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A Method for Plan-View FIB Liftout of Near Surface Defects with Minimal Beam-Induced Damage

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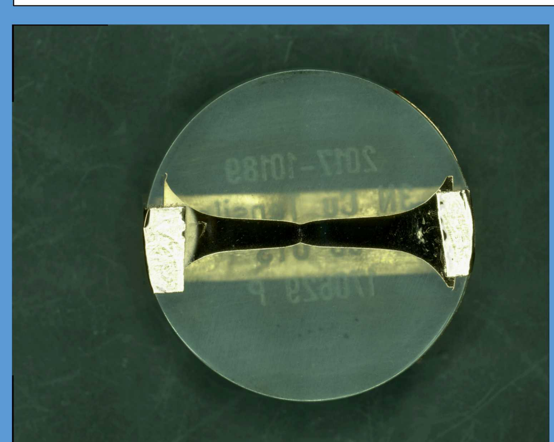
Introduction

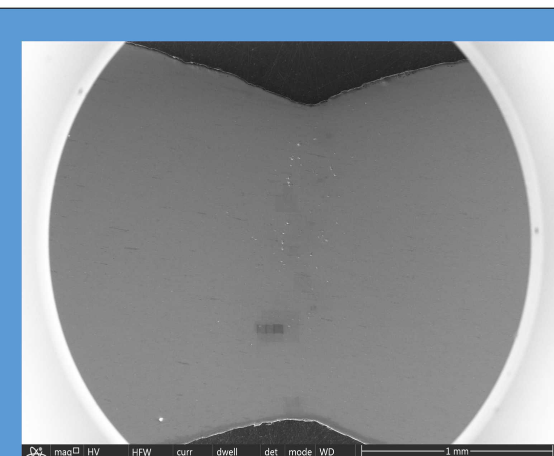
Focused Ion Beam (FIB) is a widely used technique in preparing traditional lamella type Transmission Electron Microscopy (TEM) samples with a cross sectional geometry [1]. However, additional challenges exist when preparing plan-view (i.e., with TEM sample parallel with surface) specimens of near surface, fine-scale features, while still ensuring minimal damage is caused by the ion beam. Among these potential challenges are, maintaining the area of interest in the center of the liftout, ensuring that the near surface region remains in the thinned region of the lamella, and working with the plan-view geometry that is quite different from the normal cross-section geometry. Presently there are several plan-view TEM preparation techniques described in the literature, for example reference [2]. Here, we investigate how to minimize Ga-induced beam damage and prepare a site-specific plan-view specimen of a near-surface feature (<50 nm from the surface).

The sample chosen was a Cu tensile specimen strained to the point of void nucleation in the center of the necked gauge region. TEM analysis to understand the dislocation structure around incipient voids was desired, so a plan-view specimen was prepared with FIB/SEM from the microstructure around one such void.

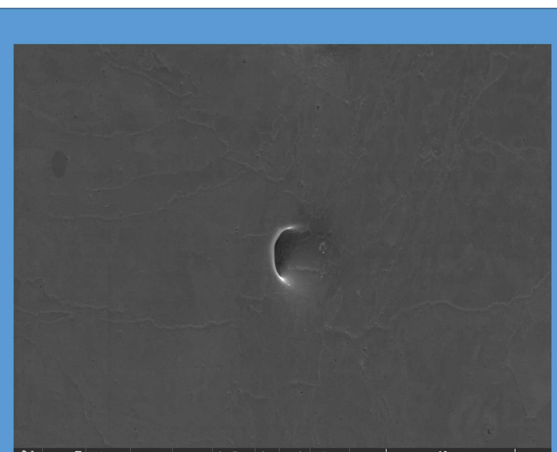


0.5 inch

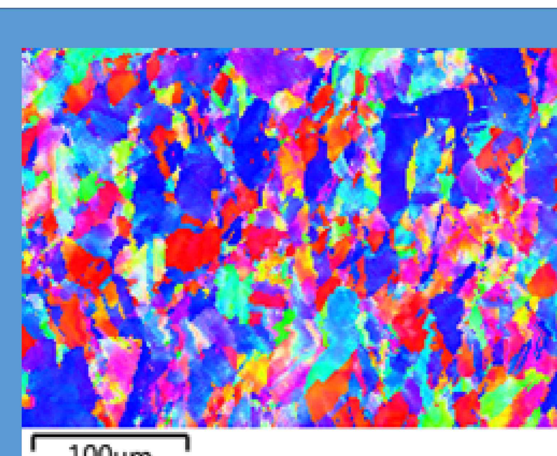
Optical micrograph showing overview of entire tensile specimen.



