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EBSD Sample Preparation: Techniques, Tips, and Tricks

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Automated analysis of Electron Backscatter Diffraction (EBSD) patterns for orientation imaging and phase identification in materials and earth sciences has become a widely accepted microstructural analysis tool. To briefly review, EBSD is a scanning electron microscope (SEM) based technique where the sample is tilted approximately 70 degrees and the electron beam is positioned in an analytical spot-mode within a selected grain. An EBSD pattern is formed due to the diffraction of the electron beam by select crystallographic planes within the material. The EBSD pattern is representative of both the phase and crystallographic orientation of the selected area. The pattern is imaged by a phosphor screen and recorded with a digital CCD camera and then analyzed. For orientation imaging, EBSD patterns are systematically collected and analyzed from a specified array of measurement points. The microstructure can then be visualized by coloring points on the array according to information derived from the EBSD pattern. For example, orientation imaging maps from a duplex stainless steel sample are shown in Figure 1. In Figure 1a, the colors represent the crystallographic directions aligned with the sample normal direction. The shaded stereographic triangle provides the color key. In Figure 1b, the colors represent the phases within the material; red is BCC ferrite while blue is FCC austenite. In Figure 1c, measurement points of similar crystallographic orientation are grouped together to define the grains within the material. The measured grains are then randomly colored to illustrate the grain size and morphology. Each of these maps is derived from the same acquired data, and many other analytical possibilities exist. In addition, quantitative microstructural and orientation measurements can be made. For example, in this duplex stainless steel the average grain size has a diameter of 32 microns assuming a circular grain shape. However, analysis of the grain shape shows this is not the case. By fitting an ellipse to the points defining a grain, the aspect ratio of the minor to major axes can be calculated. In this example the average aspect ratio is 0.34. The average grain size can then be more accurately defined as an average grain area of 828 square microns.

EBSD patterns are generated within a small interaction volume located at the surface of a sample with a penetration depth typically less than 50-100 nm. Because of this, EBSD pattern quality is extremely sensitive to the integrity of the crystallographic lattice order at the surface of the sample. While some samples, such as ECD deposited metal films, require no preparation prior to analysis, often samples must be sectioned and prepared to obtain useable EBSD patterns [1-2]. When considering how to prepare a sample for EBSD analysis, it is important to recognize that in order to obtain high-quality patterns the surface deformation that is typically introduced during standard metallographic preparation should be minimized. This deformation will disturb the crystallographic lattice and will result in more diffuse diffraction bands and a loss of intensity and contrast within the pattern. While EBSD patterns can be obtained from rough surfaces, the topography of the surface will often block the diffraction signal from reaching an EBSD detector, and will in turn reduce the yield of usable EBSD patterns obtained across such a surface. For orientation imaging, a flat surface is therefore desirable. Proper sample preparation will result in optimized pattern quality and subsequent high-confidence orientation imaging data. In this work, the sample preparation procedure developed to obtain high-quality EBSD patterns from a nickel-based superalloy will be presented.

A rod (6.35mm diameter) of INCONEL 600 (Ni72/Cr16/Fe8) from Goodfellow was selected as a standard sample material. The relatively high effective atomic weight of this alloy provides a strong backscatter signal and pattern intensity. The rod was sectioned with a low-speed diamond saw (Buehler Isomet) at approximately 200 RPMs. This sectioning method was selected as to minimize the damage introduced during cutting. A cutting lubricant (Isocut Fluid) was used to improve cutting rates and minimize frictional heat generated.

After sectioning, the samples were then mounted in a copper-based conductive mounting powder using a compressive mounting press (TechPress2 – Allied High Tech Products). This conductive mounting material is preferred for EBSD analysis as it helps minimize electron beam charging effects which can cause intensity blooming in the EBSD patterns and image drift and distortion in orientation maps. Often, even non-conductive samples can be mounted in this material and observed with EBSD without requiring a conductive coating. However, the samples are subjected to both pressure (3800 PSI) and temperature (175°C) during the mounting process. If the samples cannot withstand these influences then

