Low angle polishing of large areas using plasma FIB-SEM to reduce sample surface damage when preparing samples for EBSD

Introduction

To understand how a material will behave under specific conditions requires a comprehensive analysis of its properties. In addition to the structural and compositional information provided by SEM and FIB-SEM imaging and analysis, Electron Backscatter Diffraction (EBSD) reveals crystallographic phases and their distribution, as well as providing information about grain orientation.

EBSD uses electron backscattered diffraction patterns to reveal a material's crystallography. When an electron from the SEM column interacts with the material's atoms, it inelastically scatters some of the electrons, which results in a divergent source of low-energy electrons near the sample surface. These electrons are then used to form EBSD patterns. Successful EBSD analysis, therefore, requires a deformation-free material surface to achieve the necessary yield of the electrons.

Sample preparation for EBSD analysis can be challenging, especially for materials with two or more phases of differing hardness or materials that are sensitive to the beam energy used for polishing. One option is to use mechanical polishing. While this technique is suitable for many materials, it is unable to achieve the required surface quality for multi-phase or sensitive materials because the softer phase is more easily removed, resulting in one phase being over-polished. Additionally, mechanical polishing is not suitable for materials that might recrystallize and transform to different phases, for example, duplex steel. Electrochemical polishing is another option, but it too has restrictions for some materials. Thus, it can be difficult to balance polishing efficiency for materials containing more phases and with different chemical compositions.

Ion polishing methods permit the use of different ion beam energies to reduce deformation on the sample's surface, removing the top surface to produce a pristine material surface for analysis. In this application note, we will present TESCAN's Low Angle Polishing technique for preparing multi-phase materials for EBSD. Combining the Low Angle Polishing technique with plasma FIB enables the processing of large areas, providing data from a larger area and improving the size of the statistical sampling for more accurate conclusions about a material.

Materials and methods

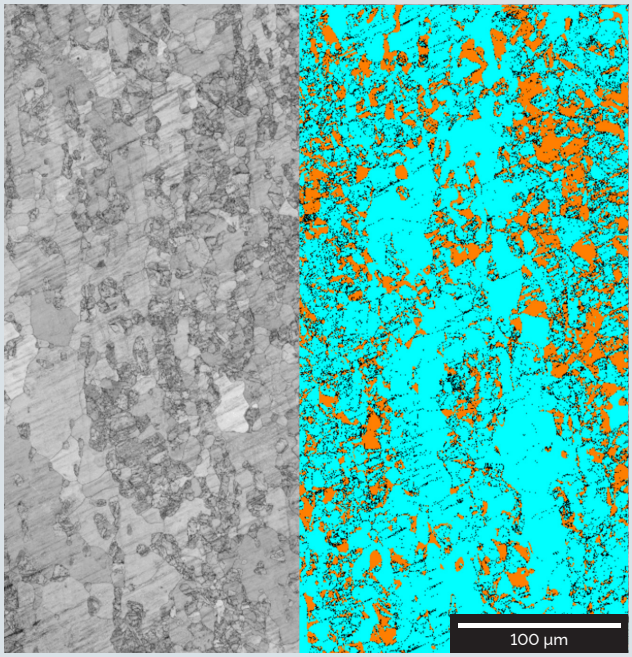
Three materials were prepared and characterized for this use case study. The first sample was duplex steel, and the other two samples were from the WC-Co materials group.

A TESCAN AMBER X plasma FIB-SEM was used for both the material polishing and the EBSD analyses. The instrument was configured with an EBSD camera from Oxford Instruments and TESCAN's patented Low Angle Polishing method was used to polish large areas of each sample. This method was developed by TESCAN primarily for applications in the semiconductor industry, however, it is also proven to produce high quality uniform surfaces on otherwise difficult to polish materials science samples. The Low Angle Polishing method uses low ion incident angles to minimize damage to the sample surface. The entire process is controlled from the Essence™ Low Angle Polishing software module, which automates the adjustment of the sample's surface position to mitigate the occurrence of polishing artifacts and supports effortless switching between beam energies during the polishing process, thereby allowing the use of the lower ion energies as needed.