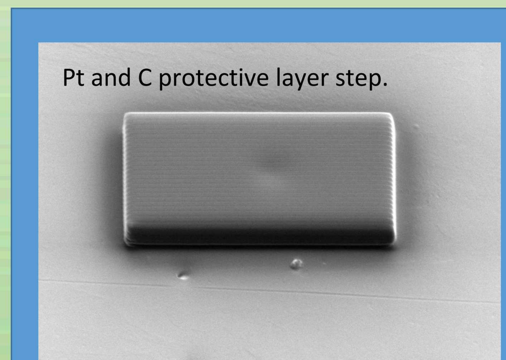
SEM image of necked region showing the presence of voids at the surface.



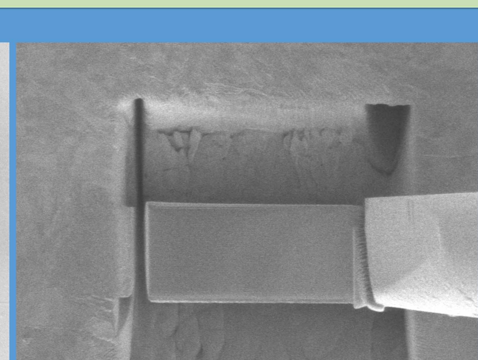
SEM image of surface void selected for TEM analysis.



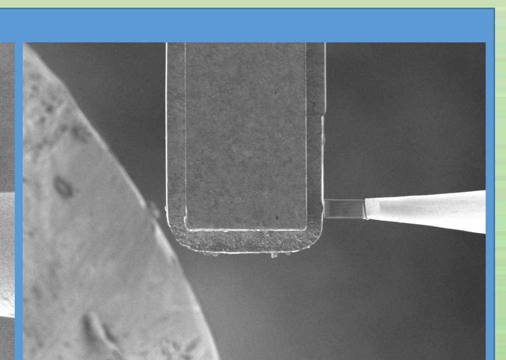
EBSD analysis of region surrounding void to show large scale crystallographic information.



SEM image of Pt (500 nm) and C (1500 nm) pad covering the void and region of interest.

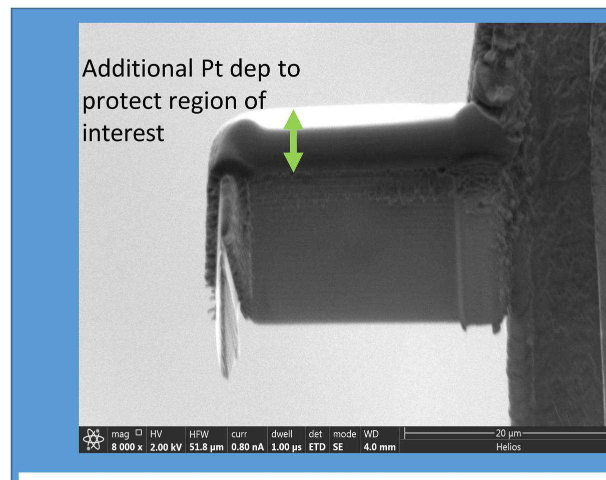


Sample is cut at 60° to surface to create a equilateral triangle shaped liftout.