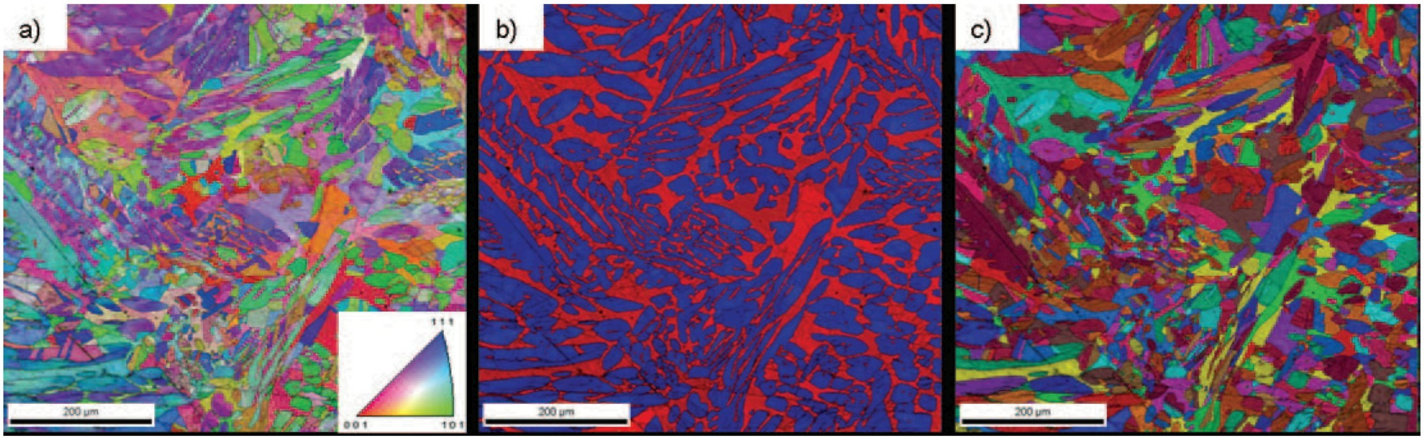

Figure 1: Orientation imaging maps from duplex stainless steel showing (a) orientation-colors indicate which crystal directions are aligned with the sample normal direction, (b) phase-red is BCC ferrite and blue is FCC austenite, (c) grains-randomly colored to show size and morphology.

