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Using a Plasma FIB Equipped with Xe, N_2, O_2 and Ar for Atom Probe Sample Preparation – Ion Implantation and Success Rates

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Focused ion beam (FIB) is a common tool for the preparation of site-specific atom probe specimens. However, for conventional Ga^+ ion beam instruments, the removal rates are relatively slow for large sections of material (> 50 µm), as the Ga source has a low angular intensity, causing spherical aberrations that affect the milling rate [1]. Furthermore, using Ga^+ ions for the removal of material leads to damage in the form of amorphisation, defect generation in the topmost layer, and implantation of Ga^+ ions into the microstructure, which can lead to artefacts or even specimen failure during the atom probe analysis [2]. The development of the Xe plasma FIB (PFIB) has enabled much faster removal rates, as the milling rate is governed by the column rather than the ion source [1]. Xe ions also have a much lower penetration depth and radial range, causing less damage.

In this study the new Helios Hydra PFIB UXe, which is equipped with four different ion sources, Xe, N_2, O_2 and Ar, was used to prepare atom probe specimens. For each of the four sources, acceleration voltages of 30 kV, 12 kV, 5 kV and 2 kV were used to prepare atom probe tips from pure tungsten and aluminium. LEAP 3000Si and LEAP 4000XSi instruments were used, in voltage mode, to conduct the atom probe tomography (APT) experiments, with a pulse rate of 200 kHz, pulse fraction of 20%, target evaporation rate of 0.5 % and a temperature of 50 K. APT was conducted to determine the implantation depth of the different ions and to see how well the tungsten and aluminium specimens run compared to electropolished specimens, and specimens prepared using a Ga FIB. All obtained data was compared to the calculated stopping range of ions in matter (SRIM) [3]. These calculations, performed for all of the conditions named above, are shown in Figure 1.

Preliminary evaluation of the atom probe data indicates that specimens prepared using 30 kV acceleration voltage throughout the preparation fracture during the APT analysis, presumably due to ion damage. Lowering the acceleration voltage to 5 kV increased the success rate to almost 100 %.

The atom probe reconstruction in Figure 2, show Ar ions that were implanted into the W tip using a 30 kV acceleration voltage. To obtain accurate measurements, the dataset was corrected for time-of-flight (TOF), image compression factor (ICF) and field reduction factor (kF). It can be seen that the ions reach into a depth of 60 nm, at which point the tip fractured. The measured implantation depth of Ar in tungsten is much greater than determined by the SRIM calculations, where an average implantation value of 13.5 nm and a maximum implantation depth of 51 nm was determined. This discrepancy could be due to the fact that SRIM calculations do not consider the crystal structure of a material. It is therefore important to test different preparation parameters to be able to determine the capabilities and artefact creation of the PFIB when used for APT sample preparation.

References:

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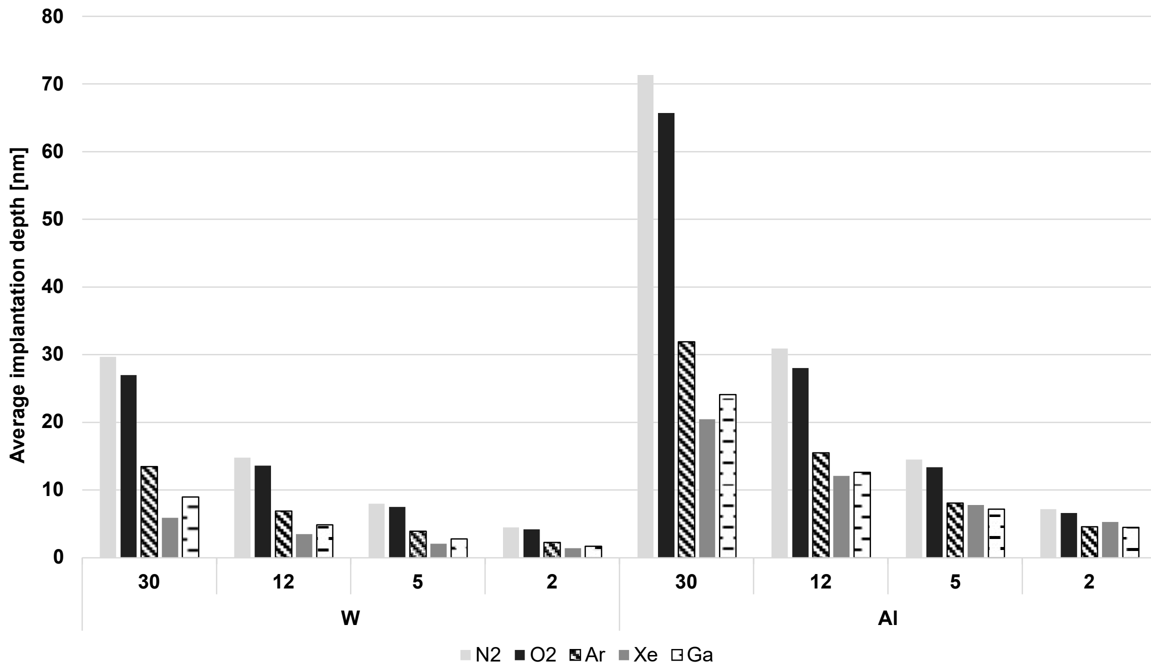

Figure 1. SRIM calculation of average implantation depth of different ion sources in tungsten (W) and aluminium (Al) at 30 kV, 12 kV, 5 kV and 2 kV.

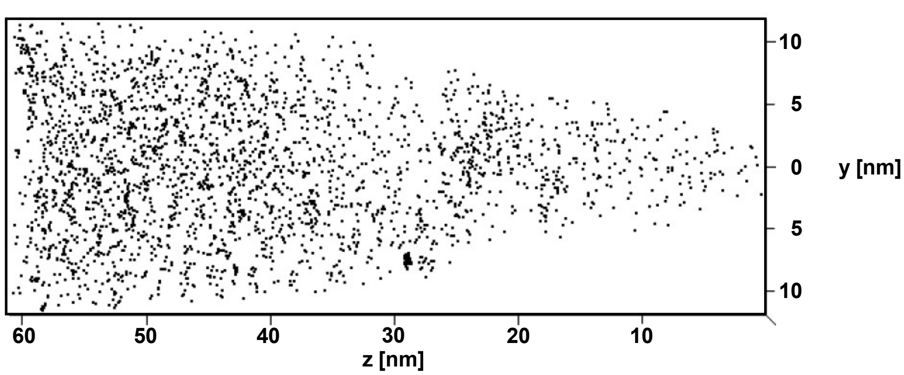

Figure 2. Atom probe reconstruction of a tungsten tip prepared at 30 kV acceleration voltage using Ar ions. Only Ar ions are shown in the reconstruction.

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