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APPLICATION NOTE

µPolisher System

A low-energy ion polishing solution for localized surface cleaning in SEMs and DualBeams.

The new Thermo Scientific µPolisher System boasts a great number of potential applications, including fine polishing of the sample surface in order to obtain electron backscatter diffraction (EBSD) patterns of the highest quality and removal of residual hydrocarbons from the sample surface prior to high-resolution SEM imaging.

Principle of work

The cleaning mechanism involves the ionization of the primary atomic or molecular gas flow by the electron beam of the SEM. Argon gas is delivered through a small nozzle, where an electron beam of low energy and high current is scanned through a slit in the nozzle, generating ions by direct ionization and from beam interactions with the tungsten wall of the nozzle. The ions are then accelerated towards a biased sample located a short distance from the nozzle, typically about 100 µm. The energy of the ions can be varied in the range of 20 to 500 V. The width and direction of the ion beam depends only on geometry, not on the charge and mass of the ion.

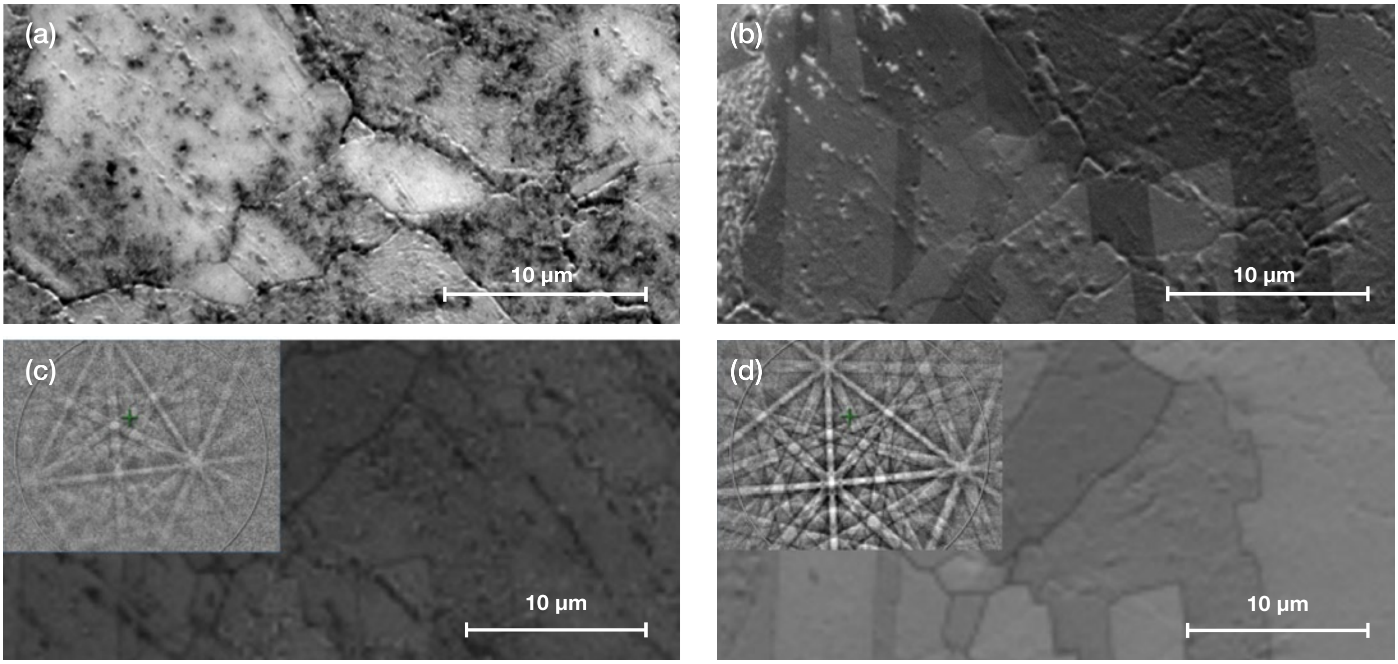

Figure 1. SEM images of a copper surface before (a) and after (b) polishing by argon ions using the µPolisher System. Band contrast images and Kikuchi patterns (inset), acquired at 20 kV, 2.8 nA before (c) and after (d) polishing, show the improvement of pattern quality.

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SEM images (Figure 1) comparing the same region of interest on the surface of copper before (a) and after (b) polishing show improvement in surface quality due to the removal of hydrocarbon contamination and oxide layers, thus revealing the native surface. The corresponding band contrast images (Figure 1c and 1d respectively) along with Kikuchi patterns (inset), show the enhanced pattern quality after the surface has been polished. These EBSPs, acquired in spot mode from a Fast CCD camera before and after polishing, demonstrate the improvement in pattern quality and signal to noise ratio after polishing.

