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Removal of Ga Implantation on FIB-prepared Atom Probe Specimens Using Small Beam and Low Energy Ar^+ Milling

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Atom probe tomography (APT) is a powerful characterization technique for obtaining three-dimensional structure and materials composition at the near atomic scale. It is also a complementary with other analysis techniques, such as transmission electron microscopy (TEM). In tandem, the two techniques provide a detailed characterization of structure and chemistry. APT specimens are typically prepared using a dual beam focused ion beam (DB-FIB), which is an efficient tool for removing a substantial amount of material, and in situ electron beam imaging allows more control when shaping the APT specimen tip [1]. However, Ga-induced damage and implantation from FIB milling can result in ambiguous results, especially for Al/Al alloys [2] and Ga containing materials [3]. Low energy (< 1 keV) Ar^+ milling has been shown to improve TEM specimen quality by removing Ga damage and implantation from FIB preparation [4-5]. Here, we present the use of small beam (< 1 μm), low energy Ar^+ milling for the removal of FIB-induced damage from APT specimens.

Si and Al APT specimens were prepared on a Si half-grid with multiple needle carriers. The needles were prepared in a FIB system [Thermo Fisher] using standard lift-out methods and annular milling at 30 kV [1]. Final cleaning steps were performed using an Ar^+ milling system [Fischione Instruments] prior to APT acquisition using a LEAP 5000 XR [CAMECA Instruments]. Ar^+ ions were rastered within a defined area and directed longitudinally at the needle at decreasing milling energies (900 and 500 eV). The protective Pt cap on the needle was removed by the ion milling system; its back-scattered electron detector was used to monitor the needle shape and size in situ. TEM, energy dispersive X-ray spectroscopy, and APT characterization were performed before and after ion milling to determine the removal of FIB-induced damage.

APT 3D reconstruction of Ga distribution on the Si needle shows Ga mostly concentrated on the tip and sidewalls of the needle (Figure 1)—note that APT only captures an interior sub-volume of the full needle, so the Ga damage on the sides of the needle extend from the reconstruction boundary to the real specimen needle boundary (an additional 20-30 nm). TEM similarly shows that these Ga-containing areas are amorphous and are 50 nm in thickness at the tip and 20 to 25 nm in thickness on the needle's sidewall (Figure 2b-c). After ion milling at 900 eV, these amorphous layers were significantly reduced to 5 nm at the tip and less than 1 nm on the sidewalls (Figure 2b) and the Pt cap completely removed. Furthermore, Ar^+ milling of the Si needle resulted to a very sharp tip (Figure 2c) which is ideal for APT. APT acquisition and TEM analysis of Ga distribution after argon ion milling are underway. The removal of the FIB-induced Ga damage using argon milling will be applied to the Al APT specimens.

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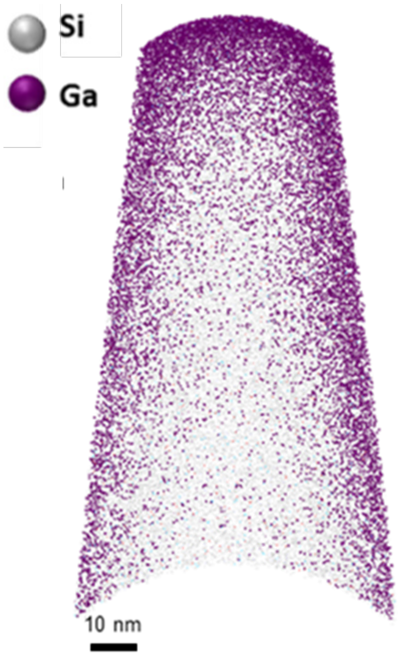

Figure 1. Three-dimensional APT reconstruction of Si APT specimen showing the Ga distribution after 30 kV FIB milling without a cleanup step.

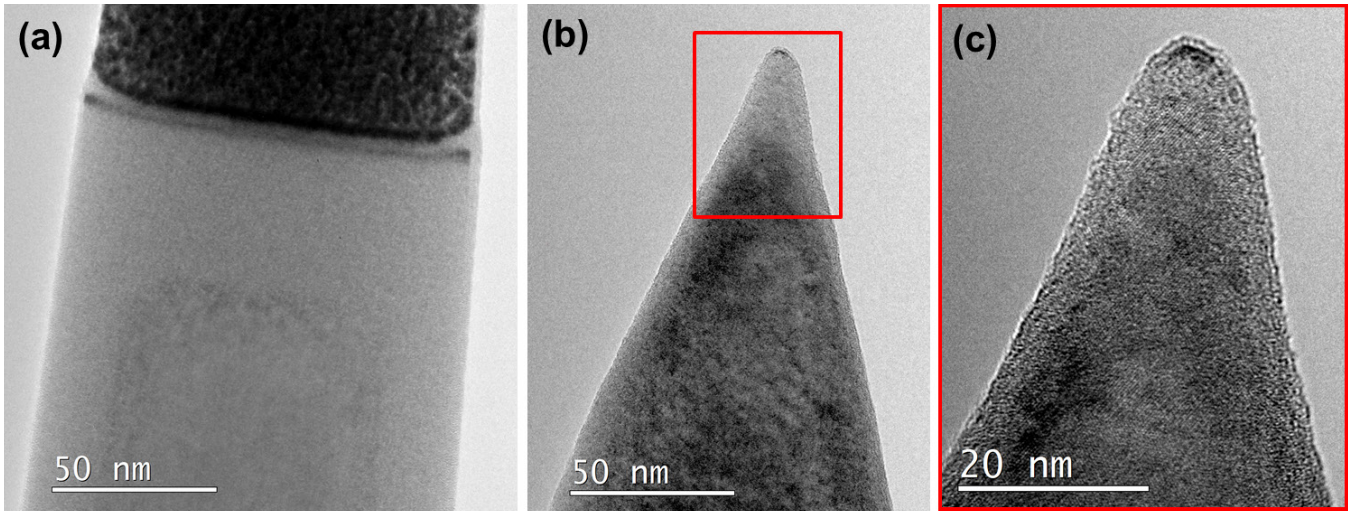

Figure 2. TEM images of the Si APT specimen after 30 kV FIB milling with the Pt protection cap (**a**) and following 900 eV argon ion milling (**b**). After ion milling, a significantly reduced amorphous layer and the sharp tip of the needle are seen (**c**).

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