Site-Specific Specimen Preparation for LEAP

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The use of Focus Ion Beam (FIB) technology in the preparation of thin film transmission electron microscopy (TEM) specimens has been widely demonstrated. This same type of FIB technology, which can allow for site-specific specimen preparation, is proposed for chemical analysis using a Local Electrode Atom Probe (LEAP). The coupling of these two techniques offers a wide range of possible applications of atom probe tomography (APT). These applications include APT analysis of a specific grain boundary, precipitate interface, junctions in semiconductor devices, near surface regions and low volume fraction phases. This technique also reduces the number of steps required to prepare thin film specimens since the milling may be performed directly on a wafer. In addition, the preparation of brittle specimens should be more efficient due to the reduced amount of handling during machining steps.

A Ti-42% Al specimen has been used to demonstrate the potential of this technique. The Ti-42% Al specimen was water quenched into a super solid saturation of the ordered hexagonal phase, α_2. This specimen is extremely brittle and difficult to prepare with conventional electropolishing techniques. A standard 3-mm TEM disc was electrode discharged machined from the bulk specimen and subsequently mechanically ground to approximately 200 μm in thickness using 600-grit SiC. The TEM disc was then mounted on a standard Cu atom probe tube with silver epoxy. The top surface of the Cu tube was previously cut with a 2º angle to facilitate the alignment of the apex of the specimen with the aperture in the counter-electrode of the LEAP. The TEM disc was then placed in a Philips db-235 FIB. A specific site was chosen on the surface and a 3 μm x 3 μm x ~1 μm Pt mask was deposited on the surface at this position. The Pt mask is essential as it acts as a sacrificial and protective layer over the desired feature to prevent significant Ga implantation. A 10-20 μm diameter x 1.5 μm deep trench was milled (5 nA, 30 keV) around the point of interest leaving a conical protrusion. The diameter of the conical protrusion was sequentially ion milled from 3 μm (500 pA, 30 keV) to 0.7 μm (500 pA, 30 keV) to 0.3 μm (100-300 pA, 30 keV) to 0.06 μm (30-50 pA 30 keV). The spread of the gallium ions from the nominal inner diameter in the final milling stage was found to be effective in both controlling and accurately terminating the removal of the final portions of the Pt mask and also sculpting the apex of the specimen to a desired taper angle. The ease in the milling preparation should make it possible for the automation of this technique to allow for multiple tips to be prepared in a reasonably short period of time. The deposition of platinum markers was also found to be beneficial for locating the finished protrusion.

Research at the SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.