3176
doi:10.1017/S1431927622011795
Microsc. Microanal. 28 (Suppl 1), 2022

Argon Milling of Bulk and Post-FIB Specimens for Multi-Length Scale Analyses by EBSD, TEM, and APT under Controlled Environments

C.S. Bonifacio[1], D. Perea[2], P. Nowakowski[1], M.L. Ray[1] and P. Fischione[1]

[1.] E.A. Fischione Instruments, Inc., Export, PA, USA.
[2.] Pacific Northwest National Laboratory, Richland, WA, USA.
* Corresponding author: cs_bonifacio@fischione.com

Correlative investigations involve structural characterization by transmission electron microscopy (TEM) combined with compositional analysis by atom probe tomography (APT) using the same specimen. A critical component of successful correlative analyses is specimen preparation; probing individual atoms requires a surface and subsurface with minimal defects, beginning with the bulk material through to the prepared specimen for TEM or APT analysis. Our previous work showed that low-energy concentrated ion beam Ar ion milling improves TEM [1] and APT [2,3] specimen quality by removing surface oxides and Ga damage caused by focused ion beam (FIB) preparation. Further enhancement in specimen quality of APT specimens is achieved in the established workflow under controlled environments, as shown in [4]. Here, we present specimen preparation using Ar ion milling techniques not only for the APT specimen, but also the bulk sample for subsequent multi-length scale analyses by electron backscatter diffraction (EBSD), TEM, and APT under controlled environments. The removal of surface damage and oxidation, which is crucial for probing atomic layer specimens by broad and concentrated Ar ion beam milling techniques under controlled environments, is highlighted.

Using the controlled environment workflow from our previous work [3], bulk Mg ribbons were initially prepared using a broad Ar ion beam (BIB) milling system [TrionMill, Fischione Instruments] for removal of surface artifacts. The bulk sample was then transferred under protected environment to a FIB system [Scios DualBeam, Thermo Fisher] for imaging and EBSD analysis. FIB specimen preparation of the APT specimens followed by concentrated Ar ion beam (CIB) milling [PicoMill® TEM specimen preparation system, Fischione Instruments] and subsequent TEM and APT characterization were performed under protected environments based on work in [4]. APT specimen was then transferred to a local electrode atom probe system [LEAP, CAMECA] by way of an environmental transfer hub (ETH) station [5] for further analyses. Based on EBSD analysis, specific regions on the BIB milled Mg specimen were selected; from these regions APT specimens were prepared and characterized under controlled environments.

Figure 1a and 1b show before and after BIB milling of the Mg bulk sample's surface. A significant improvement in the specimen surface is observed with the individual grains of the sample revealed after Ar milling (Fig. 1b). After FIB preparation and post-FIB Ar milling of the APT specimen, improvement in overall contrast, surface homogeneity, and distinction of grain boundaries were observed in the TEM image of the APT specimen prepared from the BIB-milled bulk sample (Fig. 1d). This difference can be related to the removal of surface artifacts, such oxides, contamination, and structural defects from materials processing present on the unpolished specimen (Fig. 1a and 1c). Without Ar milling on the bulk sample (BIB milling) and FIB prepared APT specimen (Fig. 2a), Ga and a significant amount of Mg oxides (MgO_3^{2+}, Mg_2^{2+}, $Mg_3O_2^{2+}$) were detected. These oxide species were absent in the specimen prepared from the BIB-milled bulk sample and post-FIB CIB-milled specimen (labels between Fig. 2a and Fig. 2b). Additionally, Ga was found throughout the tip (not shown) after FIB preparation. Ga was

reduced to only the apex of the tip (inset of Fig. 2b) and Ga^+ was absent after post-FIB CIB milling (Fig. 2b). EBSD analysis from the polished bulk specimen is underway. Results from APT analysis by controlled environments on the preselected region will be correlated to the EBSD and TEM results [6].

