three variants that need be considered are as follows because we are seeking the minimum value of the rotation angle.

$$\begin{matrix} (q_1 & q_2 & q_3 & q_4) \\ (q_1 - q_2 & q_1 + q_2 & q_3 - q_4 & q_3 + q_4)/\sqrt{2} \\ (q_1 - q_2 + q_3 - q_4 & q_1 + q_2 - q_3 - q_4 & -q_1 + q_2 + q_3 - q_4 & q_1 + q_2 + q_3 + q_4)/2 \end{matrix} \quad (11)$$

Therefore with operations involving only changes of sign, a sort into ascending order, four additions and a comparison of three numbers, the disorientation can be identified. The contrast is with the use of matrices where each symmetrically equivalent variant must be calculated through a matrix multiplication and then the trace of the matrix calculated; each step requires an appreciable number of floating point operations, as discussed above. On a DEC Alpha workstation, 50x50 DCF maps can be calculated in a few minutes.

Figure 5 presents four DCF maps calculated from the EBSD data for the scans of 100x100 μm (45° rotated), fig. 5a, 500x500 μm (reference position), fig. 5b, 750x750 μm (reference position), fig. 5c, and 1000x1000 μm (45° rotated), fig. 5d. The maps show that there is a mild gradient in misorientation across the scan area such that points that are far apart have larger misorientations between them than points close together. The gradient in misorientation is anisotropic in the sense that there is little variation in misorientation for horizontal vectors for small scan areas, fig. 5a. On the tilted specimen, this is the direction in which the working distance is constant as the electron beam is rastered over the specimen surface. For large scan areas, however, the line of minimum gradient in misorientation is tilted up to approximately 30°, fig. 5d. The reason for this tilt is unclear at this time.

DISCUSSION

The results obtained in this work are in good agreement with those obtained by other authors. As noted by Humphreys (1999), there is little sensitivity to accelerating voltage when a field emission electron source is used unless extremely low or high values are employed. The optimum spot size appears to be the intermediate one between 3 and 4 nm. Even when user errors such as pattern center determination are eliminated, the accuracy of the technique is clearly limited by inherent machine broadening. The variation in apparent orientation from a highly perfect single crystal is of order 1° at full width, half maximum (FWHM). This dispersion is related to the finite width of the diffraction maxima, i.e., the lines in the Kikuchi maps, and their pixelized representation for image analysis (Lassen, 1998). Higher accuracy would require a larger separation between specimen and camera. This would, however, reduce the solid angle subtended by the specimen at the camera (for a fixed size of scintillation screen) and thus decrease the number of zones visible in the diffraction pattern. Since the indexing method requires a minimum number of low-index zones to be present in the diffraction pattern, there is an effective upper limit for the specimen-camera separation.

Crude tests of spatial variations in measured orientation, e.g., pole figures of subsets of a scan, do not show any systematic variation. When a more general technique such as the DCF is applied, mild gradients in (average) misorientation are revealed. These gradients may be related to variations in the geometry of the incident beam with respect to the specimen, which is tilted at a steep angle (70°) in order to maximize the efficiency of diffraction. Note that the average misorientation in each DCF map is related to the dispersion in measured orientation at each point. As the standard deviation of orientation rises with increasing scan area, so too the average misorientation increases in the DCF maps. One important limitation of EBSD arises in connection with the investigation of dislocation structures. Subgrain boundaries often have misorientations of two degrees or less. The inherent 1° scatter in orientation means that it is inadvisable to use a misorientation criterion of less than 2° for identifying boundaries.

SUMMARY

Various sources of inaccuracy in the EBSD technique have been discussed. In addition to errors introduced by the user, there is an inherent machine broadening such that the minimum measurable dispersion in orientation is of the order one degree. Application of the Disorientation Correlation Function reveals gradients in (average) misorientation across a scan obtained on a single crystal. The accuracy with which orientation can be measured decreases (monotonically) as the scan area is increased.

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FIGURE CAPTIONS

Figure 1. Orientation spread within the (001) plane: (a) Area of 500x500μm^2 with 10μm step size and (b) Area of 100x100μm^2 with 2μm step size.
Figure 2. (a) Spot Size Dependence of in-plane orientation spread (3~1.5 nm, 4~3.0 nm, 5~5.8 nm) and (b) Voltage Dependence of in-plane orientation spread (10 kV, 25 kV).
Figure 3. Scan size and in-plane rotation dependence of orientation spread.
Figure 4. Pole figures for Single Crystal Silicon sample, (a) and (b) are two different randomly chosen regions within a 750x750 μm^2 scan area.
Figure 5. DCF maps for the scans of a) 100x100 μm (45° rotated), b) 500x500 μm (reference position), c) 750x750 μm (reference position), and d) 1000x1000 μm (45° rotated). Note that the size of each DCF map is the same as the scan size. The contour labels are in units of 0.01°.