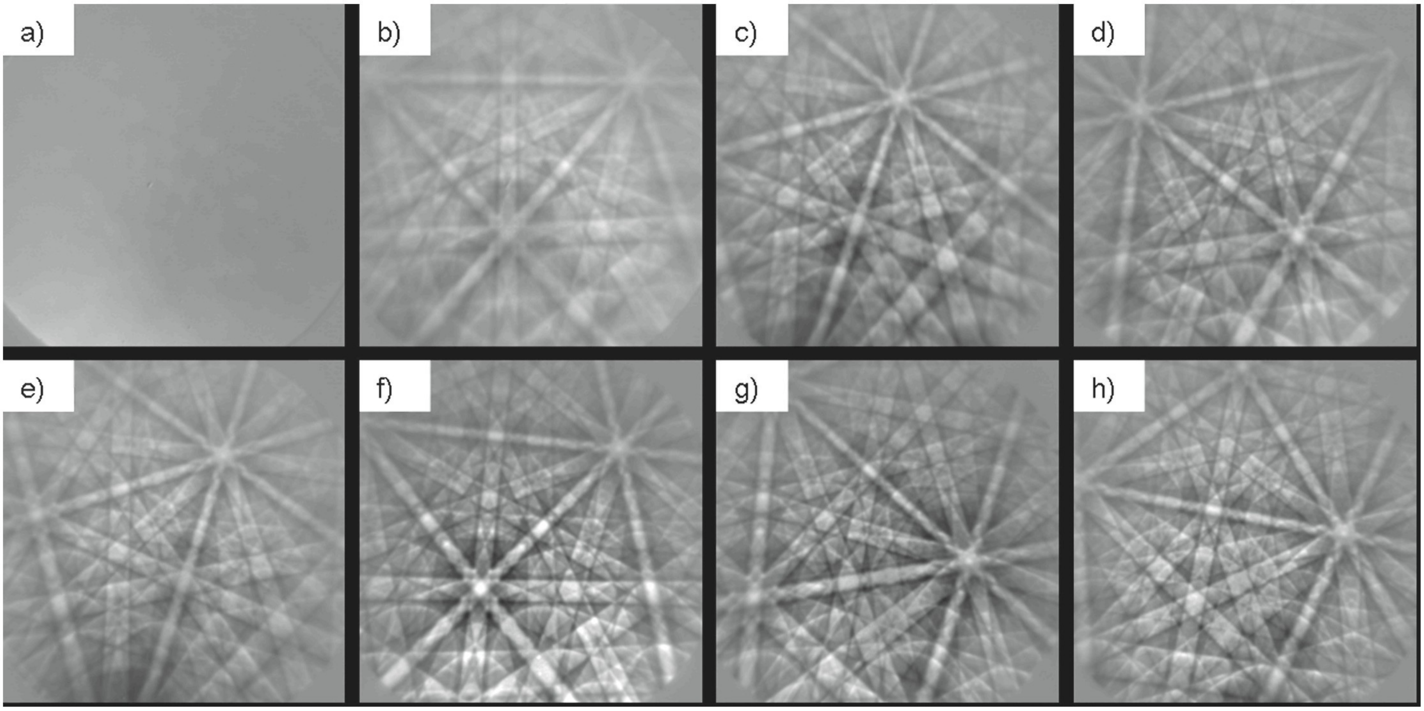

Figure 2: EBSD patterns after polishing with (a) 1200-grit SiC, (b) 1 micron alumina, (c) 0.3 micron alumina, (d) 15 minutes 0.05 micron colloidal silica (CS), (e) 30 minutes CD, (f) 60 minutes CS, (g) 120 minutes CS, (h) 240 minutes CS.

other mounting materials may be necessary. For example, ceramics are often very brittle and cannot be subjected to this pressure without fracturing. In this case, a cold-mounting epoxy may be more appropriate. During analysis, the non-conductive surface of the epoxy may be painted with conductive silver or carbon paint to minimize charging effects.

The next steps in the preparation procedure are grinding and polishing, although it can sometimes be difficult to define when the transition from grinding to polishing occurs. Conceptually they are similar, as an abrasive is initially used to remove damage introduced during sectioning and to produce a planar surface. Then subsequently smaller abrasives are used to remove the damaged introduced by the larger abrasives. In this work, grinding is defined as using 240-, 320-, 400-, 600-, 800-, and 1200-grit SiC abrasive papers in conjunction with a water lubricant. Although other abrasives are available, SiC is commonly used due to its high hardness. A force of

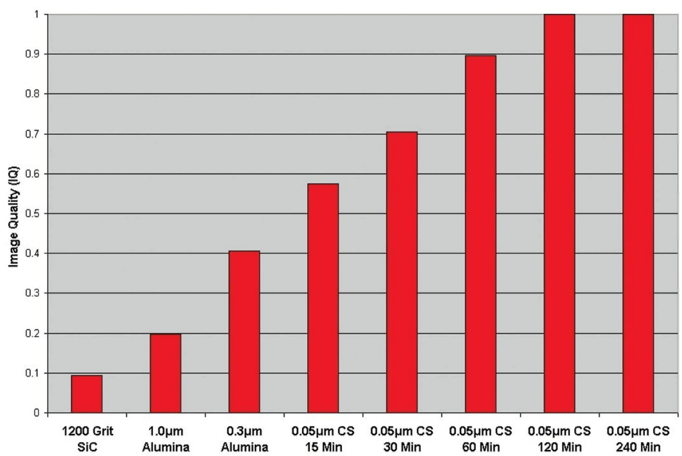

Figure 3: Normalized image quality values after polishing with (a) 1200-grit SiC, (b) 1 micron alumina, (c) 0.3 micron alumina, (d) 15 minutes 0.05 micron colloidal silica (CS), (e) 30 minutes CD, (f) 60 minutes CS, (g) 120 minutes CS, (h) 240 minutes CS.

