Chapter 9

Collection, Processing, and Analysis of Three-Dimensional EBSD Data Sets

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9.1 Introduction

Three-dimensional (3D) characterization methods are required to completely determine microstructural descriptors such as the true shape and size of features, the number of features, and the connectivity between these features. Experimental methods to characterize microstructure in 3D have undergone dramatic improvements in the past decade, and there now exists a host of methodologies that are capable of determining 3D microstructural information, ranging from counting individual atoms to imaging macro-scale volumes. The state of the art for this field has been reviewed recently in a Viewpoint Set for Scripta Materialia (Spanos 2006).

This chapter focuses on the specific topic of experimental interrogation and analysis of microstructural features such as grains or precipitates in 3D that includes crystallographic orientation data. Currently there are two main experimental pathways to collect such information. Serial sectioning experiments are more commonplace, but consume the sample as part of the experiment; while X-ray methods are nondestructive, but typically require the use of high-intensity X-ray sources. For the X-ray methods, there are a handful of groups world-wide that are working towards spatially-resolved crystallographic analyses of grain structures in 3D using high-intensity X-ray systems (Schmidt et al. 2004; Lauridsen et al. 2006; Budai et al. 2004, 2008; Lienert et al. 2007). These methods have a significant advantage in that the sample remains intact after analysis, allowing for the possibility of time-dependent studies of microstructural changes due to thermal or mechanical input. Nonetheless, these experiments require the high brilliance of a synchrotron source, which puts significant restrictions on the general applicability of the methods.

Therefore, the focus of this chapter is on the collection, processing, and analysis of grain-level data using more universally-accessible serial section experiments, where electron backscattered diffraction (EBSD) maps are used as an integral component of the characterization method. The incorporation of crystallographic maps enables a straightforward approach to defining the individual grains or precipitates that compose aggregate assemblies, and also allows for orientation-based data analysis—for example, determining the disorientation between neighboring grains, or assessing the complete character of grain boundaries (Saylor et al. 2004a; Kim et al. 2006).

The authors of this chapter have assumed that the reader has knowledge of most basic aspects of crystallography and is aware of some of the commercial packages pertaining to EBSD data collection and analysis. We recommend the first volume of this book to anyone interested in additional background information.

9.2 Data Collection

Serial sectioning experiments are comprised of two main tasks—sectioning and data collection—which are repeated until the desired volume of material has been interrogated. The sectioning process involves the removal of a known volume of material, usually with

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the requirement that the newly-exposed surface be as planar as possible. The amount of material removed, i.e., the serial sectioning thickness, is primarily determined by the size-scale of the microstructural features that are to be examined. A typical rule of thumb is to acquire a *minimum* of 10 sections per average feature if feature shape is to be determined. The section thickness is often a compromise between the desire for high-fidelity data and the constraints of both personnel and instrument time to collect the data sets. Note that the serial section thickness is usually uniform throughout the experiment to simplify data processing, but fiducial marks can be added to track differences due to intentional variations or experimental error.

After sectioning, the surface of interest is characterized through standard imaging methods, or other mapping methods such as EBSD. There are a couple of factors that one must consider in order to successfully incorporate EBSD data into a serial sectioning experiment. Obviously, the material sectioning process must be capable of producing a "low damage" surface that allows for the EBSD patterns to be successfully indexed. For example, mechanical sectioning procedures often produce some damage, and so multiple processing steps may be required in order to minimize surface damage at the completion of the sectioning process, such as the combination of mechanical polishing followed by vibratory, chemical, ion, or electropolishing. Also, another practical concern is that EBSD data collection typically adds significant overhead to the time required to complete an experiment, because of the relatively slow data acquisition speed compared to standard electron imaging methods. These time constraints can be addressed by collecting complementary data, for example, both EBSD maps and SEM images, where EBSD data is collected less frequently than the image data (i.e., every *n*th slice) (Rowenhorst et al. 2006).

The combination of serial sectioning via mechanical polishing and EBSD mapping was initially demonstrated by Wall et al. (2001) and Saylor et al. (2001), and later by groups at the Naval Research Laboratory to examine grain aggregate microstructures (Lewis et al. 2006) and martensite precipitates (Rowenhorst et al. 2006). One significant advantage of using mechanical sectioning methods to perform this experiment is that the dimensions of the serial sectioning surface can be very large (>1 mm). This allows for the characterization of microstructural features that are typically larger than a few microns in scale. Also, mechanical polishing equipment is usually available in most materials science laboratories, and the procedures for producing high-quality surfaces are well understood. A possible disadvantage is that it may be difficult with mechanical polishing to consistently obtain serial section thickness below a few hundred nanometers in scale, although there are examples in the literature of successful serial mechanical polishing even at this scale (Lund and Voorhees 2002). Also, at present this experiment must be performed manually.

