Green Metallography

Introduction

EDAX has been preparing samples for Electron Backscatter Diffraction (EBSD) analysis for over 20 years. It is often surprising to visitors to our lab, that we have been able to handle such a wide variety of samples without a fume hood. Conventional wisdom suggests that neither having a fume hood nor having a permit for hazardous materials would severely hamper our ability to prepare samples for EBSD; however, we successfully prepare samples from which we obtain excellent EBSD results nearly every day. The only thing we consistently use which could be classified as a chemical is colloidal silica. In our experience, we have found easily 90% of the samples we receive can be prepared with fairly standard mechanical polishing, albeit using a vibratory polisher for the final polishing step. The remaining 10% can almost always be done using a non-chemical approach such as ion milling (e.g. zirconium and magnesium alloys).

We are not implying that good results cannot be obtained with electropolishing or chemical etching, we just emphasize that chemical preparation is not essential. A good example is tantalum. Tantalum is commonly prepared using chemical polishing and is more difficult to prepare using mechanical polishing. However, excellent results can still be obtained using mechanical polishing. We have found this to be true on most metals, alloys, ceramics, and minerals.

Non-mechanical methods like etchants often degrade Orientation Imaging Microscopy (OIM™) results. Etchants can lead to trenches along grain boundaries. These lead to 'wide' boundaries in the OIM™ maps. Other etchants may attack grains at different rates depending on orientation or deformation state. This preferential etching leads to topography and shadowing due to the high tilt required for EBSD, which will be detrimental to the OIM™ results. The same problems arising from topography are often observed in etched multiphase materials, where the etchants attack the different phases at different rates.

OIM™ as a Metallographic Tool

It is easy to get lost in the complexity of the crystallographic and texture data that OIM™ is capable of measuring. Luckily, the operator does not need to be expert in these fields to perform EBSD analysis. Anyone who is familiar with a Scanning Electron Microscope (SEM) can learn the additional procedures needed to operate an OIM™ system. Most analysts will already be familiar with crystallographic orientation as a contrast mechanism, for example polarized light optical microscopy or backscattered electron imaging in the SEM. EBSD measures and quantifies this orientation and the measured data can be used to perform most types of traditional metallographic analyses.

Is that a Grain Boundary?

The boundary between two grains of different lattice orientation is termed a grain boundary. The ability of OIM™ to quantitatively measure lattice orientations makes it the ideal instrument for the statistical characterization of grain boundaries in polycrystalline materials. EBSD provides a quantitative measure of the misorientation across a grain boundary, which enables a materials scientist to classify boundaries as low-angle grain boundaries (or subgrain boundaries), special boundaries of specific types (like twin boundaries), or random high-angle boundaries. This eliminates the need for sophisticated image analysis procedures to identify grain boundaries (Figure 1). Generally, the more complex the procedure the more sensitive it is to the control parameters, for example several parameters may be adjusted in processing procedure, such as the erosion or the skeleton operations often used to delineate grain boundaries (Figure 2).

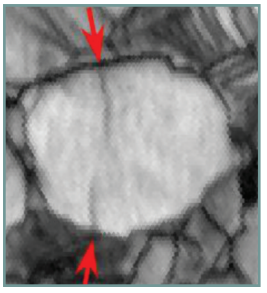

Figure 1. Uncertain grain boundary in α/β titanium.

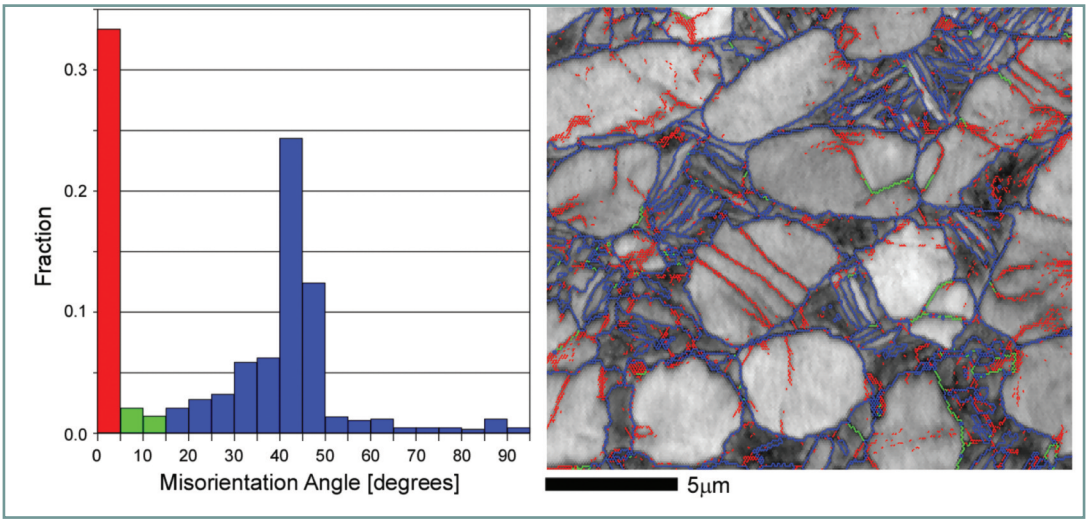

Figure 2. Grain boundary distribution in a α/β titanium sample and boundaries color coded by misorientation angle overlaid on a gray scale intensity map based on EBSD image quality (IQ). Lighter areas correlate to points with high quality diffraction patterns, whereas darker areas correspond to more diffuse patterns.

