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Carbon Capping for Specimen Preparation of Atom Probe Samples with Features of Interest Near the Surface

Edwin Supple[1*], Chomani Gaspe[2], Kevin J. Dwyer[2], Robert Butera[2], Christopher J. K. Richardson[2], and Brian Gorman[1]

[1.] Colorado School of Mines, Golden, CO, United States of America

[2.] Laboratory for Physical Sciences, College Park, MD, United States

* Corresponding author: esupple@mines.edu

Site specific sample preparation of Atom Probe Tomography (APT) specimens is possible using Focused Ion Beam (FIB), but standard techniques make accessing regions of interest near the bulk sample surface challenging. Conventionally, atom probe specimen liftouts are protected with material deposited by Gas Injection System (GIS) in the FIB or with material deposited ex situ before liftout, frequently platinum but also gold, cobalt, iron, and other materials [1]. Once lifted out, the GIS deposition protects the region of interest during specimen sharpening but must be fully removed to yield a specimen that can be analyzed by APT. It has been theorized that specimen failures are likely due to the high porosity and carbon content of the GIS Pt as well as the possible evaporation field mismatch between the sample and the Pt GIS deposit. Other protective materials with higher density and better matched evaporation fields (i.e. sputtered or evaporated Ni on Si) can be left on the tip during specimen preparation. These coatings can still allow the opportunity for stochastic tip fracture during the experiment due to random chance or poor bonding between the sample and the applied material. Atom probe specimens are routinely plasma cleaned before analysis regardless of preparation technique to remove hydrocarbons and improve specimen yield [2].

In this work, we demonstrate a method wherein a Sharpie® is used to apply a carbon coating above the region of interest. FIB specimen preparation including liftout and sharpening is demonstrated to be compatible with the C coating. Plasma cleaning the specimens to remove any carbon remaining on the end of the specimens leaves clean surfaces that include the top surface of the bulk sample but with minimal additional material above to evaporate during the atom probe experiment (Figure 1). This technique was applied on germanium quantum wells located 20 nm beneath the sample surface in silicon-germanium as well as on boron delta doped layers in silicon located 20 nm beneath the surface. Carbon capping was applied using a black Sharpie® fine tipped felt pen (Newell Office Brands) and allowed to dry in atmosphere for 5 minutes. One spot deposits roughly a micron of material. Specimens were prepared following established procedures [3] on an FEI Helios Nanolab 600i DualBeam FIB. Typically, 100-300 nm of carbon remained on the tip. Transmission Electron Microscopy (TEM) images were recorded of each tip, then the attached plasma cleaner on the LEAP 4000X Si was used to destroy the carbon capping. 30-60 minutes of plasma cleaning were sufficient to remove the carbon, dependent on the tip diameter (Figure 1).

The process yielded a high rate of successful experiments per tip fabricated (>80%). Specimen diameters at the top sample surface were up to 140 nm, providing a wide field of view with minimal field evaporation from the specimen required before reaching the ROI. Experiments using similar diameter specimens with GIS or sputtered capping material remaining would need to collect many millions of ions of capping material before reaching the sample itself.

The planar features of the sample surface as well as TEM images were used to determine layer depth and thickness and constrain the atom probe reconstruction (Figure 2). Amount of material evaporated during experiment setup is often an unknown when using TEM pre- and post- experiment images to shape the reconstruction. The sample surface provides an evident start to the experiment.

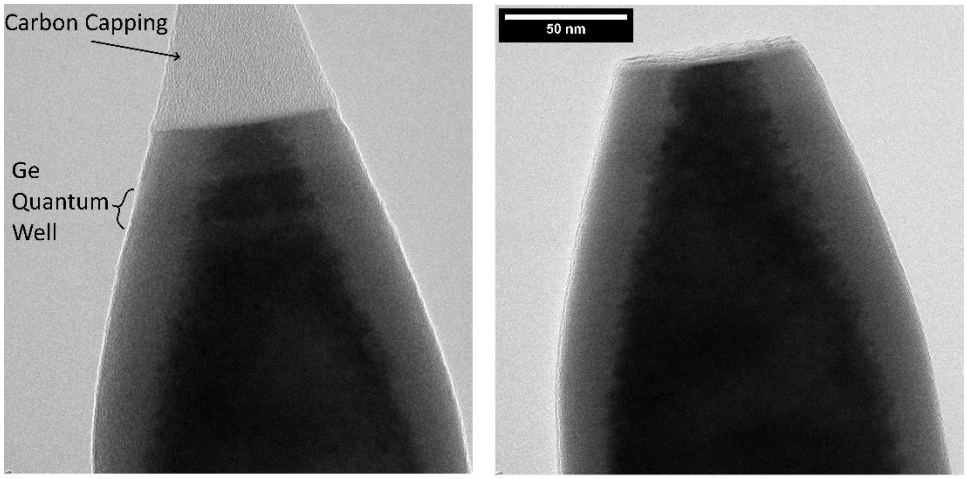

Figure 1. Atom probe tomography specimen of germanium quantum well surrounded by SiGe, showing carbon capping material (left) and specimen after 1 hour plasma cleaning showing removed carbon with exposed sample surface at specimen tip (right).

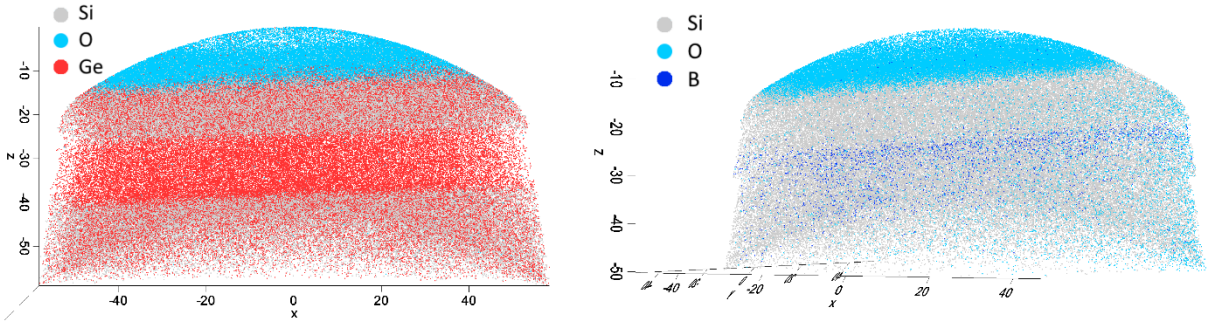

Figure 2. Atom probe tomography reconstructions showing germanium (red) quantum well in SiGe (left) and B (dark blue) delta layer embedded in Si (silver) (right). Both specimens contain the sample surface with minimal field evaporation required to expose the surface. Both the surface and the ROI layers are flat, and layer width and spacing match TEM images in the reconstruction. Oxygen (light blue) from the plasma cleaning process is detected from the surface and fills the reconstruction volume at the tip of the specimen.

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