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Expanding the Capability of Xe Plasma Focused Ion Beam Sample Preparation for Transmission Electron Microscopy

Suzy Vitale[1] and Joshua Sugar[2]

[1]Carnegie Institution for Science, Washington, District of Columbia, United States, [2]Sandia National Laboratories, Livermore, California, United States

Xe plasma focused ion beam (PFIB) has several advantages over Ga focused ion beam (FIB) that makes this capability very desirable for high resolution transmission electron microscope (HR-TEM) lamellae preparation: it has a faster milling rate [1], it is Ga-free which allows for investigation of Ga sensitive materials (e.g. Al and Al alloys) [2-4], and it minimizes amorphous damage of sample material because of its higher atomic number [5]. It is well established that a Ga liquid metal ion source (LMIS) FIB can produce high quality lamellae for HR-TEM and electron energy loss spectroscopy (EELS) [6]. It is less understood if the same can be said for the Xe PFIB with its larger ion size and larger beam currents. Beam profile, beam control, and material deposition are just a few ways in which Xe PFIB TEM sample preparation diverges from standard Ga FIB techniques. Though it has been demonstrated that it is possible to make TEM lamellae using only Xe PFIB [7-8], in this study we sought to determine whether a Xe PFIB could produce Ga FIB-quality lamellae for HR-TEM-EELS on a sample known to be sensitive to Ga contamination. We define this quality as a curtain-free, uniformly thin region of at least 5x5 um where t/λ is ≤ 1, and we directly compare Xe PFIB and Ga FIB-prepared samples.

A polycrystalline Al thin film was thermally evaporated on to a silicon wafer ([100] Si). Thin TEM lamellae were prepared using a ThermoFisher Helios G4 PFIB and an ThermoFisher Helios G3 Ga FIB. Compositional and thickness analyses of the samples were done using a ThermoFisher Themis Z (S)TEM operated at 300 kV and a Gatan 969 Quantum GIF. We developed milling strategies that take into account the larger currents and larger beam size of the Xe beam in the PFIB specimen. In particular, stage tilts and milling pattern placement were adjusted with Xe to encourage more milling with the beam tails. Several different tilts were investigated to identify the optimal configuration to fabricate a uniformly thin sample. All samples were finished using a low energy 5kV beam.

HR-STEM imaging easily resolved the [100] Si dumbbells with 76 pm resolution demonstrating the high quality of the specimens. Samples were curtain-free and thinned regions were 10x10 um (Figures 1, 2). TEM-EELS thickness maps showed a correlation between higher stage tilts and nonuniformity of sample thickness. These samples showed a “wedge” shape where the thinnest region ($t/\lambda = 0.55$ 50 nm) [9] was at the bottom of sample and the thickest region ($t/\lambda = 1.1$ 120 nm) was at the top (Figure 1). As expected, in samples where a smaller stage tilt was used, the samples were uniformly thin with a larger minimum thickness than the wedge ($t/\lambda = 1$ 110 nm) (Figure 2). It was difficult to produce a uniformly thin sample less than 100 nm due to the beam tails milling away the W deposition at which point further thinning was not possible. Despite the differences between Ga and Xe, we were able to use PFIB to produce Ga FIB-quality samples using only moderate adjustments to the typical in-situ Ga FIB TEM sample preparation methods.

