PRE-THINNING FOR FIB TEM SAMPLE PREPARATION USING THE SMALL ANGLE CLEAVAGE TECHNIQUE

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Introduction

The Small Angle Cleavage Technique (SACT) can produce samples from brittle and cleavable materials very quickly. It is possible for a skilled person to produce about 10 XTEM specimens in an hour. However, there are materials-related limitations to the technique. These include 1) the ductile/brittle nature of the substrate, i.e. the fracture mechanics of the materials, 2) the strength of the interfaces between multi-layers, 3) the thickness of any ductile layers, and 4) the strength of the layer materials. In most of the cases where SACT fails, the sample is near electron transparency at the apex of the sample. Where there is a weak interface as in some multi-layer systems, or where the material itself is weak, some of the film near the substrate is sufficiently thin, but there is a jog in the film stack and further back from the apex it is not transparent. If such a SACT sample is viewed from the surface in an SEM, this apparent delaminating of the top layers is evident and usually within 1 µm of the apex.

The purpose of this paper is to show that the geometry of a SACT-prepared sample is well suited for applying FIB milling for further thinning of the sample to electron transparency. In addition, the FIB can be easily applied to SACT specimens that fail to produce usable samples for the reasons cited above.

Preparation Options Using SACT/FIB

➢ Method I: Post-FIB Grid Mounted

- The SACT samples are prepared by the Small Angle Cleavage Technique and then several are mounted on a flat, SEM specimen stub.
- Advantages: multiple samples can be cut without specimen exchange, samples are mounted flat
- Disadvantages: chances are high of losing a sample during mounting on the grid.

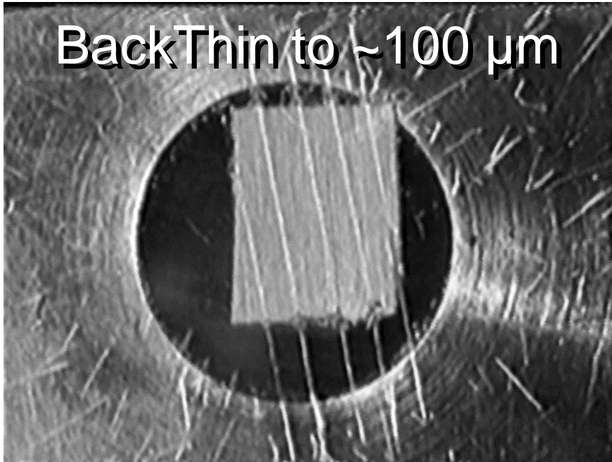
➢ Method II: Pre-FIB Grid Mounted

- The SACT sample are mounted on the grids and held vertically in a holder in such a way that the Pt source needle does not touch the grid or the sample when it is inserted.
- Two methods are available for preparing the grids, folding and cutting.
- Advantages: Less likely to lose a sample, ready to put in TEM.
- Disadvantages: Sample surface is difficult to get perpendicular to beam, the cut grids are less likely to protect the sample.

Types of Samples Using SACT/FIB

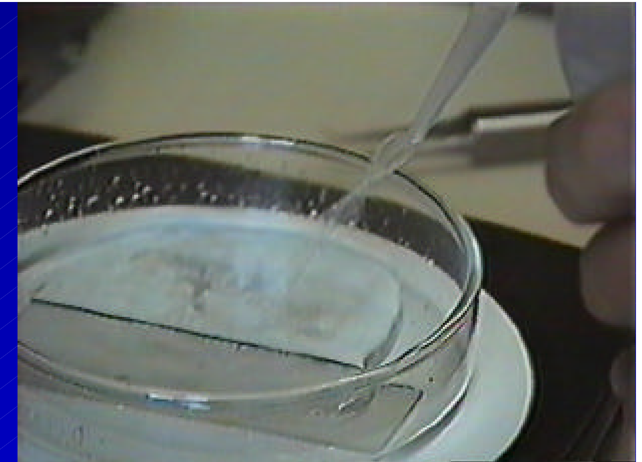
- **Apex**
  - Quick, with simple cuts
  - Useful for SACT samples that "almost work."
- **Pre-Thinned –Near Apex**
  - This is useful when there is a distribution of desired areas near the apex.
  - Also, useful when multiple areas are desired on the same sample.
  - AutoTEM mode available.
- **Pluck-Out**
  - There is plenty of area away from the apex to gather samples using the pluck-out method.
  - AutoTEM mode available.

