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Optimal Specimen Preparation for Correlative Atom Probe Tomography and Electron Microscopy of Environmentally Sensitive Materials

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Correlative studies by atom probe tomography (APT) and transmission electron microscopy (TEM) are powerful characterization techniques for analyzing structural, morphological, and chemical properties of environmentally sensitive materials. A critical component of successful analyses is specimen preparation; probing individual atoms requires a surface and subsurface with minimal defects. However, environmentally sensitive specimens are susceptible to oxidation and contamination as they are transferred between instruments at ambient conditions during preparation and eventual characterization. The fast surface transformations that occur upon exposure to ambient conditions can be significantly suppressed by protecting the specimen's environment during the transfer from specimen preparation to characterization. Our previous work showed that concentrated ion beam (< 1 μm), low-energy Ar ion milling improves TEM and APT specimen quality by removing surface oxides and Ga damage caused by focused ion beam (FIB) preparation [1, 2, 3]. Here, we present specimen preparation using Ar ion milling techniques for subsequent atomic-scale characterization by TEM and APT under controlled environments. Broad and concentrated ion beam milling techniques are introduced to remove surface damage and oxidation, which is critical for probing atomic layers of specimens. Bulk Mg ribbons were initially prepared using a broad ion beam milling system [Model 1061 SEM Mill, Fischione Instruments] for removal of surface artifacts using the controlled environment workflow from our previous work [3]. The bulk sample was then transferred to a FIB system [Scios DualBeam, Thermo Fisher Scientific] in which the APT specimens were prepared. A FIB vacuum transfer system [PP3004 QuickLok, Quorum Technologies] was used to transfer the specimen between a glove box, FIB system, and concentrated ion beam milling system [Model 1080 PicoMill® TEM specimen preparation system, Fischione Instruments]. For ease of specimen handling and transfer, a half-grid with the mounted APT specimens was secured in the cartridge of a TEM vacuum transfer specimen holder [Model 2560 Vacuum Transfer Tomography Holder, Fischione Instruments]. Concentrated ion beam milling was performed to remove the FIB-induced damage on the specimen. Post ion-beam milling, energy dispersive X-ray spectroscopy (EDS), and TEM characterization were performed. Subsequently, the specimen was transferred to a local electrode atom probe (LEAP) [CAMECA Instruments] system by way of an environmental transfer hub (ETH) station [4] for further analyses. As control, specimens from bulk samples without broad ion beam milling will be prepared and analyzed under ambient and controlled environments. APT analysis of specimens exposed at ambient conditions and after Ar concentrated ion beam milling (Fig. 1) show trace amounts of oxides, hydroxides, and Ga (inset). The Ga was located primarily at the tip of the specimen which is possibly some damage layer not completely removed during Ar ion milling. However, no Ar implantation was observed on the specimen after Ar ion milling. Under controlled environments, EDS at 5 kV (Fig. 2a) acquired after Ga FIB and Ar concentrated ion beam milling preparation shows a significant decrease in O and Ga signals. The reduced O signal following Ar ion milling, as compared to post-FIB milling shows the elimination of surface oxides and the effectiveness of the transfer systems. The EDS maps (Fig. 2b) indicate a substantial amount of Ga, which is associated with the Pt cap; this was significantly removed during Ar ion milling at 1.5 kV. Analyses of specimens prepared by Ar ion milling (broad and concentrated) under ambient and controlled environments are underway so that the results can be compared to the initial results described here.

