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Applications of an in-situ Low Energy Argon Ion Source for Improvement of TEM and SEM Sample Quality

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An in-situ low energy ion source for SEM and DualBeam [1] is a new tool that could have a great number of applications that include, for example, reduction of the layer damaged by Ga ions in the TEM lamellae fabricated with Focused Ion beam, fine polishing of the sample surface in order to obtain EBSD patterns of the highest quality, or removal of residual hydrocarbons from the sample surface prior to high resolution SEM imaging.

The work principle of the source is based on the ionization of the primary atomic or molecular gas flow by the electron beam of the Scanning Electron Microscope. In this work we have used Ar, but other gases – O_2, Xe, Ne, etc. can be used as well depending on the application. Schematic view of an in-situ low energy ion source is shown on Figure 1. Gas is delivered through a nozzle with typical inner diameter of 30μm. Electron beam of low energy and high current is scanned through the slit in the nozzle thus generating ions by direct ionization and from beam interactions with the wall of the nozzle. The ions are then accelerated towards a biased sample located at a short distance, typically around 100um, from the nozzle. Energy of the ions could be varied in the range of 5 to 500V and width and direction of the ion beam depend on geometry only, and not on the charge and mass of the ion.

It is well known that production of thin lamellae with Ga FIB is an extremely efficient and fast method to create TEM sample, but surface of such lamellae is amorphized and implanted with Ga, which degrades their quality. Cleaning with 200V Ar ions helps to reduce this damaged layer. So ~9nm amorphous layer of a Si lamella fine polished with 5kV Ga ions was decreased to about 2nm after 1 min exposure to the low energy Ar ion source (Figure 2). Time that is necessary for the exposure can be determined from the milling rate estimated experimentally for given ionization conditions. EDX analysis showed a decrease of the Ga concentration to a level below the EDX measurement sensitivity, indicating complete removal of Ga. Note that sputtering of the material from the supporting grid, usually Cu, and its redeposition to the lamella during the cleaning are minimized, since the FWHM diameter of the static ion beam is comparable to the size of the lamella itself, resulting thus in truly localized polishing. In total, more than ten lamellae were processed in that way, confirming the reproducibility of the method.

The low energy ion source was also shown to improve the surface quality of bulk samples observed in SEM or analyzed further with EBSD. Figure 3 shows a bulk stainless steel sample that was electrochemically polished to create smooth scratch-free surface and then left in air for extended time. After exposure to a 200V Ar ion beam for 2min, an area of about 400um^2 was cleaned from contamination and ready for investigations – EBSD, or Electron Channeling Contrast Imaging of dislocations. In principle, the size of the cleaned area can be increased by changing the distance between the sample and the gas nozzle.

Applications of the low energy ion source are versatile, covering the interaction types from chemical reaction to ion milling. Very promising results are shown in creating a damage-free TEM lamella.

References:

[1] Johannes Mulders, Piet Trompenaars; An in-situ Low Energy Argon Ion Source for Local Surface Modification. The 16th European Microscopy Congress, Lyon, France.

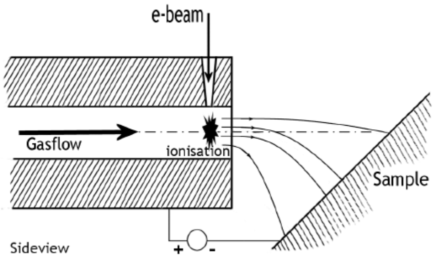

Figure 1. Schematic view of an in-situ low energy ion source. Gas is delivered through a nozzle, where it is ionized with the SEM’s electron beam. Ions are the then accelerated towards a biased sample.

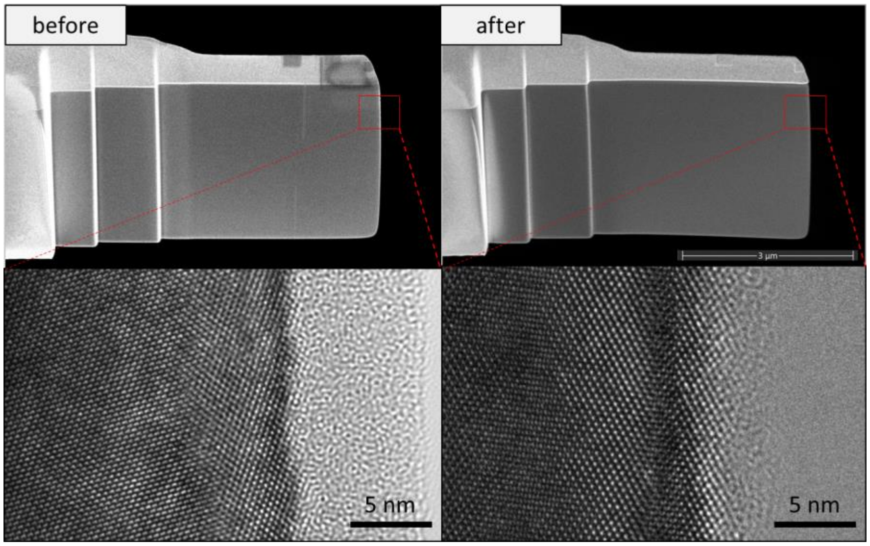

Figure 2. Si lamella before and after cleaning. SEM images show the removal of contamination from the e-beam imaging; HRES TEM images show the reduction of amorphous layer from about 9nm to less than 2nm.

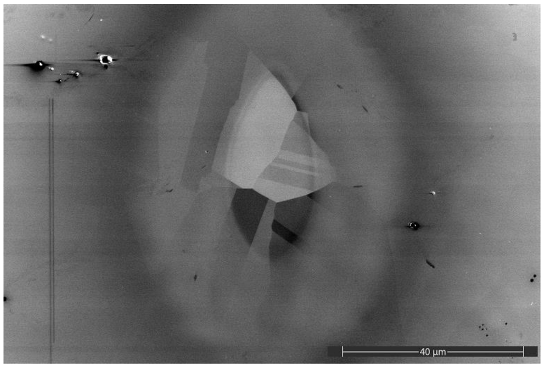

Figure 3. Stainless steel 316 cleaned for 2min. Central spot reveals the microstructure on the contaminated background.