In addition to mechanical sectioning, ion-based micromilling using Focused Ion Beam-Scanning Electron Microscopes (FIB-SEMs) have also been used for EBSD-inclusive serial sectioning studies (Groeber et al. 2004, 2006; Zaafarani et al. 2006). The high precision of FIB milling can produce a finer serial sectioning thickness than can be performed by mechanical polishing, but the limited milling speed of FIB microscopes usually results in a much smaller analysis volume. The surface damage produced by FIB milling using 30 kV Ga ions can be moderate enough to allow for EBSD pattern collection from some materials such as Ni and Fe. It is worth noting that the process for serial sectioning and EBSD pattern collection has been automated on two of the commercial microscope systems. Note that this technique is covered in more detail in the chapter in this book by Zaefferer.

9.3 Processing Strategies

9.3.1 Registration and Alignment of Sections

It is often the case that misalignment between sections occurs during the sectioning experiment and can cause difficulties in the subsequent analysis of the dataset. These misalignments can be minimized through the use of fiducial marks coupled with manual and automated alignment tools during the data collection process, but can rarely be totally avoided. This section will address techniques used to adjust sections after the collection process has completed. First, the generalized alignment solution is discussed and then the special condition involving EBSD data is presented. It is important to note that manual sectioning

processes are typically more susceptible to misalignment errors. Automated processes, such as FIB-OIM data collection, can incorporate procedures for very precise positioning of the sample following movements after sectioning and imaging. In the case of the FIB-OIM, image recognition software has been demonstrated to be able to place the sample to within 0.1 microns and 0.1 degrees of the desired position, removing nearly all misalignment (Groeber et al. 2006).

The most straightforward registration procedures involve applying simple translations and rotations to sections to improve section-to-section alignment. In the general case, this can be done with images and image processing techniques like least-square difference fitting or image convolution. In these procedures, images are translated (generally at multiples of the pixel size) or rotated in the plane of the image until either a minimum difference or maximum product between pixels in consecutive images is obtained.

Often the registration of separate serial sections requires more than simple translations and rotations of the sections to gain proper alignment of the image stack. This is most prevalent when combining data collected by different methods, such as micrographs from a light optical microscope and EBSD scans from a SEM, where there may be different coordinate orientations, and scaling between pixels. However, these same techniques can be applied to remove distortions or drift during data collection within a set of serial EBSD scans.

The alignment of the images begins by identifying a number of point features (examples would include the centers of grains, or triple junctions). One then must choose a reference frame to which the image is aligned, either a SEM/EBSD image that is relatively free of drift, or an optical image (hereby noted as X', Y'). The points in the EBSD scan (X, Y) should then be brought into coincidence with the reference frame. The transformation from the EBSD image to the reference optical image is given by:

$$\begin{bmatrix} X' \\ Y' \\ 1 \end{bmatrix} = T \begin{bmatrix} X \\ Y \\ 1 \end{bmatrix}, \tag{9.1}$$

where T is a two-dimensional transformation matrix of the form:

$$T = \begin{bmatrix} t_{11} & t_{21} & t_{31} \\ t_{12} & t_{22} & t_{32} \\ 0 & 0 & 1 \end{bmatrix}.$$

In the most general case, T describes an affine transformation. An affine transformation is a transformation that preserves colinearity and the ratio of distances, but not necessarily angles. Affine transformations can be considered as linear combinations of image rotation and independent scaling and shearing in each direction, combined with a translation. The components of the affine transformation matrix, T, can be solved using the pseudo inverse matrix (a least squares fit to a system of linear equations).