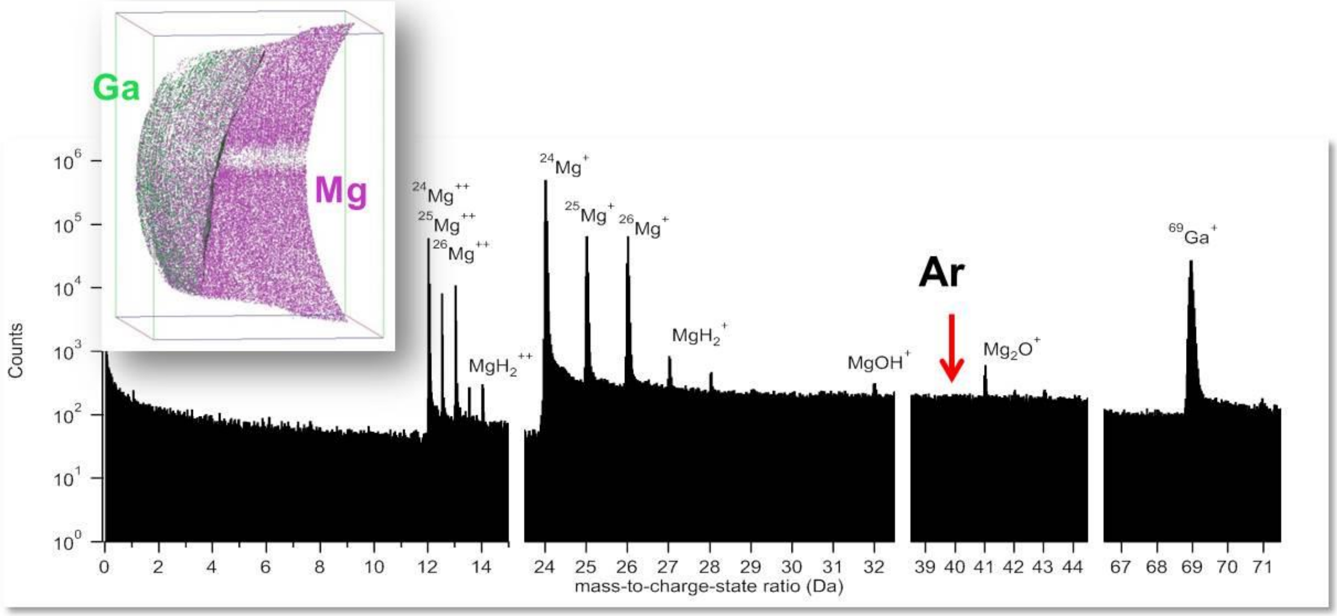

Figure 1. Atom probe tomography mass spectra and elemental reconstruction (inset) of the Mg specimen after Ar ion milling at ambient conditions shows traces of oxide and hydroxides and no Ar implantation. Inset shows that the presence of Ga is primarily confined to the tip of the specimen and was left-over Ga from Ar milling.

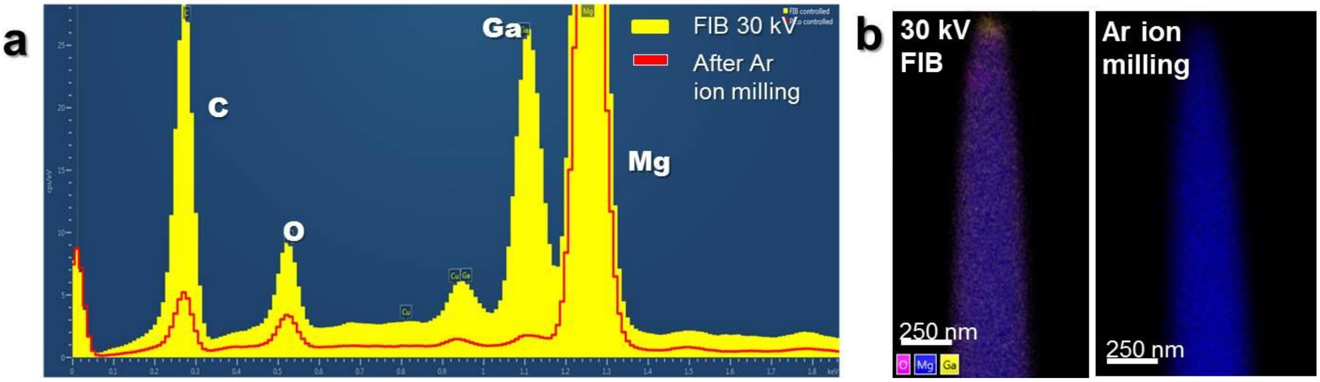

Figure 2. EDS spectra at 5 kV acquired from an Mg APT specimen prepared in controlled environments (a). After 30 kV FIB, Ga is primarily distributed at the cap; O is present on all surfaces of the APT specimen (b, left). After 30 kV FIB followed by Ar milling, no Ga nor O are detected (b, right).

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