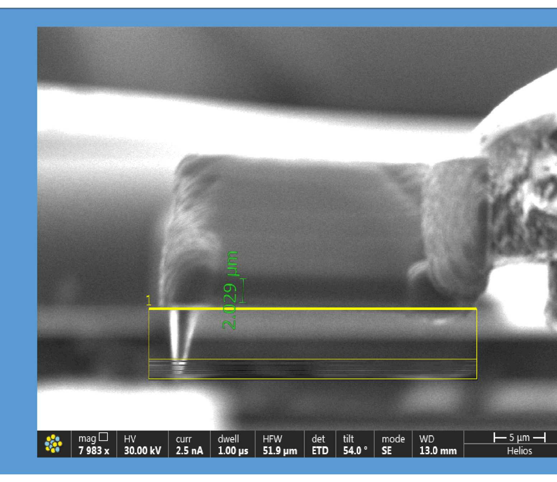


Sample is mounted on a horizontal grid so that the region of interest is parallel with the grid surface.

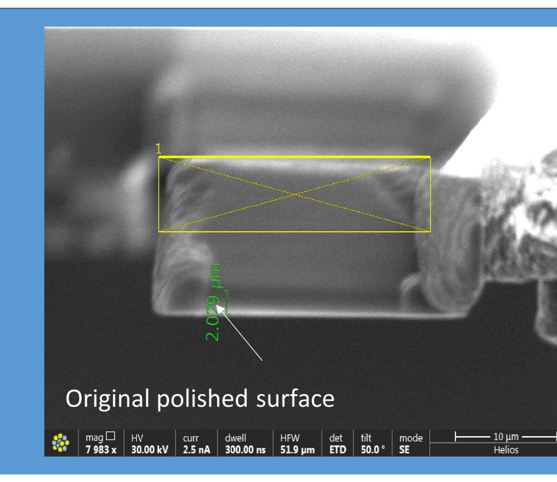
The grid is removed from microscope vacuum and rotated 90° to the vertical position. An additional Pt layer is deposited on the top surface in this orientation to protect the sample during thinning. The region of interest must be approached from the backside in order to ensure the area of interest is not milled through in the final thinning steps.



E-beam side-view of sample after second deposition step.

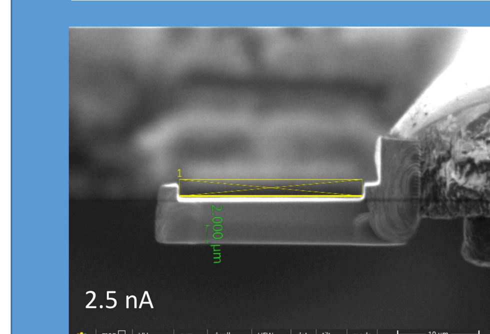


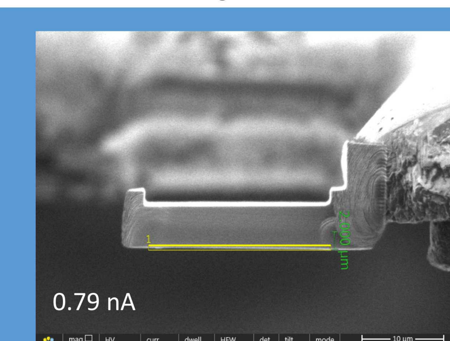
I-beam top-down image of specimen with box showing first cut (2.5 nA) to clean up surface. Green marker shows original Pt location from protective layer step.

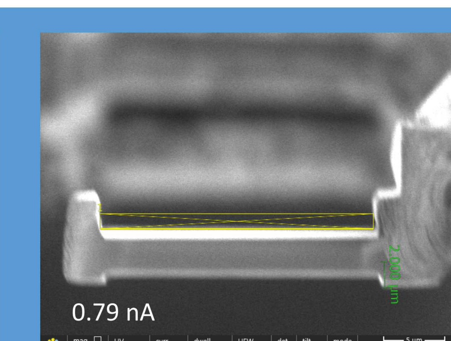


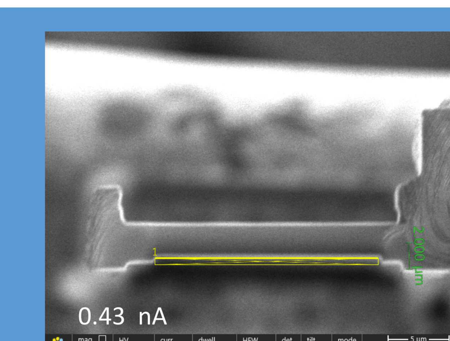
I-beam top-down image of specimen with box showing back side cut (2.5 nA) to approach surface of interest. Green marker shows original Pt location from protective layer step.