Note: All of the above techniques work with samples either Pre- or Post-FIB Grid Mounted.



The scribe lines should be relatively deep into the sample, and ideally, they should not be visible from the good side when sample is removed.

Remove the samples by heating or soaking in acetone. Use a brush to remove the delicate samples. A 2nd rinse in fresh acetone is recommended.

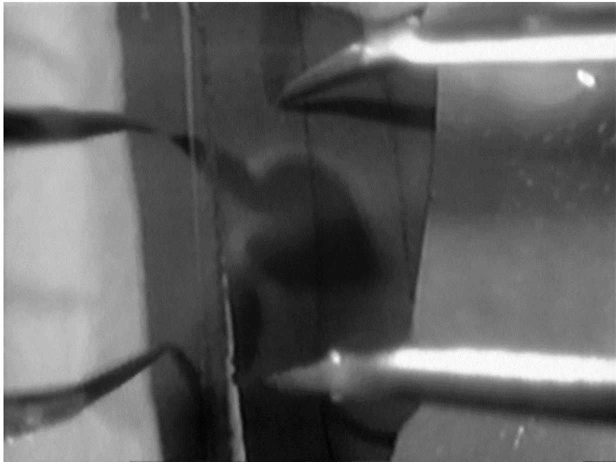


Permanently mount a glass slide in a Petri dish. Fill the dish with distilled water for each new sample. Thoroughly clean dish between uses.

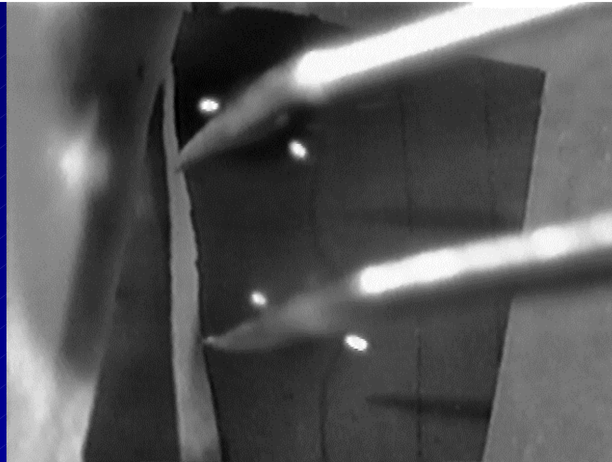
Carefully place sample onto the glass slide with scribe marks facing up. Angle the sample into the water so that it does not float.

Alternative: Use no water. Some samples may be water sensitive. SACT can be performed without liquids. The water prevents the sample from flying away during the fracturing and cleavage steps. The surface tension of the water also helps clean any debris and "nano-dust" from the surface when the sample and the tweezers are withdrawn from the water. If no water is used, the samples will be found somewhere in the Petri dish. In either case, after fracturing the sample along the scribe lines (2 steps away), flip them over (face up) for cleaving.

