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The Utility of Xe-Plasma FIB for Preparing Aluminum Alloy Specimens for MEMS-based *In Situ* Double-Tilt Heating Experiments

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In situ heating and gas-reaction capabilities utilizing MEMS-based heater devices have become popular over the past decade, with applications primarily in the catalyst and nanoparticle research communities, because the preparation of specimens relies simply on depositing powders or liquid suspensions onto the devices (such as the "E-chips" provided by Protochips Inc. (Raleigh, NC)). Studies of specimens of "bulk" material specimens require, however, depositing electron-transparent lamellae on the order of tens of microns in size onto E-chips, and this process is best performed using focused-ion-beam (FIB) milling techniques, usually with *in situ* lift-out capabilities [1-3]. Our interests are to study precipitation processes in Al alloys, but using conventional Ga FIB-milling methods invariably leaves residual Ga back-deposited or implanted onto the surface of the thin lamellae, and Ga is especially detrimental for Al alloy studies [4]. As described here, we have recently begun testing the utility of Xe-plasma FIB methods for Al alloy specimen preparation for *in situ* heating, along with the use of a new double-tilt capability with the MEMS heater devices, necessary for precise foil alignments.

In order to minimize the FIB cutting process, we first prepare good foils of the Al alloy by electropolishing methods, and then locate appropriate grains (e.g. <110> and <001> zones) in the foil that are oriented within e.g. ±10° in both X and Y tilt directions. Figure 1a shows a grain of an AlCu7 alloy cut using a Ga FIB instrument (Hitachi NB-5000), with the Ga-ion beam at 40kV/1nA, affixed to an E-chip. The lift-out needle was attached to the lamella by electron-beam-deposited carbon to minimize Ga usage, but was cut with Ga from the lamella. The lamella was "tacked" onto the E-chip by again using e-beam-deposited carbon. However, even with these optimized procedures, the unavoidable deposition of Ga even in the area of the thin edge of interest in the heating experiment is clearly indicated by the energy-dispersive x-ray spectroscopy (EDS) spectrum from that area (Fig. 1b). In comparison, a lamella (Fig. 2a) was deposited onto an E-chip using a Xe-plasma FIB instrument (ThermoFisher Helios G4 PFIB) with the Xe beam at 30kV/4nA. The Xe beam was used not only for cutting out the thin lamella, but also for affixing the lift-out needle to the lamella using W deposition, cutting the needle from the lamella, and finally tacking the lamella onto the heater surface. Figures 2b and 2c show EDS spectra from the area of one of the W connection points (a "worst case" condition), and from the thin lamella area to be used for the heating experiment. The very low Xe peak in the tack area, and essentially non-existent Xe peak in the area of interest attest to the efficacy of using Xe-plasma FIB milling for this deposition process. In addition, Xe is inert with respect to reactions with Al (or any specimen), so no issues with any Xe associated with the specimen preparation are expected. The E-chip was mounted in a new Protochips double-tilt heater holder (Fig. 3a), with ±14° of tilt typically available in both X and Y tilt directions, to accommodate the tilts required for precision alignment of the desired zone axis (see inset). Figures 3b-d are an image series showing the effects of heating to a temperature of 225°C over ~1hr, on the coarsening behavior of Al_2Cu (θ') disk-shaped precipitates, which form on the Al matrix {100} planes, one habit of which is seen edge-on in this <110> Al zone-axis orientation. Studies of the initial stages of precipitation from fully solutionized foil specimens are planned, using this very promising method for making ideal specimens of Al alloys for *in situ* heating experiments.[5]

