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Methods of Evaluating EBSD Sample Preparation

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Electron Backscatter Diffraction (EBSD) has become a well accepted microanalytical technique for investigating texture, grain boundary character, strain, and phase distributions in materials and earth sciences. EBSD patterns are formed within the first tens of nanometers of material at the surface of a sample. EBSD pattern formation is dependent on the quality of the crystal lattice within this volume. Because of this, EBSD pattern quality is strongly dependent on the method and quality of the sample preparation techniques used.

Figure 1 shows an example of how sample preparation can influence the resulting EBSD measurements. Samples of Inconel 600 were polished with 240, 320, 400, 600, 800, and 1200 grit SiC abrasive papers [1]. A representative sample was taken after each polishing step, and cross-sectioned with a low-speed diamond saw. The cross-section was then polished for EBSD analysis down to 0.05µm colloidal silica. Figure 1 shows Local Orientation Spread maps from the 240 and 1200 grit polished cross-sections, with the SiC polished surfaces on the right. The deformation at the polished surface is clearly visible, and the depth of this region decreases with decreasing abrasive size.

These results show that the final abrasive size used influences the resulting EBSD patterns. Other factors such as polishing time, the size difference between abrasive used in adjacent preparation steps, and polishing consumables such as abrasives and cloths used can also play a role. With such a complex matrix of preparation possibilities it is advantageous to be able to evaluate and quantify the quality of sample preparation method.

One approach is to use EBSD image or pattern quality (IQ or PQI), which measures the contrast of the detected diffraction bands via the Hough Transform. This can be difficult however, as IQ is not only a function of lattice quality but also of orientation, crystal structure, camera and SEM acquisition settings, and band detection parameters. Therefore an absolute value representing surface quality only is not obtained.

A second approach, which is particularly effective for orientation mapping, is indexing rate, defined as:

$$Indexing\ Rate = \frac{Total\ \#\ Points - \#\ Points\ Incorrect - \#\ Points\ Unsolved}{Total\ \#\ Points}$$

Indexing rate indicates the percentage of measured points from which a correct orientation was calculated. Figure 2 shows normalized indexing rates and average image quality values as a function of sample preparation for Inconel 600. Note that at 15 minutes polishing with colloidal silica, the indexing rate is essentially 100% even though the IQ value is approximately half of the value obtained with further polishing.

References
[1] M.M. Nowell et al., *Microsc. Microanal.* 11 (Suppl. 2) (2005) 504.
[2] G.M. Lucas et al., *Microsc. Microanal.* 14 (Suppl. 2) (2008) 546.
[3] S.I. Wright and M.M. Nowell, *Microsc. Microanal*. 12 (2006) 72.

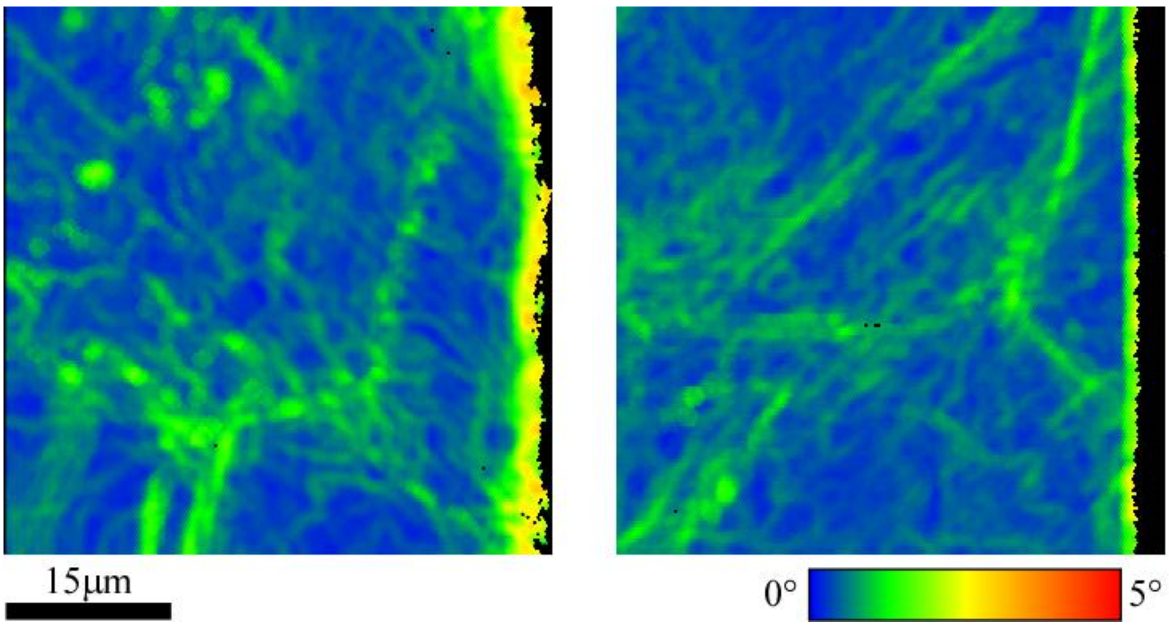

FIG. 1. Local Orientation Spread Maps from 240 grit (left) and 1200 grit (right) polished cross-sections, with polished surface on the right for each map.

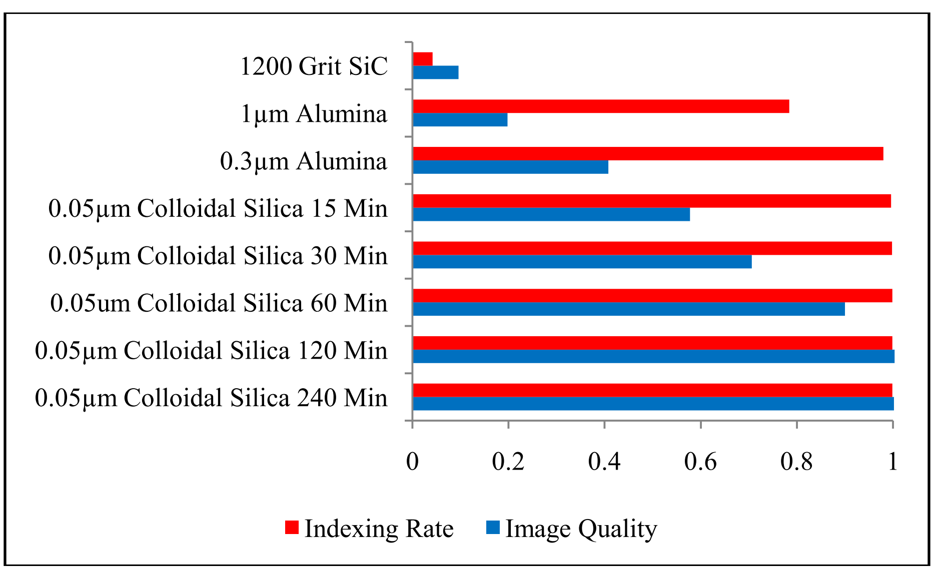

FIG. 2. Normalized Indexing Rate and Image Quality values from Inconel 600 samples polished to different final states.

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