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On-Probe Thinning of Samples for Bulk or STEM Type EDS Analysis

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Within an SEM, EDS analysis of surface or subsurface features exposed by manual polishing can be performed. To improve time to data and accuracy, it can be advantageous to use a FIB/SEM system to mill *in situ* sections for subsurface analysis. However, the EDS signal from such a FIB cross-section can be weak due to the compromised geometry resulting from creating an imaging plane inside a hole. By using a lift-out probe on a FIB/SEM system, samples can be lifted out and directly EDS analyzed while on the probe. When desired, thinning the sample on the probe can also be done. The sample can be optimized for two different geometries for EDS analysis: planar and cross-sectional. The on-tip analysis of both these geometries is achieved by lifting the sample out after it has been positioned at particular angles of stage tilt and rotation and then rotating the probe. The example presented here used a probe mounted at a 50° port elevation over the FIB/SEM tilt axis and an ion beam at 52° tilt to the electron beam. Recipes have also been developed for other configurations of port and ion beam positions.

To simplify lift-out of a rotated sample, it is best to use the Total Release™ method for lift-out [1]. Otherwise, if the sample is still attached to the bulk after the probe is welded to the sample, it can be difficult to make the final cut to release the sample at this off-axis angle. It can be done, but takes considerably more time and the user needs to be aware and carefully plan the setup.

For subsurface planar analysis, the analysis area is completely cut free by the Total Release™ method. Then the sample is rotated 47.2° counter-clockwise, after which the probe is welded to the sample and the sample lifted out. A probe rotation of 66.2° clockwise will now orient the sample for ion beam thinning. Layers now can be removed from the sample while viewing the sample with the electron beam [Fig. 1] until the region of interest is found. For EDS analysis, the sample can be rotated 66.2° counter-clockwise back to the optimum position for EDS analysis.

If the sample is thinned to TEM sample thickness, it can be moved over to a point on the stage with low background. To achieve a low background platform, a standard SEM stub was drilled with a high aspect ratio hole and a beryllium grid was carbon pasted halfway over the hole [Fig 2]. Positioning the probe against the grid produces a very stable platform for sample thinning, high resolution imaging or analysis [Fig. 3].

For cross-sectional samples, the sample is lifted out at a 52° stage tilt with the stage rotated 17.11° clockwise. After lifting the sample out, the sample can be milled directly. When analyzing the sample, the probe should be rotated 66° counter-clockwise to make the sample normal to the e-beam and ready for EDS.

References

[1] Patent, "Total release method for sample extraction from a charged-particle instrument", May 2003
[2] This work was supported by IBM Research, Almaden through system time and discussions with Phil Rice, Teya Topuria and Eugene Delenia. Additionally Kurt Langworthy at University of Oregon's CAMCOR facility helped with system time.

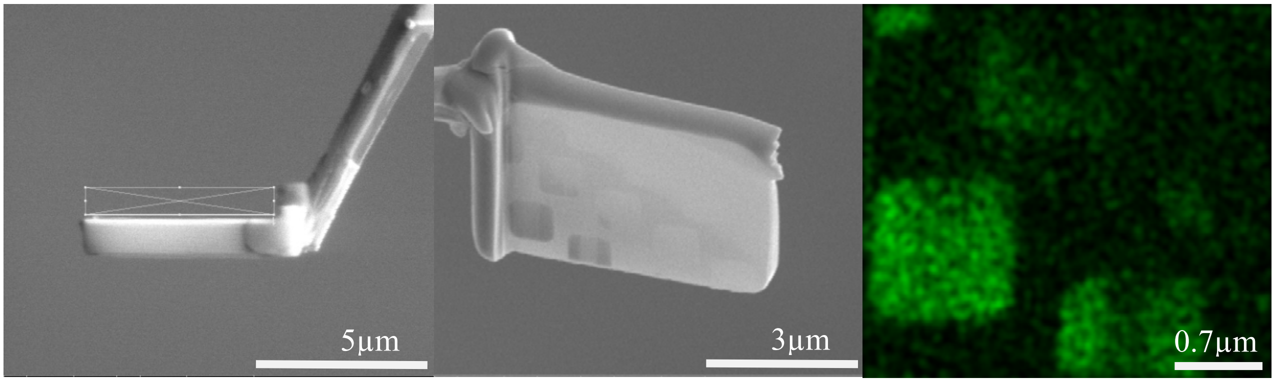

Figure. 1. The ion beam is thinning the surface of the sample extracted from the bulk (left) and the electron beam is imaging the milled face (middle). A W map was acquired with EDS (right).

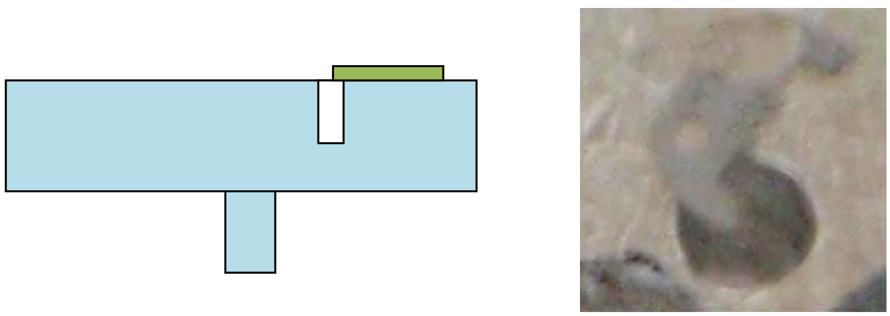

Figure. 2. The drawing shows the side-view configuration of a beryllium grid placed partially over a hole in a sample stub to create a low-background analysis area for EDS. The image on the right shows the top-down view of the grid over the hole so that the probe can be positioned against the grid while over the hole for higher resolution imaging and analysis with low EDS background.

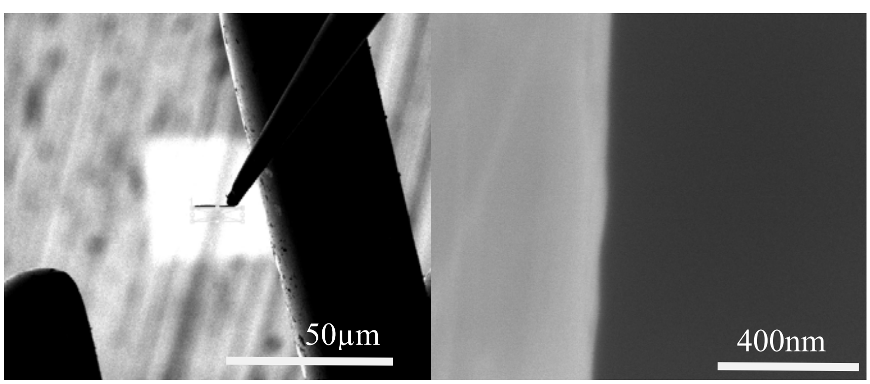

Figure. 3. The image on the left shows the probe positioned against a grid for support during final TEM sample thinning. The image on the right shows that there is virtually no vibration at 400kX magnification or 1.28 µm Horizontal Field Width.

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