Thus, collecting high quality EBSD data may be more complex than collecting an optical or SEM image. Analyzing the EBSD data is fairly straightforward because the data produced by an OIM™ system are real quantitative values.

The ability of OIM™ to use quantified misorientation to identify boundaries compared to simple changes in intensity in optical microscopy, makes it particularly well suited to the study of highly deformed materials, where grains become obscured in the microstructure due to their convoluted shapes.

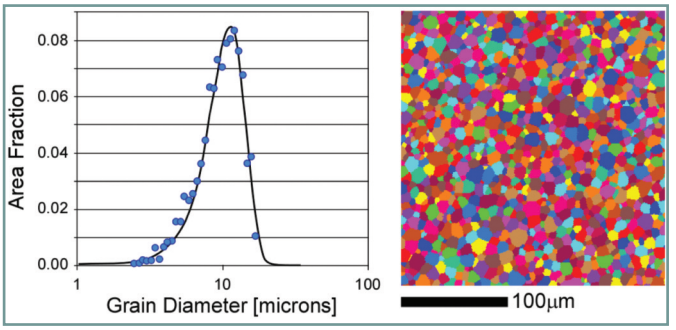

Figure 3. Grain size distribution in a yttria stabilized zirconia sample. The maps use random colors to denote individual grains.

Grain Size

The ability of OIM™ to delineate the grain boundaries in polycrystalline materials makes it capable of accurate measurements of grain size (Figure 3). Of course, the same sampling procedures as in any other grain size measurement technique should be followed to ensure statistical reliability.

Grain Shape

The ability of OIM™ to accurately measure grain boundaries makes it well suited for the analyses of grain shape in polycrystals (Figure 4).

Particle Distributions

OIM™ can be used to characterize the distribution of particles within a polycrystalline matrix. Any preference for particles to be located at grain boundaries or within the grain interiors can be studied. In addition to the size and location of the particles, their shape and orientation can be characterized as well. If the particles are less than 20 nm in size, it may not be possible to obtain indexable EBSD patterns. However, this does not preclude the ability of OIM™ to study the distribution of these particles, as they can be isolated from the surrounding indexed material.

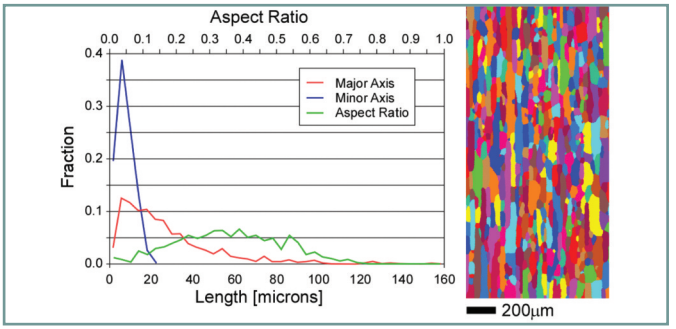

Figure 4. Distribution of grain shape parameters in an aluminum alloy. The map uses random colors to denote individual grains.

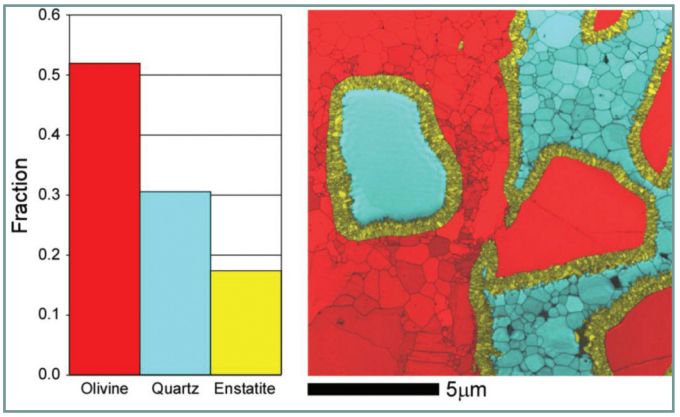

Figure 5. Phase distribution in a mineral sample. The map is shaded with colors for each phase overlaid on an intensity map based on IQ.

