Sculpting Needle-shaped Atom Probe Specimens with a Dual Beam FIB

M.K. Miller*

*Metals and Ceramics Division, Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6136

The goal of atom probe specimen preparation is to produce a needle-shaped specimen with an end radius of less than 50 nm on a smooth uniform shank with a taper angle of less than 5°. Neither the microstructure nor the solute distribution should be altered during fabrication. Traditionally, electropolishing has been the dominant method for producing these needles. Focused ion beam (FIB) instruments enable specimens to be fabricated from a wider range of materials and different forms [1].

Local electrode atom probe specimens may be made in-situ in bulk or powder specimens with the moat method, Fig. 1. In this method, a large diameter (typically 50-100 μm) moat is milled around the region of interest with a high ion current. The outer diameter of the moat has to be sufficiently large to allow the local electrode in the atom probe to define the field at the specimen and not interfere physically or electrically with the sides of the moat. The central region is then milled into a sharp point with a succession of annular masks of decreasing inner diameters and with decreasing ion currents [2]. Before milling, platinum deposits are used to protect the region of interest from damage associated with gallium implantation, Fig. 2. Platinum deposits along the side of the shank may also be used to improve the electrical conductivity of high resistivity materials, Fig. 2.

Atom probe specimens may be fabricated with a minimum of user interaction with graduated grey scale bitmaps, Fig. 3. No milling occurs in the central black region of the bitmap. In this example of a specimen fabricated from a Bosch etched silicon post, one low and one high magnification application of the same bitmap were used. The use of bitmaps also permits the sculpting of the shank of the post to improve its mechanical stability and electrical conductivity.

Site specific atom probe specimen preparation may also be performed with a lift-out method, Fig. 4. The ~3 μm square by 5-10 μm long bar may be cut either parallel or perpendicular to the surface of the bulk specimen so that grain boundaries, interphase interfaces, low volume fraction phases, surface, embedded, or implanted layers, etc. may be examined. Surface platinum deposits may also be used to ensure that the region of interest is positioned on the centerline of the resulting bar. The dimensions of the bar are selected to minimize gallium penetration to the centerline of the specimen. After lift-out, the bar is attached to a suitable support, such as a fine wire, with platinum deposit and then annularly milled into a sharp point [3].

References

[1] M.K. Miller, K.F. Russell and G.B. Thompson, *Ultramicroscopy*, **102** (2005) 287.
[2] D.J. Larson, et al., *Ultramicroscopy*, **79** (1998) 147.
[3] The author thanks Dr. L.A. Giannuzzi for invaluable assistance. Research at the SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

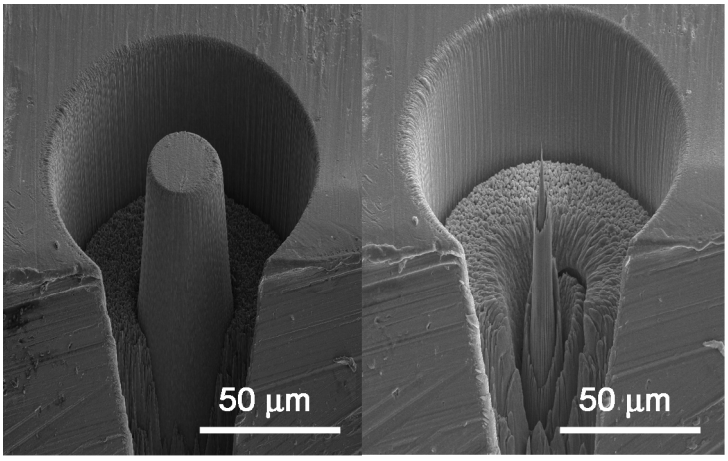

FIG. 1. Atom probe specimen fabricated in-situ in a nanocrystalline nickel specimen with the moat method.

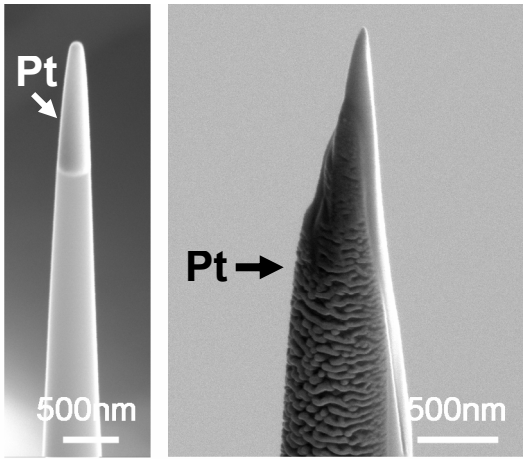

FIG. 2. The use of platinum deposit to protect and provide electrical conduction.