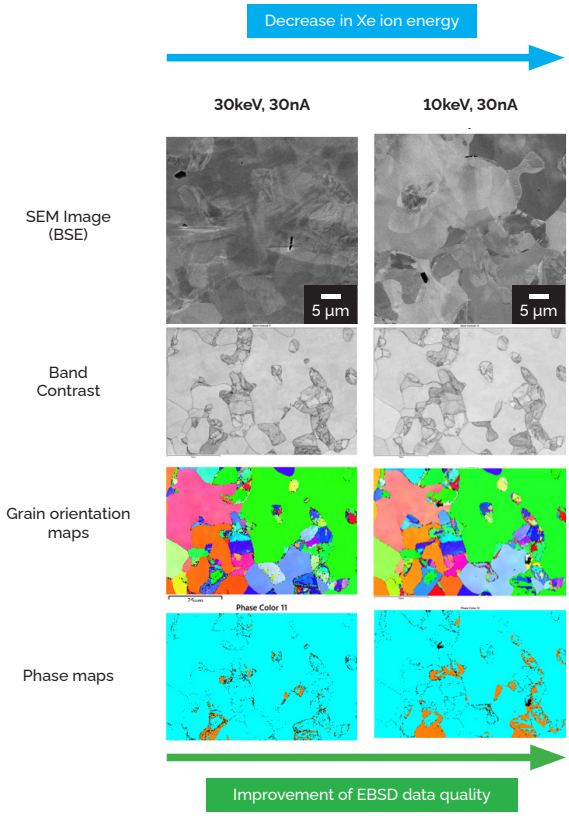
In the experiment, we performed FIB polishing using a FIB current of 30 nA. The ion beam energy was set to 30 keV, 15 keV, and 10 keV respectively. For the second experiment, the ion beam setting was 30 keV and 100 nA, and 15 keV and 75 nA were used as well to evaluate the dependency of EBSD pattern quality on ion beam current. We collected all EBSD datasets using a 20 keV and 10 nA electron beam. High-resolution mapping, even at high beam currents, was achieved through the automated beam profile adjustment, which is a standard feature of TESCAN microscopes.

Results and Discussion

The Low Angle Polishing method was very effective for producing high quality, uniform surfaces for EBSD characterization. Pairing the Low Angle Polishing method with plasma FIB-SEM extends the technique to preparing large area surfaces. Figure 1 shows the duplex steel sample with a polished area of 500 µm x 500 µm. The final polishing was performed with the Xe plasma FIB using 10 keV at 30 nA.



▲ **Figure 1:** The large area of 500 µm x 500 µm EBSD map from the surface polished by Low Angle Polishing method shows band contrast image (left) and phase distribution map (right).



▲ **Figure 2:** A comparison table showing the improved quality of EBSD data following the implementation of reduced ion beam energy during polishing.

