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Meeting-report

Elucidating Surface Properties by Correlative TEM and APT Studies of Ideal Mg Specimens Prepared Under Controlled Environments

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Probing individual atoms to reveal the underlying 3D compositional distribution using atom probe tomography (APT) and other correlative imaging techniques, such as scanning transmission electron microscopy (STEM), requires a surface and subsurface with minimal unintentional defects introduced during specimen preparation. For this reason, specimen preparation is a critical component of successful correlative analyses that involves structural characterization by transmission electron microscopy (TEM) combined with compositional analysis APT using the same specimen. The potential of these powerful analytical techniques can be hindered by surface damage, either by implanted Ga during focused ion beam (FIB) specimen preparation or environmental degradation during specimen transfer in an ambient laboratory environment. Our previous works [1-3] have shown the reduction of surface damage and oxidation by both broad and concentrated Ar ion beam milling techniques under controlled environments. Here, we extend our work to differentiate the Mg bulk surface and subsurface properties as it relates to Ga diffusion from optimal specimens that were prepared using a controlled environment workflow.

Polished and unpolished bulk Mg ribbons were transferred under controlled environment, in a manner similar to the method described in our previous work [3]. The following workflow was employed:

- Ar broad ion beam (BIB) milling system [TrionMill, Fischione Instruments] for the removal of surface damage (the unpolished bulk sample remained in a glove box during this step).
- Electron backscattered diffraction (EBSD) analysis (using SEM imaging and Ga FIB) for identifying regions of interest (ROIs) and lift-out areas. Specimens were transported under inert environment into a glove box for storage and handling.
- Bulk samples were mounted and transferred from a glove box into the Ga FIB for specimen preparation.
- Ar concentrated ion beam (CIB) milling [NanoMill® TEM specimen preparation system, Fischione Instruments] for post-FIB specimen preparation.

The transfer of the bulk sample and subsequent APT specimens between the BIB mill, glove box, FIB/SEM, CIB mill, and analytical imaging was performed while protected from the environment using various shuttle suitcase devices.

The Mg specimen prior to BIB milling (Fig 1a) shows a rough surface morphology that was caused by the commercial bulk processing of the Mg ribbon. This roughness persists even after preparation of APT needles and post-FIB Ar polishing of the APT specimen (Fig 1b). In comparison, the BIB-milled Mg specimen reveals the polycrystalline morphology of the bulk metal surface (Fig 1c) and shows an improvement in overall contrast and surface homogeneity after APT preparation and post-FIB Ar polishing (Fig 1d). These results suggest that the Mg APT specimen prepared in Fig 1b was made within the native damaged and oxidized surface region, while the specimen in Fig 1d was made from within the metallic Mg. Proximity histograms from APT analysis (Fig. 2) near the tip region centered at the interface of the surface implanted Ga and underlying Mg metal are shown as insets in Fig. 2. At the interface of the Mg and Ga implanted region, 0.5-1.5 atomic% of O and H from the unpolished bulk Mg (Fig. 2a) were observed. These composition of O and H correlate with the implied passivated oxide composition of unpolished bulk sample from which the APT specimen was extracted. Furthermore, no grain boundary inside the specimen was apparent. Conversely, the results from the BIB-milled Mg specimen showed 0.25-0.75 atomic% of O and H (Fig. 2b) and a distinct grain boundary decorated with Ga (inset of Fig 2b). The low O and H amounts (Fig 2b) indicated a metallic Mg specimen. Diffusion of Ga via the grain boundaries of the polycrystalline specimen (inset of Fig 2b) occurred during FIB preparation. Analysis of Ga concentrations on the surface and the grain boundary, as well as driving forces for Ga diffusion in metallic Mg, will be discussed [4].

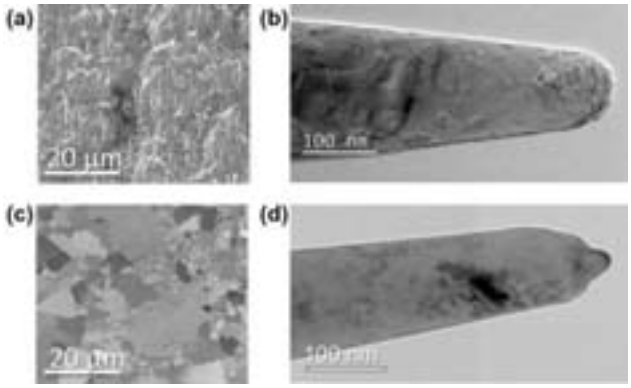

Fig. 1. Scanning electron microscopy images of the (**a**) unpolished bulk Mg sample and (**c**) the same bulk sample after broad Ar ion beam milling. Transmission electron microscopy images of the atom probe tomography (APT) specimens (**b**, **d**), which were lifted from samples **a** and **c**, respectively. APT specimens were prepared under controlled environments using a Ga focused ion beam tool, followed by Ar condensed ion beam milling.

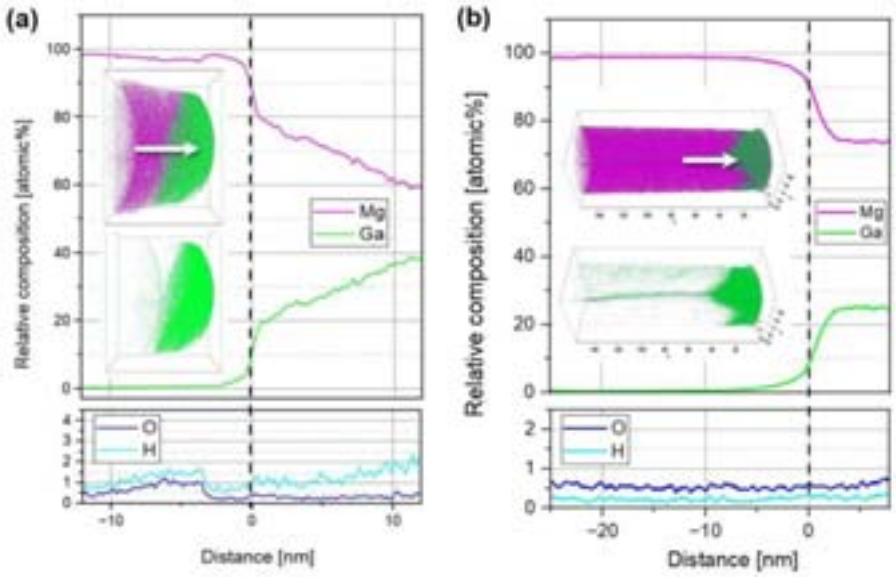

Fig. 2. Atom probe reconstructions (insets) of the APT specimens prepared under controlled environments from the Mg bulk samples shown in Fig. 1: unpolished (**a**) and polished (**b**) by Ar broad ion beam mill. Atomic composition as proximity histograms were extracted along the direction indicated by the arrow on the reconstructions (insets).

References

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