STRATEGIES FOR FABRICATING ATOM PROBE SPECIMENS WITH A DUAL BEAM FIB

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Electropolishing has been the dominant technique for preparing atom probe specimens. Recently, focused ion beam (FIB) based methods have enabled transmission electron microscopy (TEM) specimens to be fabricated from a wide range of materials. Dual beam instruments with a scanning electron microscope and a gallium ion beam column have been found to be beneficial for preparing atom probe specimens as the milling operation may be continuously monitored in real time with the electron beam. In addition, the amount of gallium implanted into the specimen is significantly lower as the electron beam may be used for imaging.

FIB-based methods for the fabrication of atom probe specimens from thin ribbons, sheet stock, and powders have been developed. Previously, standard electropolishing methods have been used for thin sheet materials. However, the cross section of the apex region tends to be non circular which leads to problems in reconstruction of the atom position. The ribbon is mounted vertically in a copper stub. To reduce the milling time, the sides of the ribbon near the exposed end may be removed by mechanical grinding. Fine powders are difficult to prepare because of their small size. With the use of a micromanipulator, a single powder particle may be mounted on the end of a needle with either epoxy or platinum deposit. In both materials, a platinum cap is laid down over the area of interest with electron- or ion-beam deposition to protect it during subsequent ion milling. The needle with a suitably sharp end radius is then milled with the use of an annular mask with successively decreasing inner radii, as described by Larson et al. [1].

Two FIB-based methods for preparing atom probe specimens at site specific locations such as coarse precipitates, grain boundaries, interphase interfaces, denuded zones, heat affected zones, implanted, near surface and subsurface regions and shear bands have been developed. In the moat method, a protective platinum cap is deposited over the region of interest. A large annular region around this cap is then removed with a high gallium ion current (>20 nA). The outer diameter (typically 100 μm) has to be sufficiently large to allow the local electrode to define the field at the specimen and not interfere with the sides of the moat. The remaining inner post is then milled into the sharp needle, as described previously. In an alternative lift-out method, a protective platinum line is deposited over the region of interest and then deep trenches are milled on either side of this line. Lines are then cut through the remaining membrane along the bottom and sides. This method is similar to the standard TEM lift out method except that the thickness of the membrane is greater and several square cross section blanks may be cut and lifted out of the membrane with an in-situ micromanipulator. The larger thickness of the membrane (~5 μm) is chosen to prevent gallium implantation into the region of interest. Individual blanks are attached to a copper grid mounted on a copper stub with platinum deposits. The blank is milled into the sharp needle, as described previously. The time consuming initial rough cuts of both methods may be automated.

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[1] D.J. Larson et al., *Appl Phys Lett.*, **73**(8) (1998)1125.