

Towards A More Quantitative Measurement of the Deformation During Metallographic Specimen Preparation Using EBSD and FIB

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Surface deformation during metallographic preparation have been previously studied using light optical microscopy (LOM) and transmission electron microscopy (TEM) [1]. With its submicron resolution, electron backscattered diffraction (EBSD) can provide quantitative deformation analysis at a smaller length scale than LOM while provide higher statistics than TEM. This work aims to determine the level of deformation produced during different metallographic preparation steps of common materials. As a first iteration, the deformation profile induced by 80, 240 and 600 ANSI grit SiC papers on commercially pure iron (BCC), copper (FCC) and titanium (HCP) was measured.

The quality of the EBSD diffraction patterns is a function of many factors, of which an important one is the level of deformation [2]. For this study, nine quality indices were calculated from the analysis of the diffraction patterns or their Hough transform to evaluate the deformation depth. As an attempt to verify the EBSD results, cross-sections of the deformed samples were made using a FIB microscope.

To prevent prior or unwanted deformation to influence the measurements, special care was taken during the sample preparation. The samples were annealed and polished up to colloidal silica before being deformed by the grinding paper. Before the final polishing of the deformation profile face of the sample, the deformed surface was nickel plated to avoid any edge deformation effect. The EBSD acquisitions were performed on a Hitachi S-4700 cold-field emitter microscope equipped with an HKL Nordlys II camera. The image analysis software RML-Image [3] was used for the post-processing of the diffraction patterns. The FIB images were taken on a Hitachi NB-5000 microscope.

Deformation profiles were obtained by dividing the EBSD maps into sections of equal width starting from the deformed surface. Four of the nine quality indices gave satisfactory profiles. Using the 90% deformation point, the depth of deformation was evaluated. Figure 1 shows these measurements and those from the FIB images. Although the calculations for quality indices are different, the EBSD measured deformation depths for a given grinding paper are similar. However, the deformation depths obtained from the FIB are much smaller than those obtained by EBSD. A possible explanation is that the deformed area seen in the FIB images corresponds to the recrystallization zone and not the complete shear-band layer. However, the FIB images (Figure 2) clearly shows the heterogeneity of the deformed layer, implying that several measurements are required to properly assess the deformation depth.

Work is currently underway to improve the statistics of our results as well as continuing this evaluation on two metals with the same crystal structure but with different stacking fault energy, in occurrence copper (40 mJ m^{-2}) and aluminum (200 mJ m^{-2}).

[1] L.E. Samuels, *Metallographic Polishing*, 4th edition, ASM International, Material Park, 2003.
 [2] S.I. Wright and MM. Nowell, *Microscopy and Microanalysis* 12 (2006), 72-84.
 [3] M. Lagacé, RML-Image (www.rml-image.com)

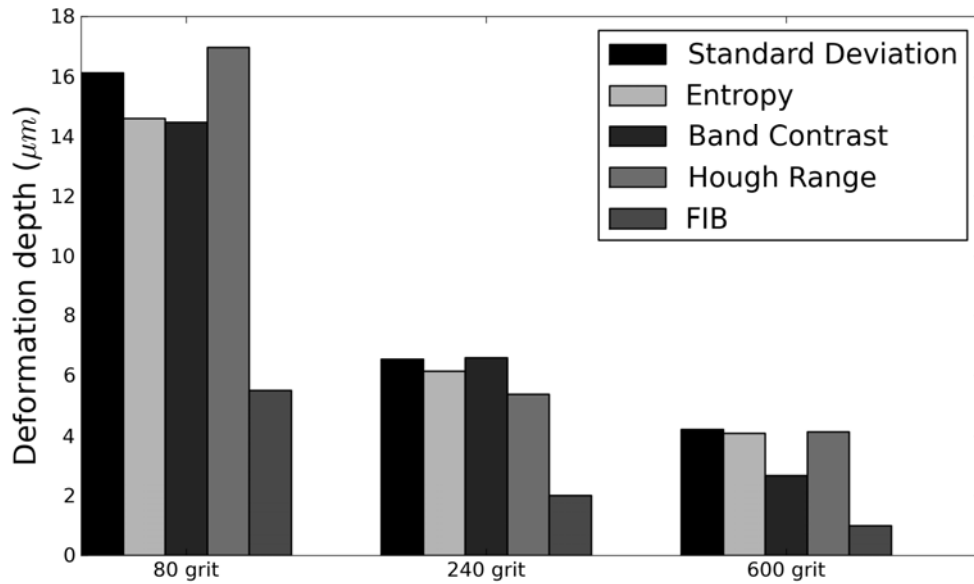


Figure 1: Deformation depth of three ANSI SiC grit papers measured using four EBSD’s quality indices and FIB measurements.

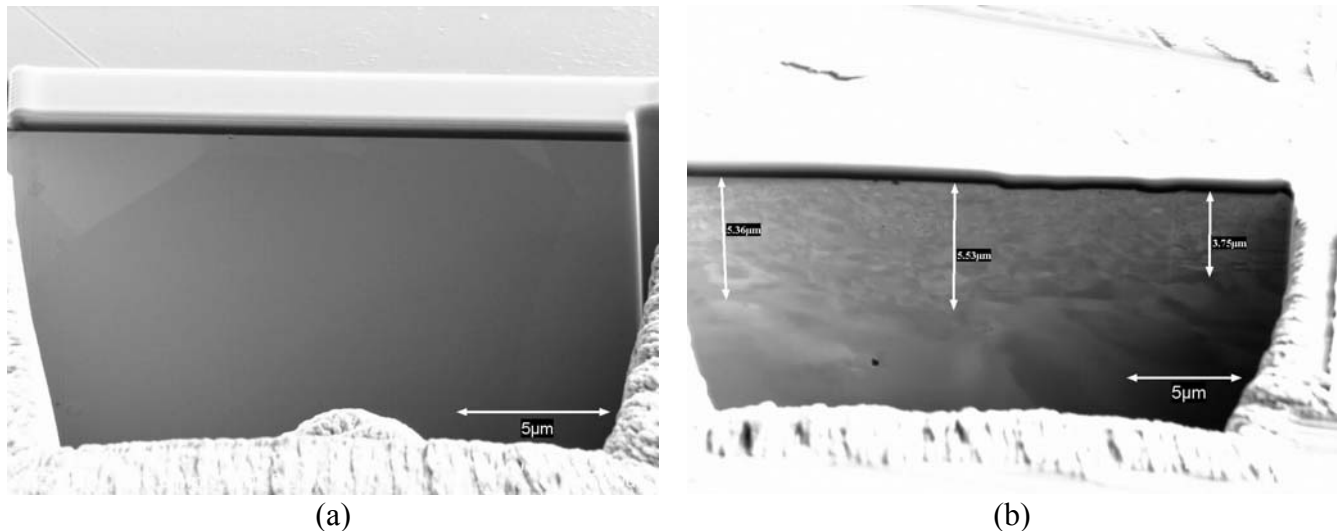


Figure 2: SEM images after FIB trench cutting on copper sample, (a) prior to deformation and (b) after 80 ANSI grit SiC paper.