FIB Specimen Preparation for Atom Probe Tomography

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The essential geometry of an atom probe tomography (APT) specimen is a sharp needle that tapers with a modest angle to an apex radius that is 50 to 100 nm. The small specimen size provides both magnification of surface features and creation of the high (~10-50 V/nm) electric field necessary for field evaporation to occur. Because the needle-shaped specimen is the primary optic of the microscope, specimen preparation is critical to successful APT analysis.

The general availability and adoption of focused-ion-beam (FIB) specimen preparation methods over the past 10-15 years, coupled with commercially available laser-pulsed APT instruments, has ushered in a period of rapid acceleration of APT application to new materials, including ceramics and microelectronic devices. Early FIB-based methods used to fabricate specimens for APT relied on attaching a volume of material to the end of a needle (using non-FIB methods) and shaping the end-form for APT analysis with FIB [1]. Optimal annular milling methods were developed that provided for improved emitter shapes, while adoption of TEM lift-out methods improved repeatability and reduced preparation time [2, 3]. Today, many applications and variations of the standard lift-out and sharpening methods have been reported. These include variations enabling analysis parallel to the original specimen surface or inverted relative to the original specimen surface. See reference [4] for a recent review of these methods.

Some preparatory preprocessing or deprocessing is often necessary before specimen lift-out and sharpening can proceed. Preprocessing might include applying a protective sacrificial cap to the top surface, while deprocessing might include removal of unnecessary superstructure material to allow easier access to a region of interest (ROI). These methods may be simple (i.e., mechanically scraping away epoxy or other protective coverings) or more sophisticated (FIB milling or chemical etching for removing superstructure layers that are not of interest). Once the ROI is properly processed and positioned with respect to the top surface of the host material, the lift-out process can be initiated. A multi-step process is employed where material is removed during a milling procedure to produce a cantilevered wedge of material containing the ROI. The wedge is then removed by a micromanipulator and transferred to carrier tips. Subsequent sharpening of the transferred material via an annular milling procedure followed by a low energy "clean-up" step is also the norm. Variations to the standard lift-out process will be discussed that allow for analyses to proceed in directions other than downward (parallel to the surface normal). Also, preparation of specimens for hybrid TEM/APT analysis will be presented.

[1] D. J. Larson, et al., *Nanotechnology* 1999, 10, 45-50.
[2] K. Thompson, et al., *Ultramicroscopy* 2007, 107, 131-139.
[3] M. K. Miller, et al., *Microscopy and Microanalysis* 2007, 13, 428-436.
[4] D. J. Larson, et al., Atom Probe Tomography for Microelectronics. In *Handbook of*

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