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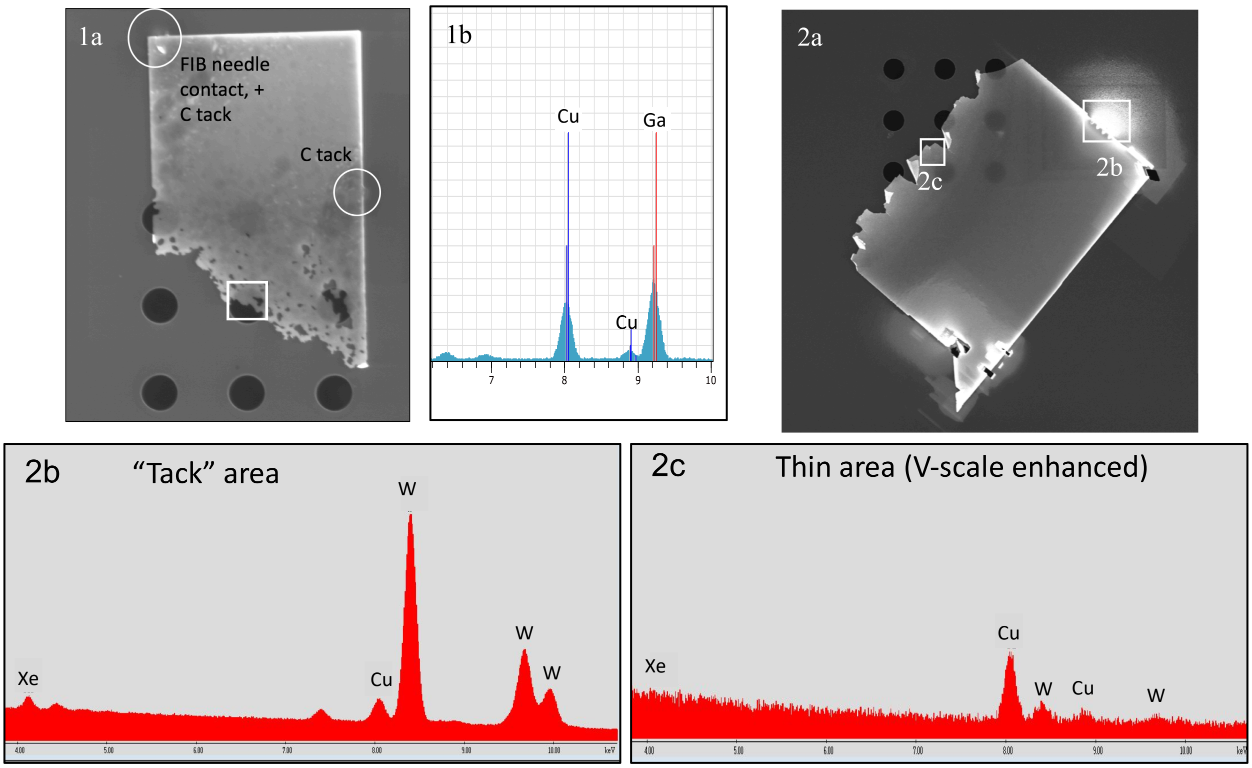

Figure 1. a) Ga-FIB-mounted Al alloy lamella, on Protochips E-chip. Inset square is area for EDS of 1b, which shows significant Ga contamination of the area of interest. The "holes" in both heaters are 8μm dia.
Figure 2. a) Xe barely detected in 2b tack area, the "worst case." b) No Xe detected in thin area 2c.

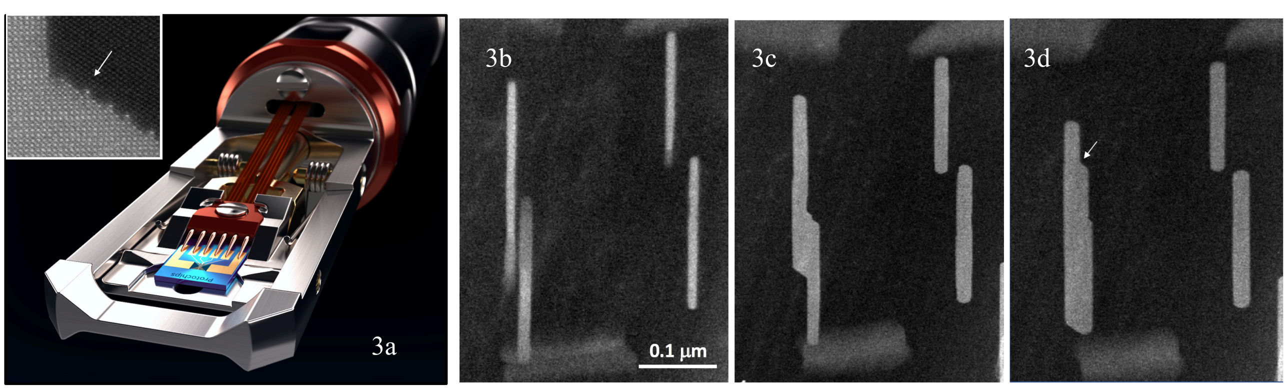

Figure 3. a) E-chip mounted in Protochips double-tilt heater holder; inset shows atomic structure of ledge of precipitate in 3d. b-d) Image sequence showing precipitate morphology changes at 225°C over ~1h.

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