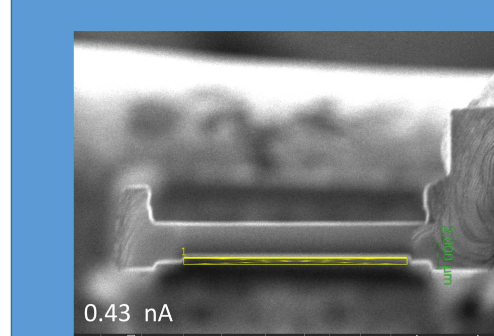
Using the green marker to keep track of the position of the original surface, it was possible to make a series of cuts that approach the surface region of interest. The original Pt protective layer is visible in the top down images, and provides a reference to ensure that the surface of interest is not milled away. The ion images below show the slow approaching of the region of interest with alternating front and back side cuts.

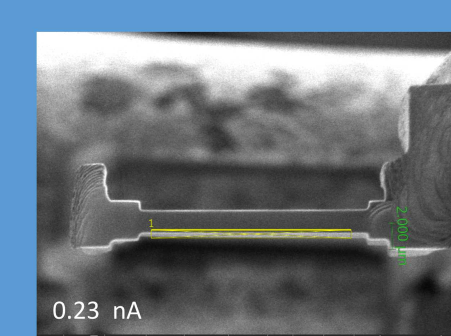


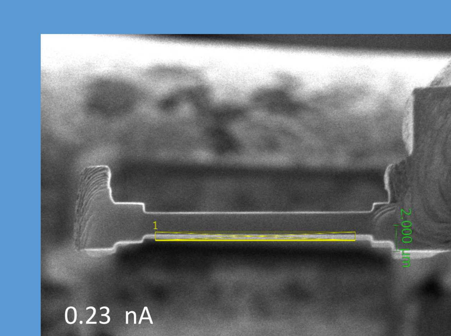


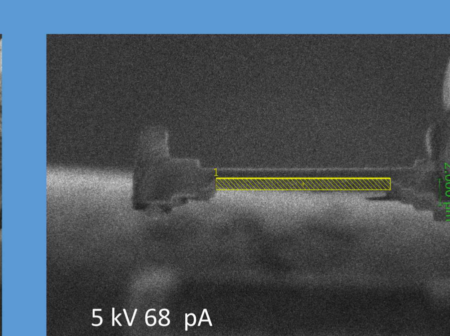




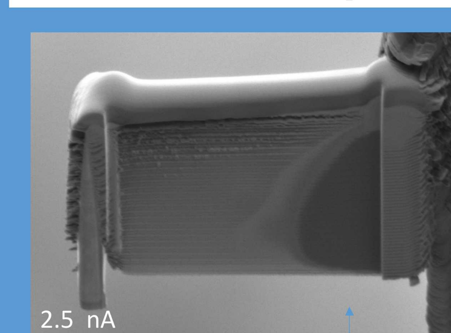




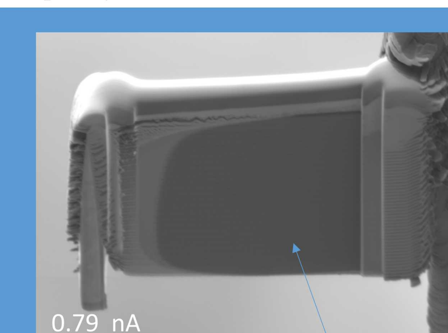




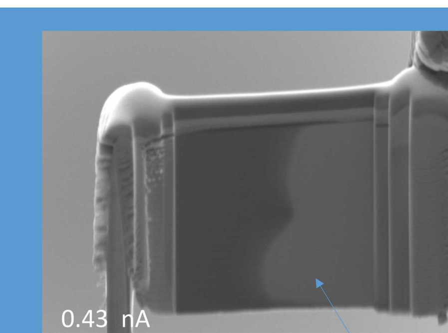
