In-situ Low Energy Argon Ion Source for the Improvement of TEM and Bulk Sample Surface Quality

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An in-situ low energy Ar source module [1], uPolisher, enables localized low energy ion polishing of sample surfaces in the chamber of the scanning electron microscope (SEM) or a DualBeam system. This technique facilitates both local surface modification in bulk samples to remove hydrocarbon contamination for high resolution imaging, as well as removal of Ga implantation in TEM lamellae prepared by focused ion beam (FIB)[2]. Surface modification may also be necessary for samples that oxidize so quickly that an external ion cleaning is not suitable, or for TEM lamellae fabricated by Ga FIB that can become amorphized. Cleaning with uPolisher is shown to reduce this damaged layer to recover an artifact-free structure.

Localized cleaning is achieved by navigating to the area of interest using the SEM and inserting a retractable gas delivery nozzle. The inert Ar gas flow is ionized by the electron beam inside the nozzle with bore size ~30m placed about 100m away from the sample surface. The ions are accelerated towards the sample surface, biased in the range of 5 to 500V, where the cleaning occurs. The sample is typically exposed to the ion beam for 1-3 minutes, depending on the material composition and desired thickness removal. The cleaned region is generally elliptical in shape given by the geometry of the setup and consequently can be identified by a contrast change in SEM imaging. The cleaned region has a diameter ~10 to 20m, however larger areas can be easily cleaned by scanning the stage under the fixed nozzle.

It is well known that preparation of the sample surface is crucial for high quality electron backscatter diffraction (EBSD) analysis. EBSD is an extremely surface sensitive technique, as backscatter signal originates at only few tens of nanometers below the surface. Residual stresses after mechanical polishing, oxidation, sample contamination after chemical etching or processing with Ga ions in the DualBeam system lead to poor pattern quality giving unreliable results and an increased number of non-indexed points during EBSD analysis. Cleaning the surface with Polisher can reduce these effects, leading to a higher number of indexed points. Table 1 shows the improvement in the number of indexed points after polishing the surface of monocrystalline iron for 3 minutes using with Argon ions accelerated at 200V.

Table 1: Comparison of indexed points in a 50 m^2 area of monocrystalline iron before and after polishing with Ar ions of uPolisher.

Phase Name	Indexed Points (%)	
	Unpolished	Polished
Iron (bcc)	74.23	91.93
Zero Solution	25.77	8.07

The production of thin lamellae with Ga FIB is an extremely efficient and fast method to create TEM samples. However, one side effect is the amorphous damage layer created by the beam. Figure 1 shows a Si lamella prepared by Ga FIB and finished with 5 kV cleaning with a ~ 8 nm amorphous layer. After cleaning the same sample with 200V Ar ions using the uPolisher, the amorphous layer was decreased to ~2 nm after 1 min exposure. Sputtering and redeposition of the material from the supporting grid, usually Cu, to the lamella during the cleaning is minimized since the FWHM diameter of the static ion beam is comparable to the size of the lamella itself, resulting in truly localized polishing.

References:

[1] Mulders, J. and Trompenaars, P. 2016. An in-situ Low Energy Argon Ion Source for Local Surface Modification. European Microscopy Congress 2016: Proceedings. 453 - 454

[2] Prokhodtseva, A., Mulders, J., & Vystavel, T. (2017). Applications of an in-situ Low Energy Argon Ion Source for Improvement of TEM and SEM Sample Quality. *Microscopy and Microanalysis, 23*(S1), 298-299. doi:10.1017/S1431927617002173

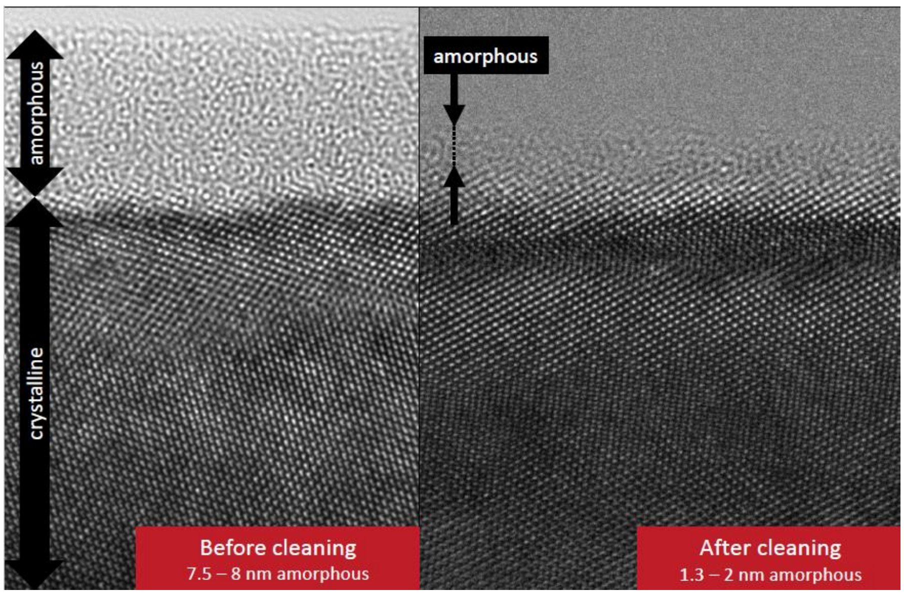

Figure 1: HRTEM images of a Si lamella prepared by Ga FIB before and after cleaning with uPolisher showing the reduction in the amorphous layer at the edge from ~8 nm to 2 nm.