Preparation of Multilayered Thin Film Atom Probe Specimens

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The ability to sample the atomic-scale structure and composition of nanostructured materials is of relevant importance as many data storage and semiconductor devices continually downscale their dimensions to the nanometer regime. A technique based on the method described by Martens *et al*. [1] was used to prepare atom probe specimens of metallic multilayers. The multilayers were prepared on 5 Ω-cm and 0.05 Ω-cm n-doped Si wafers. These wafers were photolithography patterned into a series of 5 μm x 5 μm, 7μm x 7μm, and 9μm x 9 μm islands. The patterned Si wafers were then Bosch-etched to a depth of 80μm at the Cornell Nanofabrication Facility. The Si wafers were then sputtered deposited with a Ti/Nb multilayered thin film. The Si posts with the deposited film were mechanically cleaved from the wafer and attached with silver epoxy to either a stainless steel or a tungsten needle by micromanipulation. The specimen was then milled concentrically with 30 keV Ga^+ ions using a FEI DB-235 Focus Ion Beam (FIB) instrument. The outer radius was set to equal the outer most dimension of the Si post and the inner radius was set for sequential milling at 0.7 μm (500pA), 0.3 μm (300 pA), and 0.06 μm (30-50 pA). Prior to the milling process, a sacrificial and protective Pt cap of 2.7μm x 2.7 μm x ~1 μm was deposited onto the surface of the film. This Pt cap serves as a barrier, minimizing ion damage and gallium implantation into the film. The milling process was terminated when the Pt-film interface was reached.

Atom probe tomography data suggests that the specimens of the multilayer films that were deposited on the 5 Ω-cm Si wafer underwent uncontrolled field evaporation particularly in the early stages of ion collection. This process generated increased background noise and significant high mass tails to the peaks in the mass spectrum, non-uniformity in the atom reconstruction of the individual layers in the multilayer and a propensity for specimen failure. In contrast, the specimens of the multilayer films that were deposited on the 0.05 Ω-cm Si wafers underwent more normal field evaporation and produced data typical of metallic specimens. The choice of stainless steel or tungsten needles appears to have little effect on the success of the specimen collection.

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[1] R. Martens et al. (2000) *Micros. Microanal.* **6** (suppl.) p. 522.