Multiphase Materials

EBSD can differentiate materials with dissimilar crystallographic structure, which makes it well suited to the study of multiphase materials. For example, OIM™ can easily distinguish between austenite, which has a face-centered cubic (FCC) structure, and ferrite, which has a body-centered cubic (BCC) structure. In other cases, the crystal structures may be very similar but the phases may have different chemical compositions. For instance, copper and nickel are both FCC materials and are difficult to differentiate with EBSD alone. Analysis with Energy Dispersive Spectroscopy (EDS) can help by determining the chemical composition. As both EBSD and EDS can be used simultaneously in the SEM, such materials using both techniques may be successfully analyzed (Figure 5). Coupling these two techniques together allows phases similar in crystallography or similar in chemical composition to be positively identified in a specimen, so that area fractions and size and cluster distributions can be obtained.

Crack and Void Analysis

Cracks and voids show up in OIM™ scans as scan points for which no patterns were obtained. It is thus possible to identify whether the cracks follow grain boundaries or are transgranular.

Material State

Plastic deformation manifests itself in two ways in OIM™ data. (1) The EBSD patterns are degraded, such that deformed areas appear dark in image quality maps. (2) The dislocations accompanying plastic deformation produce small rotations within the deformed materials. These local variations in orientation appear as subtle color variations in the orientation maps. Thus, image quality and local misorientation can be used to differentiate recrystallized regions from deformed regions in deformed samples. In addition, the magnitude of local misorientation is related to the amount of deformation so that the amount of plastic strain in the material can also be characterized to a certain extent (Figure 6).

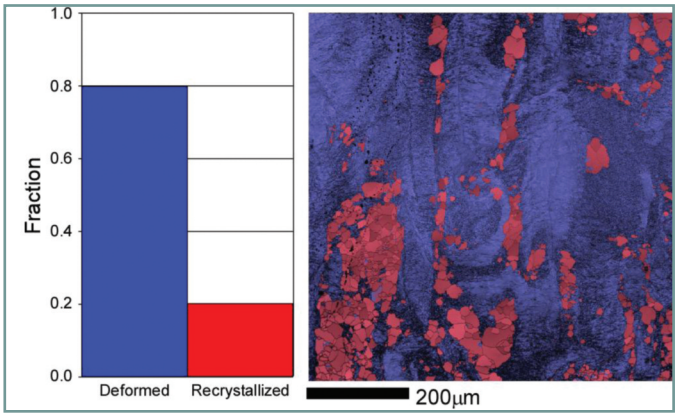

Figure 6. Distribution of deformed and recrystallized material in a low carbon steel sample. The map is shaded according to a threshold value of the local average misorientation, blue shaded areas are less than 1° and areas shaded red are greater than 1°. The colors are overlaid on an intensity map based on IQ.

Scale

Since EBSD is performed in the SEM, it is capable of making measurements at the micron scale. In some materials, measurements can be made at resolutions of tens of nanometers. By combining beam and stage movements together it is practical to scan areas as large as 6 cm (in the non-tilted direction) by 3 cm depending on the SEM geometry.

Orientation Data

The major strength of OIM™ when compared to traditional texture characterization techniques is that the source point of each individual crystal orientation measurement is known instead of being recorded as an average signal. This allows the analyst to correlate basic metallographic measures, such as grain size or recrystallization fraction with the orientation aspects of microstructure, such as crystallographic texture or grain boundary misorientation distributions. For example, Figure 7 shows the texture (preferred orientation) for the deformed material and recrystallized material for the sample shown in Figure 6.

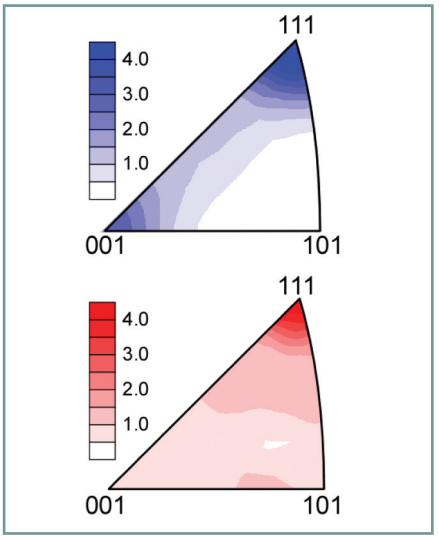

Figure 7. Texture plots (inverse pole figures) for the deformed (top) and recrystallized (bottom) material shown in Figure 6.