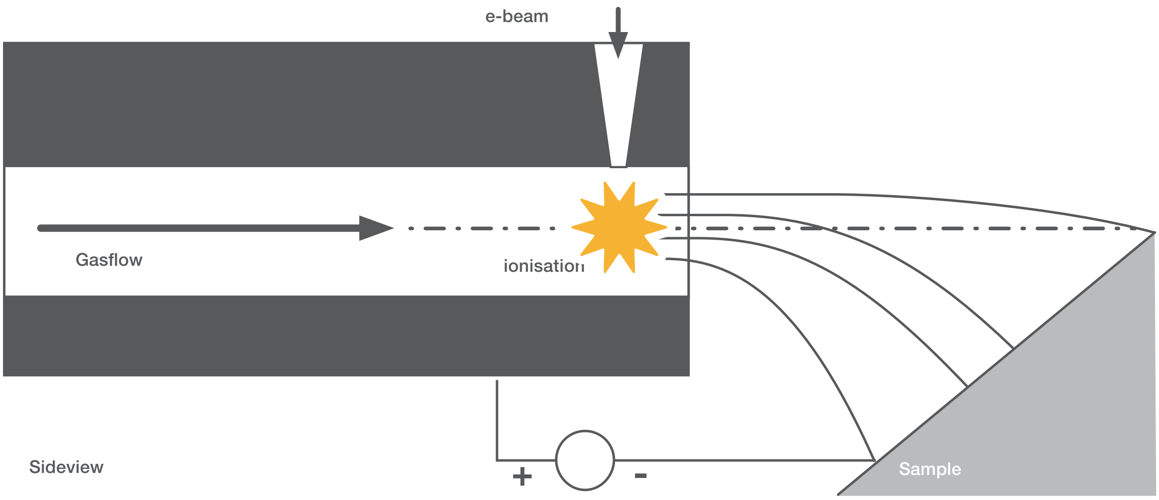

Figure 2: Schematic view of the µPolisher System, an *in situ*, low-energy ion source. Gas is delivered through a nozzle, where it is ionized with the SEM's electron beam. Ions are then accelerated towards a biased sample.

Results

This low-energy ion source can be used to improve the surface quality of bulk samples observed in the SEM or analyzed further with EBSD. It is well known that preparation of the sample surface is crucial for high-quality EBSD analysis. EBSD is an extremely surface-sensitive technique, as backscatter signal originates merely a few tens of nanometers below the surface. Residual stresses after mechanical polishing, oxidation, sample contamination after chemical etching lead to poor pattern quality, giving unreliable results and an increased number of non-indexed points during EBSD analysis. Cleaning the surface with the Thermo Scientific™ µPolisher System can reduce these effects, leading to a higher number of indexed points.

In addition to the improvement in surface quality, comparison of the surface of TRIP steel, before and after cleaning with the µPolisher System, reveals additional information, including channeling contrast within individual grains, as shown in Figure 3. The size of the cleaned area can be increased by changing the distance between the sample and the gas nozzle or by moving the stage continuously during exposure.

