Elimination of focused ion beam-induced damage from atom probe specimens by small beam, low energy, argon ion milling

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Atom probe tomography (APT) is a powerful characterization technique for obtaining three-dimensional structure and composition of materials at the atomic scale. APT specimens are typically prepared using a dual beam focused ion beam (DB-FIB) because the tool provides rapid material removal, site specificity, and in situ electron beam imaging of the specimen while milling[1]. However, Ga-induced artifacts and amorphization from FIB milling can result in ambiguous results for characterization of Al/Al alloys[2] and Ga-containing materials[3]. Here we present low energy (< 1 keV) and small argon beam (< 1 micrometer) ion milling for the removal of FIB-induced damage in APT specimens prepared using the FIB.

Standard lift-out methods and annular milling were used to create Si and Al APT specimens at 30 keV in the FIB. Subsequently, argon ion milling was performed as the final cleaning step to remove the Pt cap. The Ar ions were rastered within a defined area and directed longitudinally at the APT specimen with decreasing milling energies (900 and 500 eV). In situ imaging of Pt cap removal and tip shaping were performed using the ion milling system's back-scattered electron detector (BSED). TEM, energy dispersive X-ray spectroscopy (EDS), and APT characterization were performed before and after ion milling to determine the removal of FIB-induced damage.

Figure 1a shows the amorphous damage on the Si APT specimen; Figure 1b shows Ga distribution within the interior sub-volume of the APT specimen after 30 keV removal of the Pt cap. A comparison of the amorphous damage seen in the TEM results and the Ga distribution seen in the APT three-dimensional reconstruction indicate that the amorphous layers are Ga-rich. These amorphous layers were significantly reduced after ion milling: from 50 nm to 5 nm in thickness at the tip and from 20-25 nm to less than 1 nm in thickness on the sidewalls. TEM images of the Si specimen after ion milling show fine needle shape (Figure 2), which indicates its viability for APT analysis. EDS and APT acquisition and analysis of the specimens after ion milling are underway. The feasibility of the methodology for the removal of FIB-induced damage will be investigated first in the Si APT specimens and then applied to the Al APT specimens.

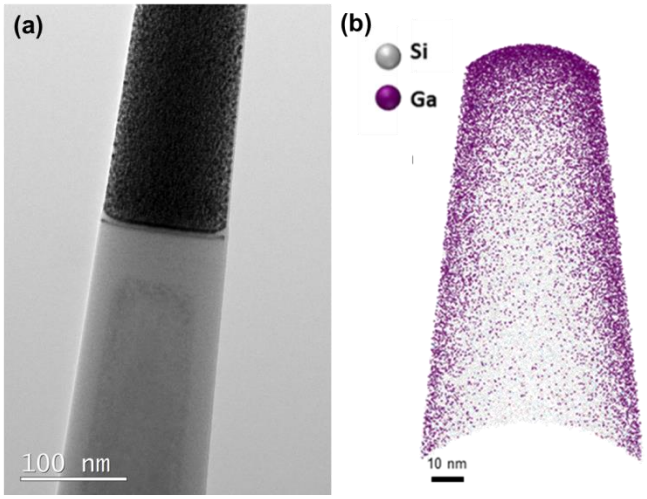

Figure 1. TEM image of a Si APT specimen as prepared (**a**) and Ga distribution from a three-dimensional APT reconstruction (**b**) after 30 keV FIB milling.

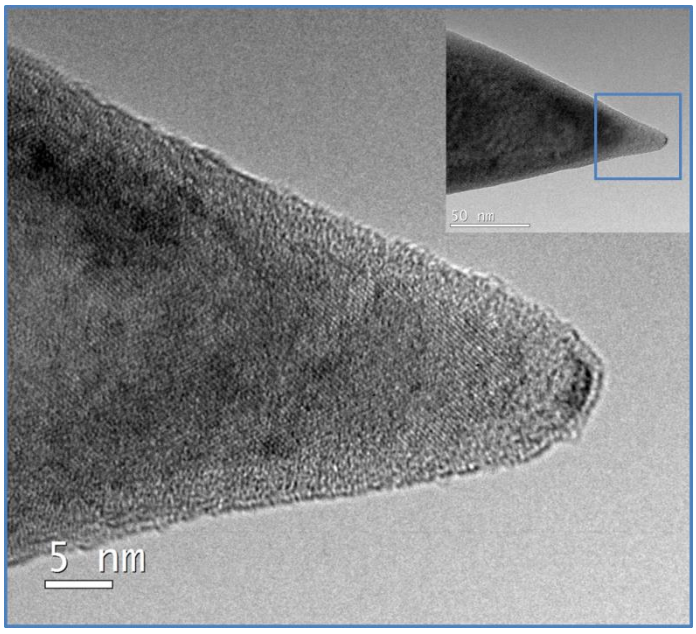

Figure 2. TEM image of the Si APT specimen following 900 eV argon ion milling shows significant reduction of the amorphous layer on the tip and sidewalls. The inset is a low magnification image with the area of interest marked.

References:
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