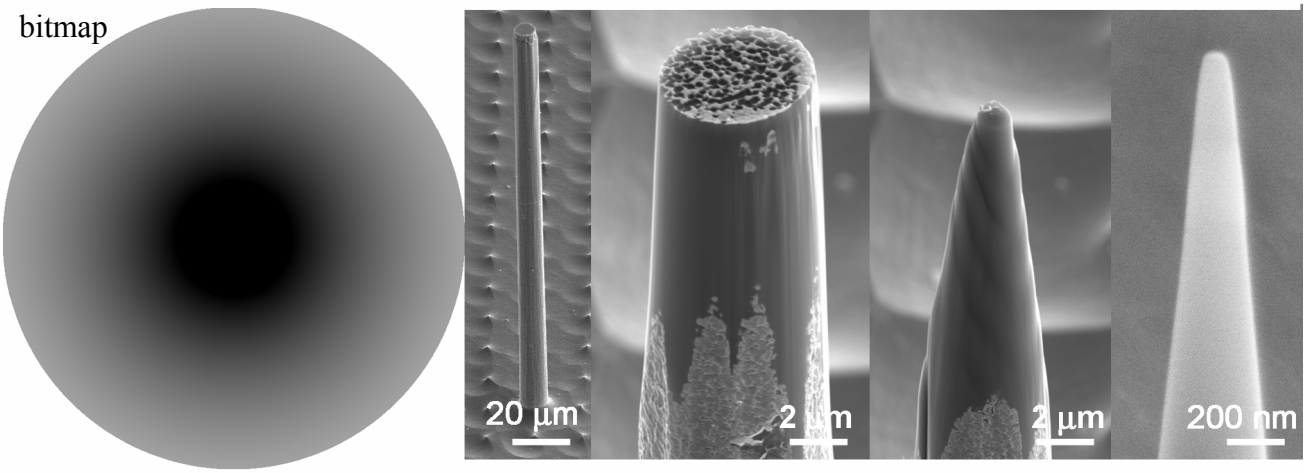

FIG. 3. Atom probe specimen fabricated from a ~5µm diameter silicon post with 2 applications of a graduated grey scale bitmap. No milling occurs in the central black region of the bitmap.

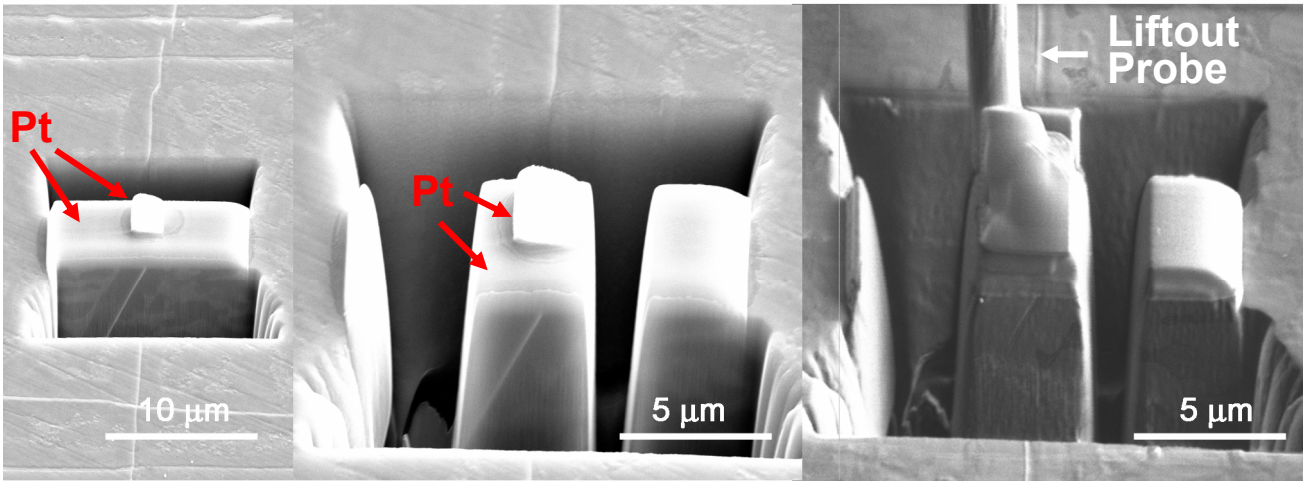

FIG. 4. Site specific atom probe specimen preparation of a thin topologically close packed (TCP) phase in a nickel-base superalloy with a lift-out procedure. The ~3 µm square post is subsequently annularly milled into a sharp needle.