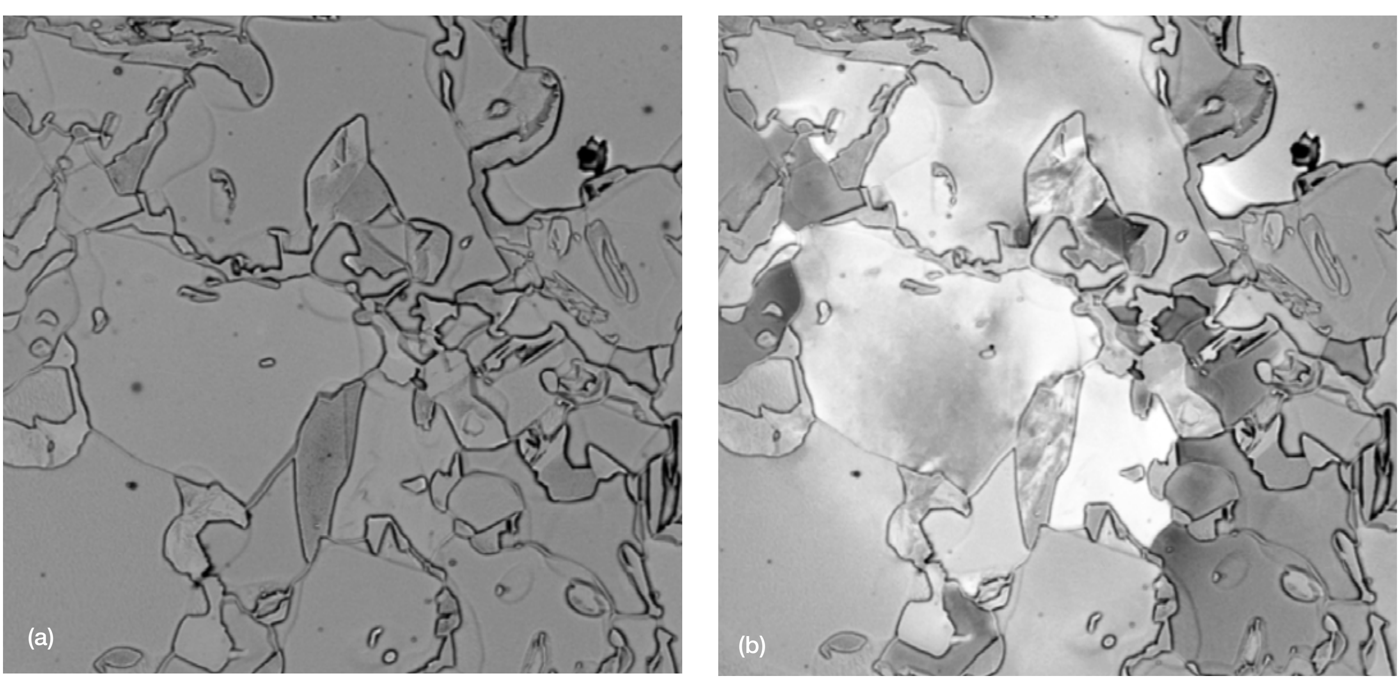

Figure 3: TRIP steel sample polished with a broad argon beam and left in air to develop an oxide layer. Comparison of the same region before (a) and after (b) two minutes of cleaning with the µPolisher System using 200 V argon ions. After the exposure, different phases in the material appear more distinct.

The production of thin lamellae with Ga FIB is an extremely efficient and fast method for creating TEM samples. However, one side effect is the amorphous damage layer created by the beam. Removal of such a damaged layer, and hence improving lamellae quality, is one of the promising potential applications of the µPolisher System. Figure 4 shows a silicon lamella prepared by Ga FIB and finished with 5 kV cleaning with an approximately 8 nm amorphous layer. After cleaning the same sample with 200 V argon ions using the µPolisher System, the amorphous layer was decreased to approximately 2 nm after one minute of exposure. Sputtering and redeposition of the material from the supporting grid, usually copper, to the lamella during cleaning is an issue for argon polishing systems using a broad ion beam, both standalone and integrated in a FIB-SEM. The µPolisher System significantly minimizes this effect since the FWHM diameter of the static ion beam is comparable to the size of the lamella itself, resulting in truly localized polishing.

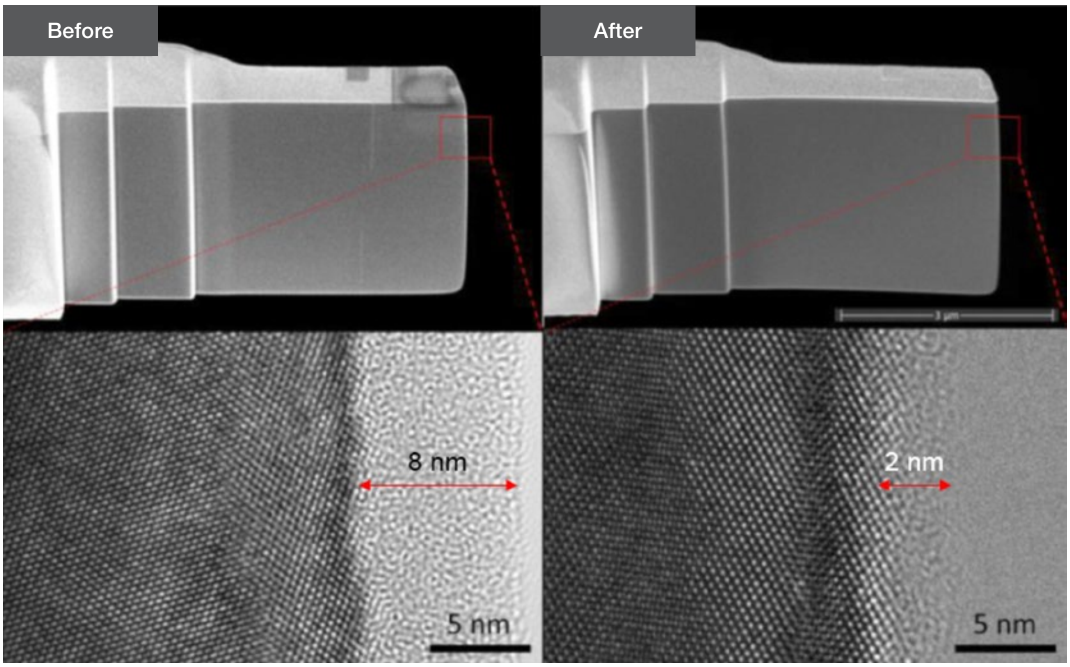

Figure 4: HRTEM images of a silicon lamella prepared by Ga FIB before (left) and after (right) cleaning with the µPolisher System, showing the reduction in the amorphous layer at the edge from approximately 8 nm to nearly 2 nm.

Conclusion

The applications of the µPolisher System, a low-energy ion source, are versatile, covering interaction types from chemical reactions to ion milling. Its truly localized targeting of the region of interest results in unique comparative capability between the same or neighboring regions before and after cleaning. The µPolisher System has been shown to be effective at removing surface contamination from metals, revealing channeling contrast within individual grains, as well as improving indexing of EBSPs in bulk samples. The reduction in the amorphous layer in lamellae prepared by Ga FIB has also been demonstrated using this technique. Surface modification using the µPolisher System may also be used for many other applications, for example, in cases where the sample oxidizes so quickly that external ion cleaning is not suitable.

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Notes

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