10lbs was used with a wheel rotation of 150 RPMs. The wheel rotated clockwise, while the sample mount rotated counterclockwise. Each abrasive size was used for 30 seconds. It is recommended that a new abrasive paper be used as the effectiveness of the abrasive decreases with use. Although this may result in an increase in the cost of polishing consumables, this will also increase the quality of the resulting polishing and subsequent EBSD patterns.

For polishing, both the abrasive material and cloth must be selected. In this work, an all-purpose, low-napped, synthetic rayon cloth (Imperial Cloth – Allied High Tech Products) was selected. While there is a wide variety of polishing cloths available, an all purpose cloth that works with a range of abrasive materials and sizes is desirable, and has proved to be effective. Alumina suspensions of 1.0 micron and 0.3 micron sizes were used. Diamond suspensions are also recommended. Each abrasive size was used for 10 minutes at 9 lbs force and a wheel rotation of 130 RPMs. Water was again used as a lubricant.

For the final polishing step, samples were placed on a vibratory polisher where the samples are subjected to low force (the weight of the sample and holder is less than 1 lb.) and moved continually over the polishing cloth by the vibratory movement. The all-purpose cloth was again used, but the final polishing medium was 0.05 micron colloidal silica (CS). This suspension has a pH of 9.8 and the chemical-mechanical polishing that results provides an excellent surface finish for EBSD analysis. Samples were polished for 15, 30, 60, 120, and 240 minutes.

To evaluate the effects of the different polishing steps on EBSD pattern quality, patterns and orientation imaging datasets were acquired after each step. For pattern comparison, patterns were collected at 1024x1024 pixel resolution. For orientation imaging, patterns were collected at 128x128 pixel resolution. Orientation images were collected from a 1.2 mm x 1.2 mm area with a 5.0 micron spacing between measurements. A hexagonal measurement grid was used, which resulted in approximately 68,000 measurements

