# Best Practices for Xe PFIB Preparation of Materials for Transmission Electron Microscopy

Suzy Vitale[1] and Joshua Sugar[2]

[1] Carnegie Institution for Science, Washington DC, 20015, USA
[2] Sandia National Laboratories, Livermore CA, 94550, USA

Over the past decade, there has been a concerted effort to develop gallium (Ga)-free focused ion beam (FIB) instruments for materials research. The helium ion microscope (HIM), neon ion microscope (NIM), and other similar inductively coupled plasma (ICP) multi-ion source focused ion beams such as oxygen (O) and argon (Ar) have been explored, though none have shown to be effective for the preparation of samples for transmission electron microscopy (TEM). Xenon (Xe) plasma focused ion beam (PFIB), thanks to its heavier ion mass (and thus high material sputter yield) and chemical inertness, has become the go-to instrument for large length scale (>100 µm) crossing sectioning and FIB-SEM tomography. But can the Xe PFIB fabricate TEM samples appropriate for nanoscale microstructural characterization?

Xe PFIB has its own set of challenges relative to Ga FIB. Xe PFIB would not typically be the go-to tool for nanoscale milling applications like TEM sample preparation because the ICP Xe PFIB will produce a beam with a larger diameter than the liquid metal ion source (LMIS) Ga FIB in the range of currents typically used for TEM prep. A beam with a larger diameter can be more difficult to use in smaller scale (< 500 nm) applications. The Xe PFIB also has a greater range of current density across the width of the beam. The outer-most edges of the ion beam, called beam tails, are more prominent in the Xe PFIB compared to the Ga FIB whose beam tails are virtually non-existent in most modern FIB instruments. The Xe PFIB beam tails add complexity to finer scale milling applications such as thinning a TEM sample to electron transparency.

In this talk, we will demonstrate that it is possible to create Ga-quality TEM samples using Xe PFIB. This quality is defined as curtain-free and uniformly thin over any 25 $\mu m^2$ area where the relative thickness ($t/\lambda$) measured with electron energy loss spectroscopy (EELS) is less than or equal to 1. We will identify best-practices and ideal parameters settings (including deposition energy (kV), deposition height, ion beam incident milling angle, and beam placement on the sample) to show that it is possible to fabricate two types of Xe PFIB-made samples: 1) a curtain-free uniformly thin electron transparent area where $t/\lambda \approx 0.9$ over 38 $\mu m^2$, and 2) a curtain-free non-uniformly thin electron transparent area where $t/\lambda$ ranges from 0.3 to 1.1 over 48 $\mu m^2$. Both sample types exhibit most, but not all, aspects of Ga-quality. Because of Xe PFIB's relatively large beam size and wide beam tails, a balance must be struck between ultimate sample thinness and the overall size of the electron transparent area.