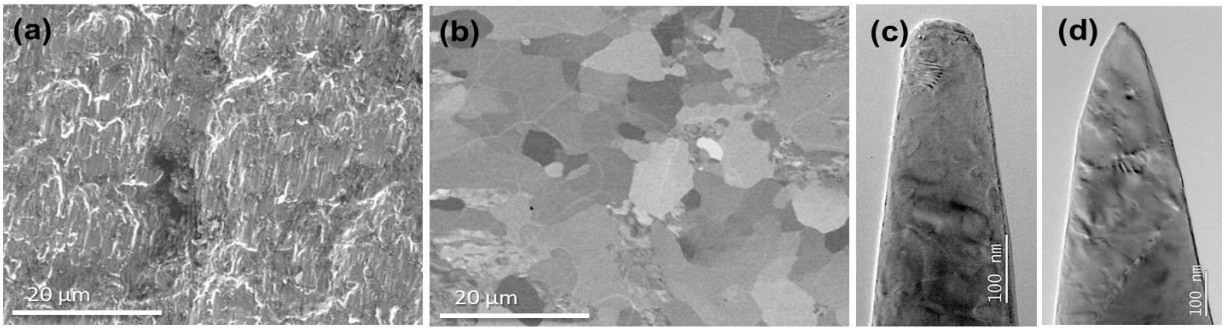

Figure 1. SEM images of the bulk Mg sample as received, unpolished (**a**) and after broad Ar ion beam milling (**b**). TEM (**c**, **d)** of the atom probe tomography specimens, which were prepared from bulk samples (**a**, **b,** respectively), under controlled environments and after focused ion beam milling (Ga) followed by condensed ion beam milling (Ar).

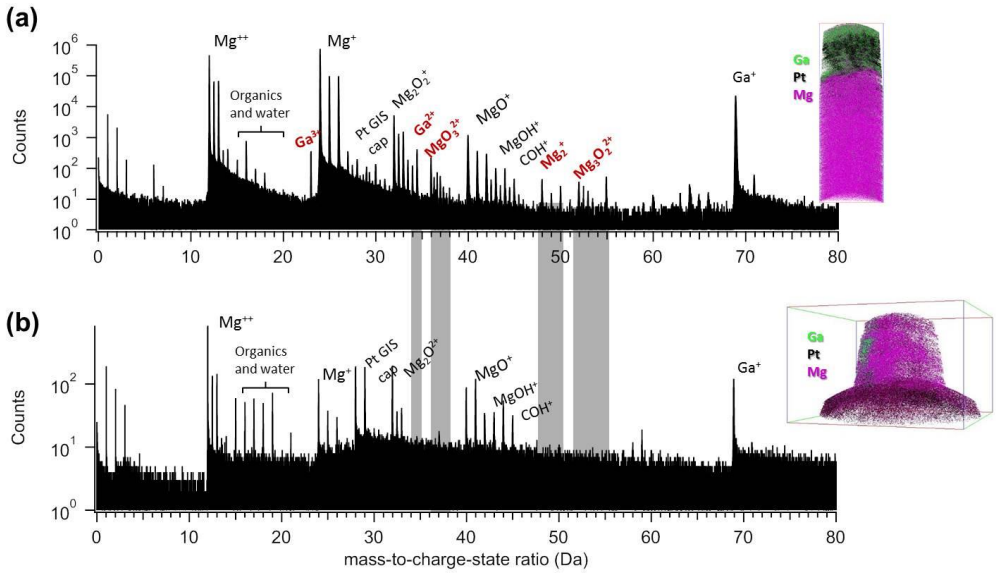

Figure 2. Comparison of APT mass spectra with elemental reconstruction (insets) from the Mg specimen: unpolished and FIB-prepared (**a**) and broad Ar ion milled polished and post-FIB Ar milled (**b**). The difference in detected species between the two specimens was highlighted by the marked areas (gray) between the two spectra.

https://doi.org/10.1017/S1431927622011795 Published online by Cambridge University Press

References:

[1] PE Fischione et al., Microsc. Microanal. **23(4)**, (2017), pp. 782-793.
[2] CS Bonifacio et al., Microsc. Microanal. **24(S1)**, (2018), pp 1118-1119.
[3] CS Bonifacio et al., Microsc. Microanal. **25(S2)**, (2019), pp. 2554-2555.
[4] CS Bonifacio et al., Microsc. Microanal. **27(S1)**, (2021), pp. 2472-2474.
[5] DE Perea et al., Adv. Struc. Chem. **3(1)**, (2017), Article ID 12
[6] The authors acknowledge resources and support from Environmental Molecular Sciences Laboratory, a national scientific user facility sponsored by the DOE Office of Biological and Environmental Research at Pacific Northwest National Laboratory.

https://doi.org/10.1017/S1431927622011795 Published online by Cambridge University Press