1634 CD
DOI: 10.1017/S1431927607071437

Microsc Microanal 13(Suppl 2), 2007

Recent advances in FIB-based site-specific atom probe specimen preparation techniques

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The technique of atom probe tomography (APT) has seen major advances in recent times. The latest developments such as wide field of view detectors and laser pulsing, allow much larger volumes of material to be analyzed in shorter times with greater mass resolution and are extending the application of APT to materials research of poorly conducting materials such as ceramics, intrinsic semiconductors and glasses.

While the research applications of the atom probe is increasing rapidly due to these technological advances, preparation of the required needle-shaped specimens with an end radius of ~100nm still remains a major limiting factor for many materials, especially where it is desired to analyse a specific region within the sample. There have also been major advances in the area of APT specimen fabrication methods, many of which utilise focused ion beam (FIB) – based methodologies.

A number of labs are working on developing and optimizing FIB-based specimen preparation techniques for the preparation of site-specific specimens. The techniques used in different laboratories vary, though most use in-situ or ex-situ lift-out methods for the most difficult site-specific samples [1-5]. In this presentation, we will show the methods that have been found to be the most effective at the University of Sydney, with an emphasis on methods of fabrication of specimens containing interfaces that are oriented both parallel to the direction of the tip *and* perpendicular to the direction of the tip. Most of the currently-used specimen preparation methods result in the interfaces at the former orientation, however, the latter is sometimes desired as it provides greater spatial resolution across the interface due to the trajectory aberrations which limit the lateral spatial resolution.

We will also describe our ongoing efforts to develop a simple, rapid and reproducible method to combine both transmission electron microscopy (TEM) and APT for the study of interfaces. This allows high-resolution structural information about boundaries to be obtained using TEM and compared to the quantitative compositional information obtainable using APT. One approach is to examine finished atom probe tips in the TEM prior to running them in the atom probe. Figure 1 shows an atom probe tip prepared from a metallic glass specimen that has been fabricated from the nanocrystalline region specified in figure 1(b). Figure 1(c) in an image of the tip in the final stages of preparation, figure 1(d) is the TEM image of the tip and figure 1(e) is the resulting atom map. This approach is not ideal for TEM due to both the size and geometry of the specimen, which makes diffraction studies practically difficult, and the proportion ion-damaged material compared to the overall specimen size (the ion damaged layer can be clearly discerned in figure 1(d)). When atom probe data is analysed, it is possible to discard the data from the outer, Ga^+-implanted region, and consider only the undamaged material deeper within the tip. However this damaged layer can prevent high quality TEM studies to be performed on the fabricated tips. To avoid this problem, our approach involves the preparation of atom probe specimens directly from electropolished TEM discs using a lift-out method, allowing both detailed TEM and APT to be carried out on a single interface. TEM may be used for structural characterisation, and to obtain information from many different grain boundaries over a relatively large area. Compositional information using EDXS and EELS can also be obtained for comparison with the APT data. APT is used to obtain information about the precise composition at the grain boundaries in three dimensions.

References

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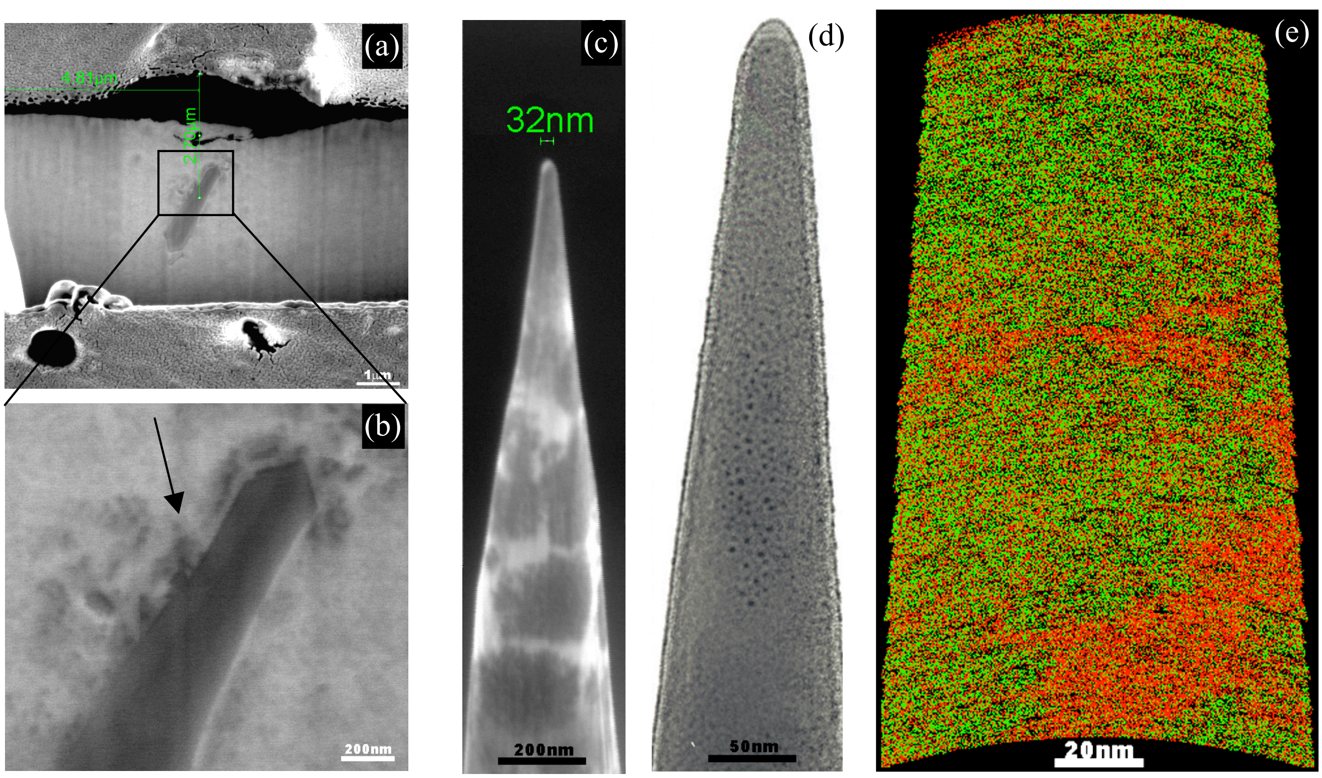

FIG. 1. A $Mg_{65}Cu_{25}Y_{10}$ metallic glass specimen cooled from 585°C at rate of 500°C/sec and heat treated at 160 or 165°C for 120 minutes. (a) and (b) specimens are fabricated from the nanocrystalline region close to the crystallite, as indicated by the arrow in (b), (c) electron beam generated FIB image during the final stages of milling, (d) TEM image, and (e) the resulting atom map. Of interest is the interface between the crystalline and amorphous regions in this sample.