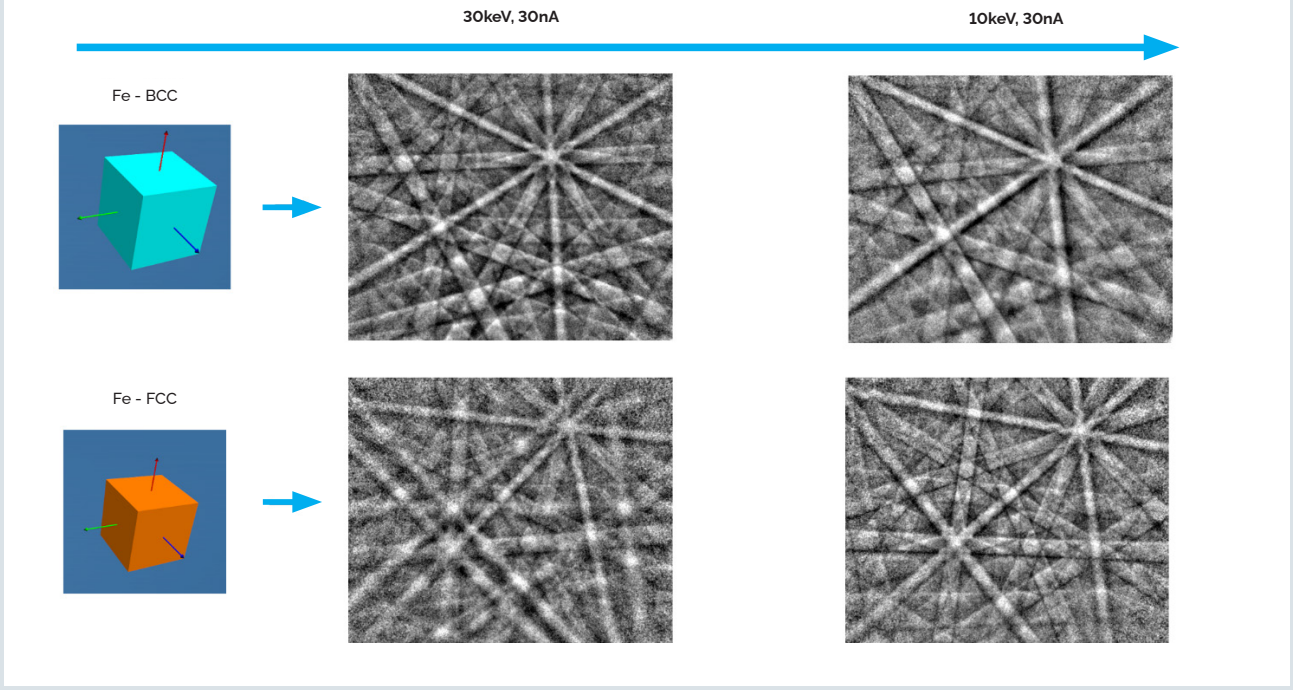
Reducing the ion beam energy resulted in better surface quality, as shown in Figure 2. The table compares the SEM images, band contrast, and Inverse pole figure (IPF) images showing the grain orientation map and phase distribution map.

The BSE image of the sample polished at 10 keV provides more channeling contrast for depicting the grain structure information. The EBSD results show a slight improvement in indexing from 2.5 zero solutions to 2.4 zero solutions.