However, by allowing shearing of the images, it is not possible to independently determine the rigid rotation of the two coordinate frames, which is necessary to correct the measurement of the crystallographic orientations between each section. Therefore a limited transformation matrix can be used that allows only independent scaling in the x-direction and y-direction, S; translations in the x-direction and y-direction, P; and finally an image rotation normal to the sectioning plane, R. Thus:

$$T = RPS, \tag{9.2}$$

where:

$$R = \begin{bmatrix} \cos(\theta) & -\sin(\theta) & 0 \\ \sin(\theta) & \cos(\theta) & 0 \\ 0 & 0 & 1 \end{bmatrix}; P = \begin{bmatrix} 1 & 0 & P_x \\ 0 & 1 & P_y \\ 0 & 0 & 1 \end{bmatrix};$$
$$S = \begin{bmatrix} S_x & 0 & 0 \\ 0 & S_y & 0 \\ 0 & 0 & 1 \end{bmatrix}.$$

The terms in the transformation matrix are then determined by a least-squares optimization.

As mentioned, errors associated with translations and rotations in the plane orthogonal to the sectioning direction can be corrected in image space by processes such as least-square difference fitting, image convolutions, and Fourier transforms. However, these methods may not utilize all of the data in an image equally, and image conditions can vary significantly between sections. EBSD data for consecutive sections enables the use of all data points equally, and generally provides a more invariant parameter to follow between sections.

The misorientation between a data point and the corresponding data point on a neighboring section can be calculated for all points in a given section. A parameter (Ψ) can then be created to define the amount of misalignment between the two sections. The sections can be translated to locate the position of minimum Ψ. The definition of Ψ is given by:

$$\Psi(k) = \sum_{j=0}^{y_{\max}} \sum_{i=0}^{x_{\max}} \Psi(i, j, k), \tag{9.3}$$

where $x_{\max}$ and $y_{\max}$ are the total number of data-points in the corresponding directions, and $\Psi(i, j, k)$ is given by:

$$\Psi(i, j, k) = \begin{cases} 1 & \text{if } M[P(i, j, k), P(i, j, k-1)] \geq 5^{\circ} \\ 0 & \text{otherwise} \end{cases},$$

where $M[P(i, j, k), P(i, j, k-1)]$ is the misorientation between points *i,j,k* and *i,j,k*-1.

Following completion of the alignment procedures, the data set exists in the form of a 3D volume made up of voxels. Each voxel contains at a minimum the following characteristics: the x, y, and z positions within the array, and quantities that describe the local orientation, such as the three Euler angles $\{\phi_1,\Phi,\phi_2\}$.

9.3.2 Segmentation of Grains

Segmentation of individual grains is necessary to allow for the measurement of each grain as a separate feature. Additionally, identifying and separating microstructural constituents provides the ability to investigate features removed from their surroundings. Grain segmentation is greatly aided by the quantitative orientation information provided by the EBSD maps, as local orientation information is the most direct means to group voxels and thus reduce issues of image contrast, thresholding, and feature identification. During segmentation, grains are identified as groups of voxels that share a similar orientation. An algorithm for identifying these groups of voxels has been presented previously (Groeber et al. 2008; Bhandari et al. 2007). Commercial analysis packages (TSL, HKL) likewise use a similar approach to identifying grains. The major steps of the algorithm are outlined in this section.

The initiation of each identified grain is the selection of a seed voxel. Generally, it is a useful idea to select a voxel that has been deemed to be of "good" quality. Quality is defined by the EBSD data collection software and refers to the sharpness of the pattern, which is often correlated with confidence in the assigned orientation. Usually the highest quality data tends to lie within the center of a grain, and these voxels serve as a reliable point to begin grain segmentation. A grain is then defined as the set of voxels contiguous to and with the same orientation as the seed voxel. The requirement of the voxels to have the same orientation (within a defined tolerance) is based on the fact that all regions within a grain should share a similar orientation. A list of voxels assigned to the grain is created, which initially contains only the seed voxel. The voxels that neighbor the seed voxel are checked to determine whether they have a similar orientation. The misorientation is measured to evaluate the orientation difference between the seed voxel and each of its neighbors. The value of the misorientation is given by the following equation:

$$\theta = \min \left| \cos^{-1} \left(\frac{tr(O_c g_A g_B^{-1} O_c) - 1}{2} \right) \right|, \tag{9.4}$$

where O_c is the crystal symmetry operator and g_A and g_B are the rotation matrices of voxel A and B, given by:

$$g_i = \begin{pmatrix} \cos\varphi_1 \cos\varphi_2 - \sin\varphi_1 \sin\varphi_2 \cos\Phi & \sin\varphi_1 \cos\varphi_2 + \cos\varphi_1 \sin\varphi_2 \cos\Phi & \sin\varphi_2 \sin\Phi \\ -\cos\varphi_1 \sin\varphi_2 - \sin\varphi_1 \cos\varphi_2 \cos\Phi & -\sin\varphi_1 \sin\varphi_2 + \cos\varphi_1 \cos\varphi_2 \cos\Phi & \cos\varphi_2 \sin\Phi \\ \sin\varphi_1 \sin\Phi & -\cos\varphi_1 \sin\Phi & \cos\Phi \end{pmatrix},$$