The side-view SEM images below show the progression of the sample after the front-side and back-side cuts. The endpoint can be determined by tracking when the original protective layer is completely removed. As material is removed, first the protective C layer is visible, then the Pt is visible when the C is completely removed, and then the Cu sample is visible when the Pt is completely removed.



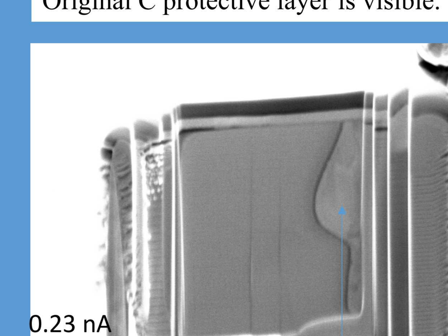
Original C protective layer is visible.



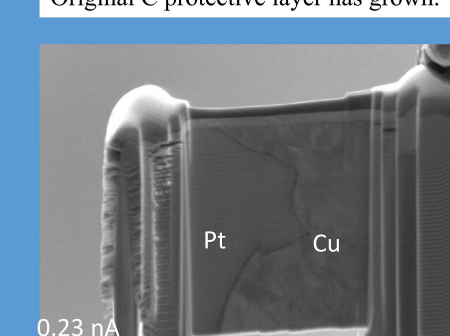
Original C protective layer has grown.



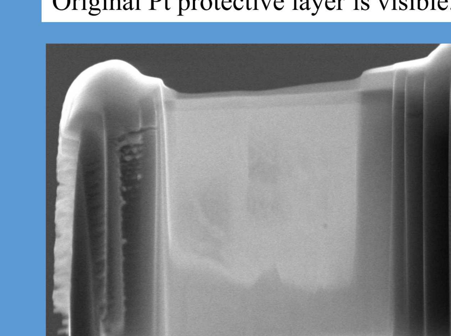
Original Pt protective layer is visible.



Cu sample is visible.

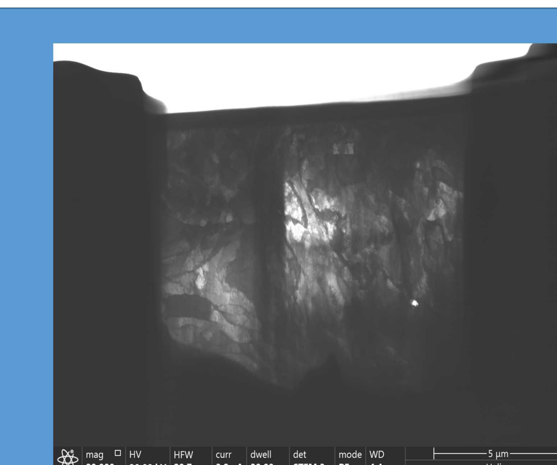


More Cu sample is visible.

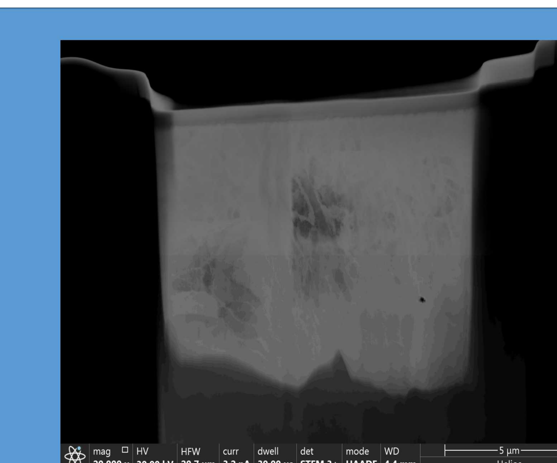


After 5kV 68 pA final polish.