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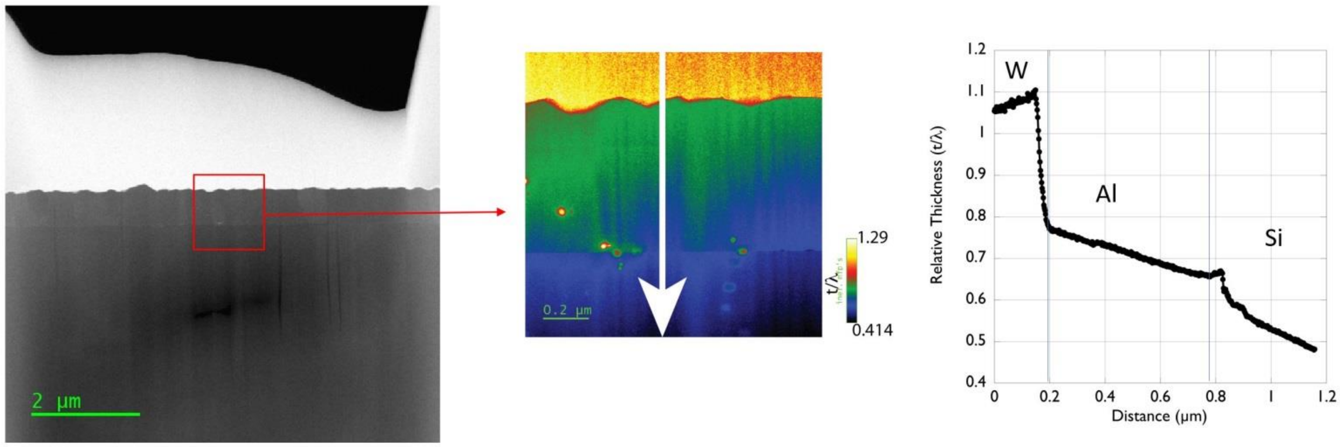

Figure 1. Figure 1. TEM-EELS thickness map shows wedge shaped thinned region with t/λ values ranging from 1.1 – 0.5).

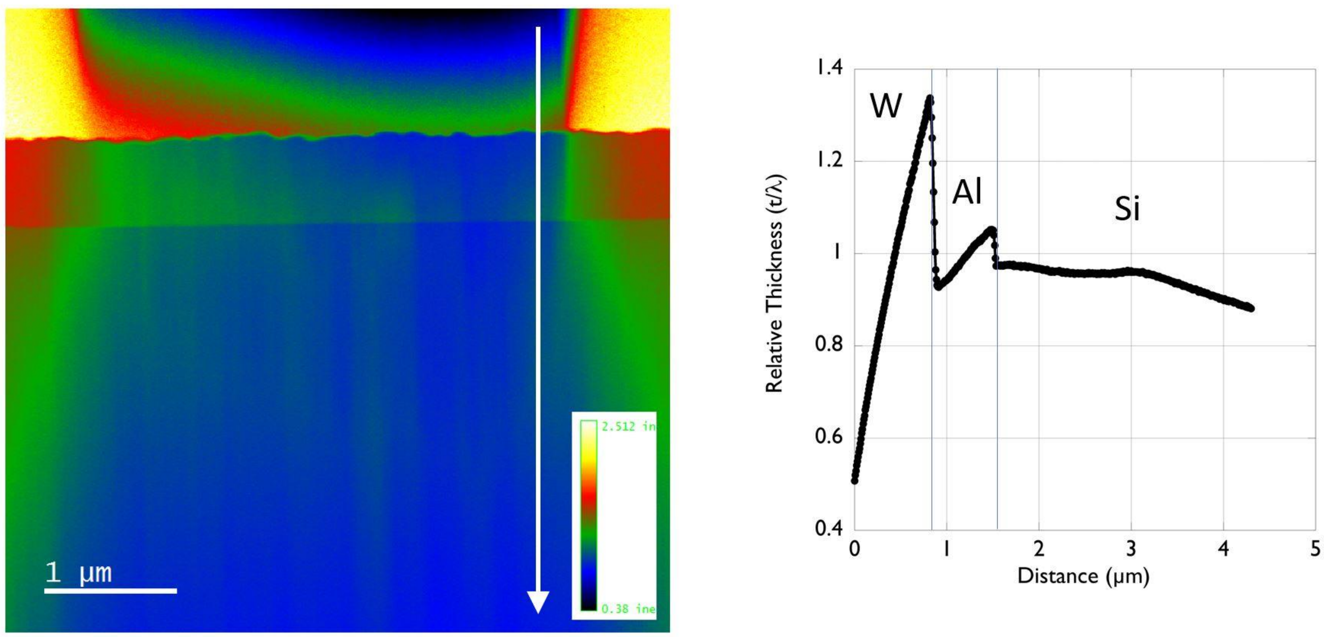

Figure 2. Figure 2. TEM-EELS thickness map shows uniformly thinned region with an average t/λ value of 1.

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