where $(\varphi_1,\Phi,\varphi_2)$ are the Euler angles of the voxel. If the misorientation is less than the defined tolerance (i.e., $\sim 5^{\circ}$), then that voxel is added to the list of voxels of the grain being segmented. Each voxel on the list undergoes the process of checking its neighboring voxels until no new voxels are added to the list. After the list is complete, a new seed point is generated and the voxel assignment is repeated for the next grain. An option during segmentation is to terminate the process when no unassigned voxels remain above a

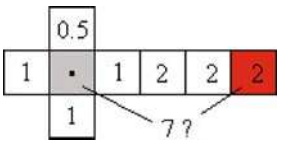

Fig. 9.1 Schematic illustrating the possibility of accumulated misorientation. If a tolerance of 5° was chosen to segment voxels, the *red* and *grey* voxels would be assigned to the same grain, due to the lack of a 5° boundary between immediately neighboring voxels. However, the *red* and *grey* voxels could be misoriented by as much as 7°. If a second criterion was used, where the misorientation between an immediate neighbor voxel (the *red* voxel) and the initial seed voxel (the *grey* voxel) also had to be below 5°, then accumulated misorientation would be limited to 5° as well

data quality tolerance. This ensures that no grains can be formed that include only low quality voxels. Techniques for subsequent treatment of the remaining low quality points will be discussed in the section on data clean-up.

Note that the relative degree of misorientation that "defines" a grain should be carefully considered. For example, if misorientation is determined by comparing any given voxel with only its immediate neighbors, rather than also comparing this voxel with the original seed point, it is possible that systematic rotations could result in misorientations inside a feature that are larger than the defined tolerance allowable between any two neighboring voxels, as shown in Fig. 9.1. This effect could become important in a deformed structure, where the lack of an abrupt change in misorientation could allow many voxels to be assigned to the same grain. Application of a second misorientation tolerance, referring to the misorientation with the original seed voxel, will set a limit on accumulated misorientation.

9.3.3 Clean-Up Routines

There are inevitably data points that are assigned incorrect orientations during data collection, as well other microstructural features that currently cannot be identified by the EBSD software. Incorrect orientations are generally either a result of data points falling very near a grain boundary, where the generated pattern may be composed of information from both grains, or a layer of mechanical damage on the surface of the sample "blurring" the pattern. As a result, the EBSD pattern for the data point is not likely to match well with any orientation. Other features, such as precipitates or pores, may also return low quality values because these features return either no EBSD pattern or the crystal structures are not selected for use in pattern fitting.

Ideally data clean-up should be performed in 3D rather than 2D. Processing each section individually blinds the cleaning procedure from what is occurring directly above and below the section, which may change the interpretation of "problematic" data. The following subsections will discuss processing options used to clean and reassign portions of the EBSD data. Figure 9.2 shows some examples of reconstructed and processed data sets obtained from FIB-based sectioning and manual polishing.

9.3.3.1 Filtering of Low Quality Data

The segmentation procedure described previously may produce "grains" that are wholly comprised of low quality data. These "grains" are often small and may result from the presence of a pore, carbide, or local surface condition. Also, there may be voxels that are unassigned during the segmentation process, if the termination condition mentioned previously is used. These low quality or unassigned voxels should be examined a second time and potentially reassigned, but care must be taken to ensure that their addition does not significantly alter the grain morphologies of the good quality grains. There are multiple strategies that can be used in reassigning these points.

One option is to rerun the segmentation process for selected voxels with an increased misorientation tolerance (for example, from 5° to 10°). Often these low quality points correspond to data that are adjacent to a grain boundary. It is sometimes the case that these points produce overlapping EBSD patterns (from the two grains) and are indexed only slightly off their true orientation. However, the misorientation tolerance of 10° is still relatively small and not likely to cause points to be arbitrarily assigned to grains.

A second option is to determine the number of contiguous low quality points. This number is important in providing some understanding of the cause of the poor quality data. For example, if there is a grouping of low confidence points located together, then it is likely that these voxels correspond to a microstructural feature that could not be indexed successfully (i.e., a void or