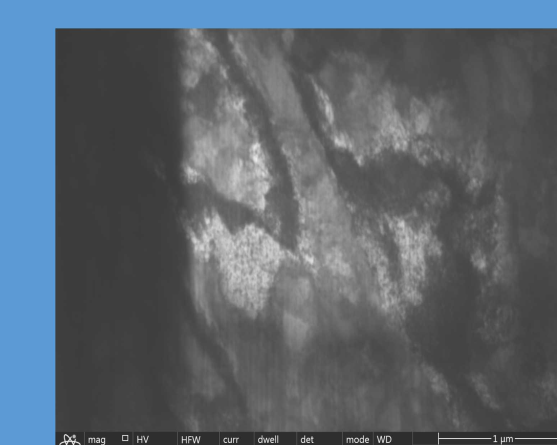
The sample was then imaged with the STEM detector in the FIB in order to determine if the sample was thin enough for TEM. The grain structure and individual dislocations are clearly visible in the image, which confirms the sample is ready for TEM analysis.



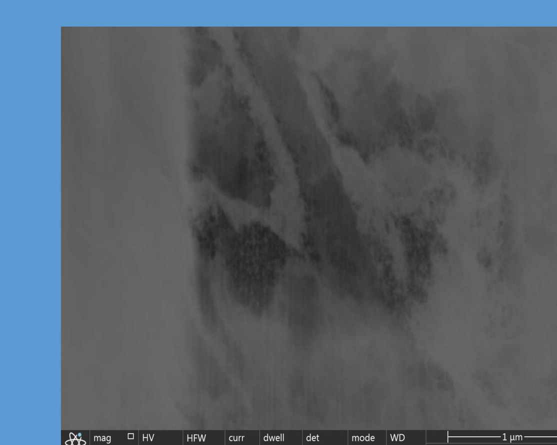
BF STEM image (30 kV) of sample after final polishing step.



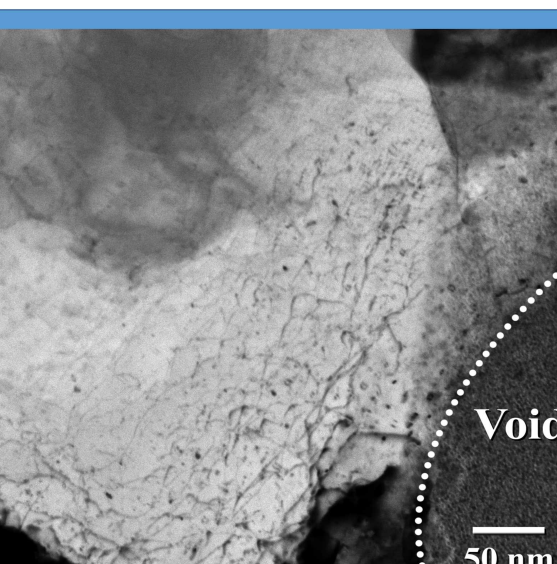
HAADF image (30 kV) of sample after final polishing step.



Higher magnification BF image STEM (30kV) of sample after final polishing step.



Higher magnification HAADFSTEM image (30kV) of sample after final polishing step



A 300 kV STEM image of sample after final polishing step showing a high-quality sample has been prepared. The original void is contained within the sample, and individual dislocation lines are visible in the image.

Conclusion

A high-quality result is highly dependent on stopping the thinning process as soon as the protective layer has been removed such that low-kV polishing can still occur on the near-surface feature and remove any high-kV beam damage. This technique provides high-quality TEM results and can be used to study near-surface defects in a plan-view orientation. This technique can also be broadly applied to other material and defect types.

[1] L.A. Giannuzzi, F.A. Stevie, Micron volume 30 issue 3 (1999), p. 197-204.
[2] Chen Li, Gerlinde Habler, Lisa C. Baldwin and Rainer Abart, Ultramicroscopy volume 184 part A (2018), p. 310-317.

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