Advantages of Non-Chemical Approaches to Sample Preparation

Ease of Use

Developing the right etchant for a new alloy or other material can be quite difficult. Even for known materials, the quality of an etchant depends on factors like temperature, agitation and age of the constituent chemicals. Thus, it can sometimes be difficult to get reproducible results. The optimal etchant not only varies from material to material but also depends on what the metallographer wants to observe. For example, one etchant may be used to characterize the phase distribution in a multiphase material, another may be required to etch the grain boundaries so that the grain size distribution can be characterized; and yet another etchant may be used to characterize the grain size distribution in a second phase. Another factor in etchant selection could be the state of the material, i.e. deformed vs. recrystallized. Whether the characterization is to be performed in an optical microscope or in a SEM can also affect the choice of etchant. A glance into classic metallography texts [5-7] shows how complex and difficult etchant selection can be.

Using electropolishing adds further complexity. In addition to the factors listed for etching, electropolishing requires the current and voltage to be determined as well. These parameters can depend on the size of the area to be polished.

Achieving a uniform polish over the entire area can also be difficult, although modern electropolishing equipment has made this easier to accomplish.

Some multiphase materials can be difficult to polish mechanically, especially when one phase is considerably harder than another. In this case, the specimen preparation requires some experimentation with different polishing media and cloths. Like developing a new etchant, this can be expensive and time consuming. However, one big advantage in this trial and error process is that failed approaches can easily be disposed of. Working closely with your sample prep equipment supplier can often help overcome these difficulties more quickly.

Cost

Before finding the right combination on a new material, a metallographer may try several different etches or electropolishing solutions. It can be quite costly to purchase all of the required chemicals to perform the experiments. In addition, these solutions are generally toxic and disposing of the unused solution can be expensive. Maintaining a safe operating environment for a chemistry lab can also be very pricey and requires frequent auditing of equipment, supplies and procedures. This can greatly add to the cost of running a sample preparation lab.

Safety

The chemicals required to compose different etching and electropolishing solutions are generally hazardous in terms of toxicity and flammability. Thus, protective equipment must be installed and maintained and strict procedural precautions must be put in place to work safely.

Environment

Most metallographic preparation chemicals are hazardous. After use, the waste products must be disposed of properly in order to protect the environment. This adds more cost and potential liability to maintaining a sample preparation facility.

- OIM™ maps can actually be used as input into digital metallographic analysis systems.
- As OIM™ can characterize the full misorientation at boundaries it is also useful in the study of twins, both recrystallization twins and deformation twins.
- OIM™ enables grain size to be correlated with crystallographic orientation. Such information gives insight into why some grains grow at the expense of others.
- EBSD enables correlations between grain shape and crystallography to be identified. For example, the long axes of elongated grains in cast materials are often linked to specific crystallographic directions.
- The ability of OIM™ to measure grain boundary misorientation makes it possible to determine whether the particles are preferentially located at boundaries of a particular type (i.e. are they more prevalent at boundaries of high misorientation or at those with low misorientation?).
- Not only can the coupled technique be used to perform a discrete yes/no type analysis for phase differentiation, more subtle microstructural features can be analyzed as well. Combined EDS/EBSD can determine at what point subtle variations in chemistry lead to changes in crystallographic structure.
- EBSD provides the further ability to determine whether crack propagation or void formation is influenced by local crystallography.
- The ability of OIM™ to measure orientations rapidly enough for statistical analysis allows the researcher to identify which grain orientations are most susceptible to yielding. This is important information for the development of accurate polycrystal plasticity models.

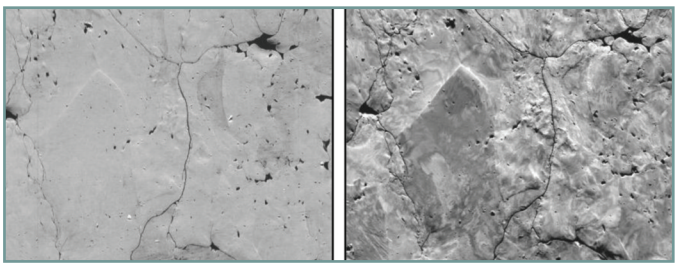

Figure 8. Secondary electron image (left) and FSD image (right) from a sintered steel sample.

Conclusions

EBSD is not just a microtexture tool. It is an excellent tool for full metallographic characterization and holds specific advantages over traditional characterization techniques. Development work is ongoing at EDAX to further automate these capabilities. Furthermore, mechanical polishing has been found to be more effective than chemical polishing techniques for EBSD sample preparation. This makes EBSD an attractive option for metallographic characterization in environments where the use of chemicals is discouraged due to cost, safety or environmental issues.

Modern OIM™ systems are fast. Very good scans for metallographic analysis can be obtained in five minutes. While this may not be quite as fast as obtaining an optical or even a slow scan SEM image, the additional information obtained makes it an attractive option to the more traditional techniques. In addition, modern OIM™ systems can be equipped with a forward scatter detector (FSD) providing outstanding image contrast relative to standard SEM contrast as shown in Figure 8.

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