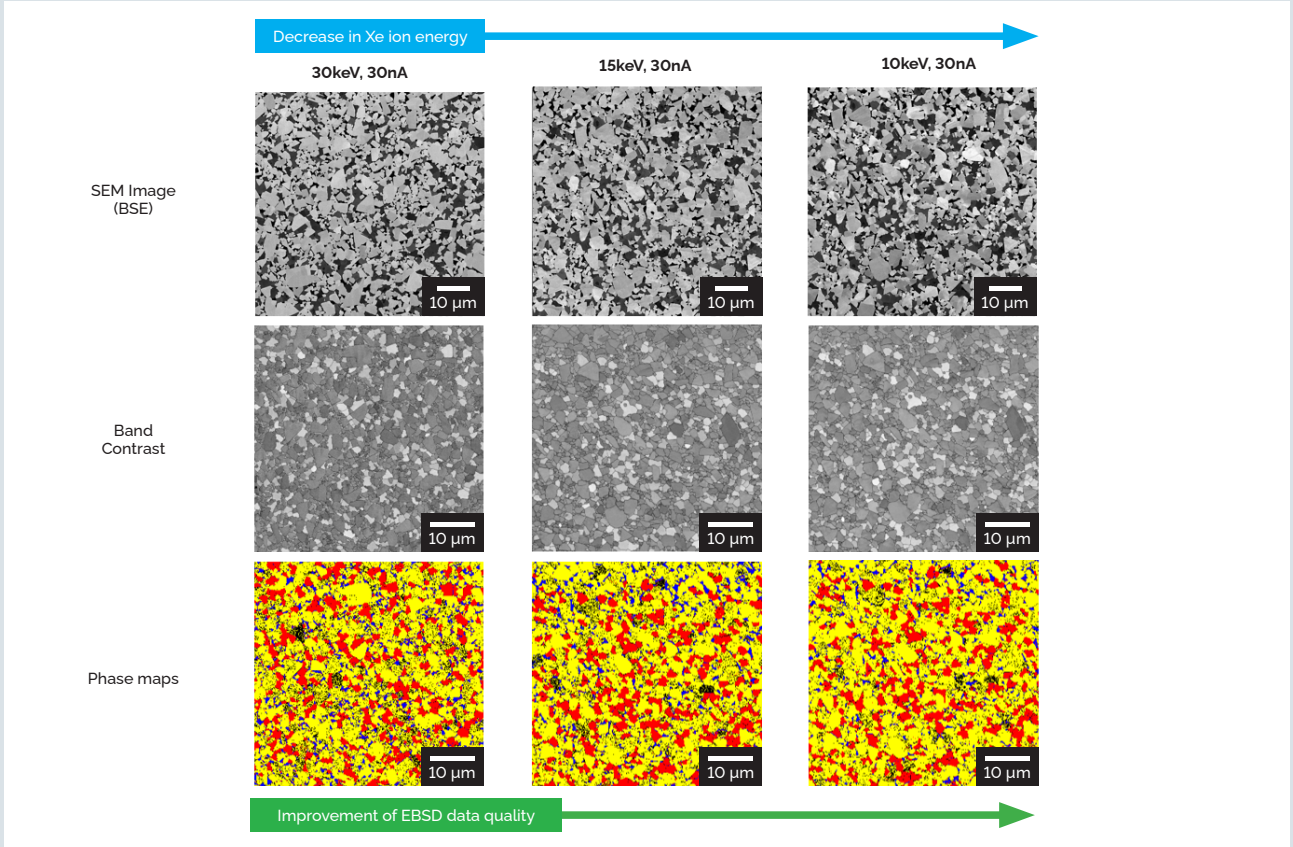
An important difference is observed when comparing the two-phase maps. Both maps were acquired from the same area on the sample. Due to the polishing, some of the materials were removed, thus the z position varies. As such, we cannot claim that the lower energy blocks the material recrystallization, but we can detect more FCC phase in a material after polishing at 10 keV. This effect can also be explained by better indexing that was caused by improved EBSD pattern quality which is shown in Figure 3. The amount of the FCC phase increased from 2.06 % when the sample was polished by 30 keV ion beam energy compared to 6.91% after being polished by 10 keV while keeping the zero solution fairly similar changing from 2.52 % to 2.35% while the ion energy decreases.

The same behavior was observed on the WC-Co material (Figure 4). Reduction of beam energy led to increased grain channeling contrast, improvement of band contrast, and simultaneous improvement in phase indexing. We can consider the total area of grain boundaries detected in a sample will be consistent from a statistical perspective. Therefore, we can assume that the improved indexing is caused due to the improved EBSD pattern which positively affects the indexing quality. The indexing improves by 10.1% zero solution for the surface polished at 30 keV, 9.5% zero solution for the 15 keV polishing step, and 8.2% zero solution for index quality from the surface polished by 10 keV beam energy.