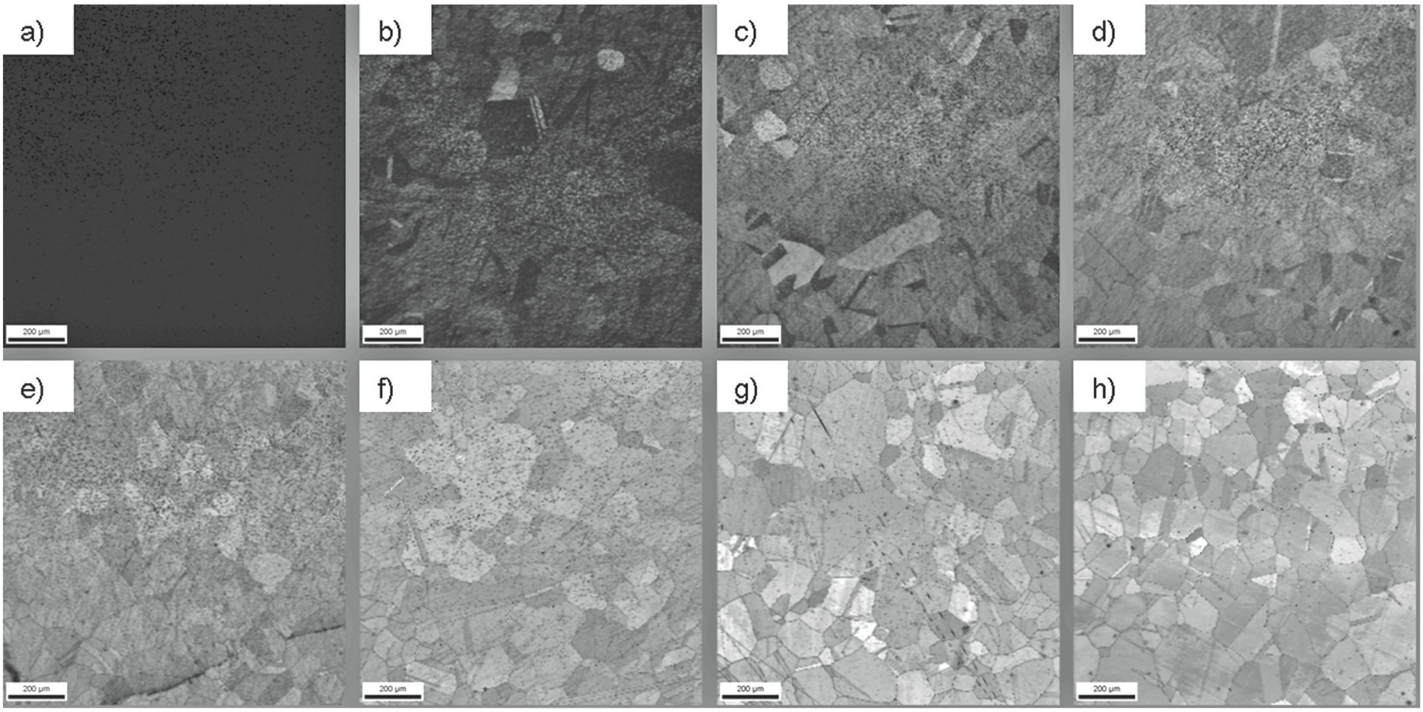

Figure 4: Normalized image quality maps after polishing with (a) 1200-grit SiC, (b) 1 micron alumina, (c) 0.3 micron alumina, (d) 15 minutes 0.05 micron colloidal silica (CS), (e) 30 minutes CD, (f) 60 minutes CS, (g) 120 minutes CS, (h) 240 minutes CS.

collected and processed at a rate of 80 patterns per second for a total acquisition time of 14.2 minutes.

The results of the polishing steps on EBSD pattern quality are shown in Figure 2. Polishing to 1200-grit SiC produces only weak patterns, while polishing to 1.0 micron alumina or beyond produced high-quality patterns, at least by visual inspection. The automated analysis of the patterns using image processing routines can provide another metric of quality termed image quality (IQ) [3]. The normalized IQ values as a function of polishing step are shown in Figure 3 as calculated from the acquired orientation imaging data. Orientation imaging data is used to sample a large number of grains ($\approx$ 500 grains) to average out the orientation effects on pattern quality. This data shows a significant increase in image quality between steps through a polishing time of 120 minutes with colloidal silica. The 240 minute polish did not result in an increase in image quality, which suggests that the 0.05 micron colloidal silica reaches the limit of effectiveness somewhere between 60 to 120 minutes polishing on this material. It is interesting to note that although it is difficult to visually observe the quality difference between the polishing steps, the measured image quality more than doubles from 15 minutes to 120 minute of polishing with colloidal silica. The normalized image quality maps derived from the orientation imaging data after each polishing step are shown in Figure 3.

Table 1: Percentage of correctly indexed points after each polishing step.

Polishing Step	Points Correctly Indexed (%)
1200-Grit SiC	4.5
1.0 Micron Alumina	77.3
0.3 Micron Alumina	97.9
15 Minutes 0.05 Micron Colloidal Silica	99.6
30 Minutes 0.05 Micron Colloidal Silica	99.7
60 Minutes 0.05 Micron Colloidal Silica	99.9
120 Minutes 0.05 Micron Colloidal Silica	99.9
240 Minutes 0.05 Micron Colloidal Silica	99.9

Although image quality is one measure of pattern quality, another potentially more important metric is how well the automated pattern analysis routines function on patterns of differing image quality. In this work, the accuracy of the EBSD pattern indexing was quantified using the confidence index (CI) value [4]. The EBSD pattern was considered to be correctly indexed if it had a CI value greater than 0.3, which corresponds to 99% probability of accuracy. The percentage of correctly indexed points after each polishing step is shown in Table 1. After 15 minutes of colloidal silica polishing, 99.6% of the points are correctly indexed, while after 60 minutes this value increases to 99.9%. Clearly, although the image quality continues to rise after the 15 minutes, the patterns are of sufficient quality for accurate analysis at this point. This however may not be true for all materials. Generally EBSD pattern analysis routines function better with higher-quality input patterns.

The preparation routine described here has been used to successfully prepare a wide-range of materials for EBSD analysis. It should be noted however that exceptions do occur. Very hard materials would require substituting diamond for alumina to improve polishing performance while some multi-phase materials may require shorter final polishing times to minimize the effects of preferential polishing and etching rates. Conceptually, however the goal remains the same, to produce a flat, damage-free surface that is representative of the material of interest. ■

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