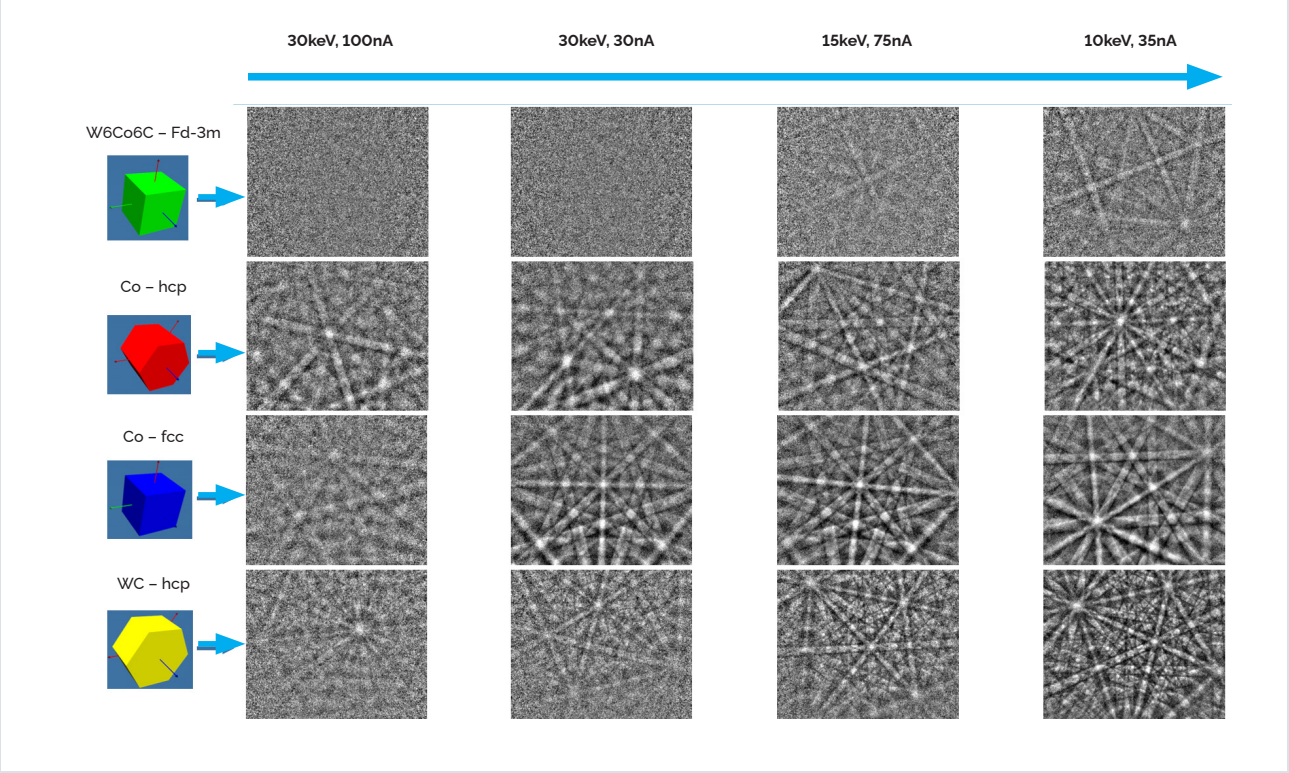
The influence of ion beam energy on the sample's surface quality was studied on a second similar sample from the WC-Co materials group. Figure 5 shows the pattern quality produced by different polishing conditions for each of the



▲ **Figure 3:** Corresponding EBSD patterns for the two phases of duplex steel polished at 30 keV and 10 keV with a beam current of 30 nA.



▲ **Figure 4:** A comparison table showing the difference in the quality of EBSD data after polishing at decreased Xe plasma FIB beam energies.



▲ **Figure 5:** EBSD pattern phases were detected in WC-Co cutting material polished at different ion beam conditions: 30 keV 100nA; 30 keV 30nA; 15 keV 75nA and 10 keV of 30 nA.

various phases found in the material. Polishing conditions vary from 30 keV at 100 nA, 30 keV at 30 nA, 15 keV at 75 nA, and 10 keV at 35 nA. The acquisition time of a single pattern was 5 ms for the surface processed at 30 keV at 100nA and 8 ms for the remainder of the milling conditions.

The data in Figure 5. clearly shows the pattern improvements related to the beam energy and to the ion beam current. The lower energies are beneficial for high-resolution EBSD mapping when the high-quality EBSD pattern is mandatory or to polish samples that consist of different phases or are sensitive to high energies.

Conclusion and outlook

Low Angle Polishing is a universal polishing method that can be used for EBSD sample characterization. The method is also suitable for materials that might be problematic to polish using conventional methods such as mechanical polishing because the technique minimizes sample surface damage and the creation of curtaining artifacts. A part of the process of polishing is automized in TESCAN Essence™ software. The Essence™ Low Angle Polishing software module enables working with different ion beam settings. The possibility to adjust ion beam settings for Low Angle Polishing plays a crucial role for samples that are difficult to polish because they contain multiple phases with differing hardness. The implementation of the Low Angle Polishing method on Plasma FIB-SEM enables large area polishing which allows researchers to characterize a more statistically relevant sized sample.

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