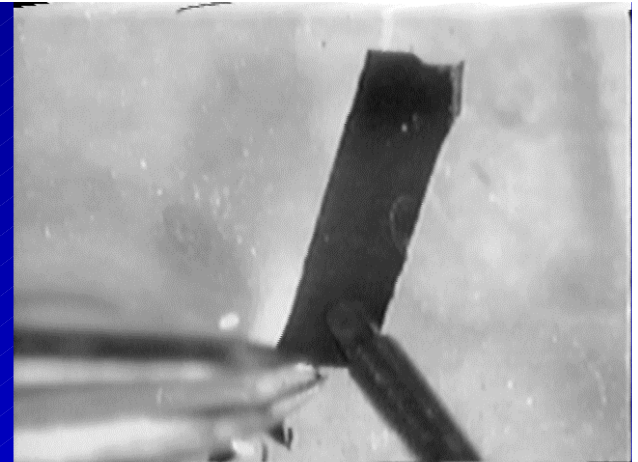
For the no-water approach, after fracturing along scribe lines (next step), place face up on the sticky part of a small Post-it™ note taped to a block.



Carefully align the scribe marks along the edge of the glass slide. Gently press sample down. The tweezers should be pressing evenly on either side of line.

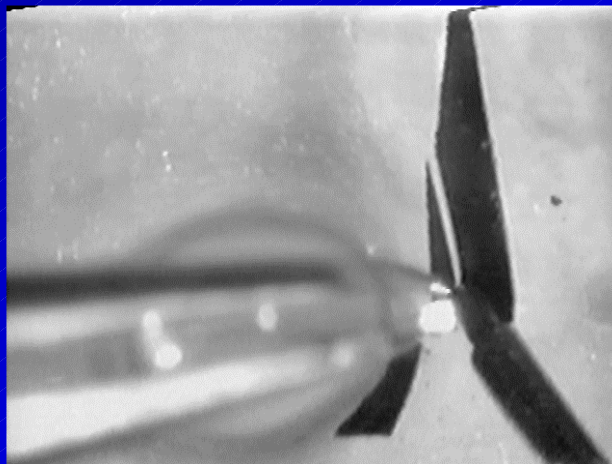


Carefully pick up the resulting piece, flip it over, and place it on the glass slide. Be careful of the edges of the sample. Repeat for all of the strips.



Gently brace the bottom edge of the sample and start a scribe line along normal cleavage direction. It is a trial and error process where to begin line.

By gently applying pressure, a cleavage line will propagate up and intersect the edge at a 12-15° angle.

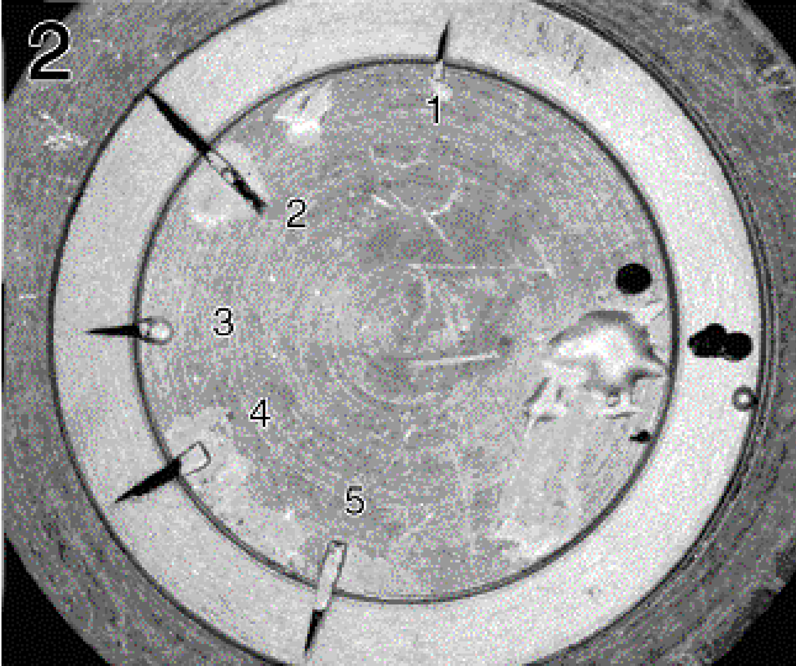


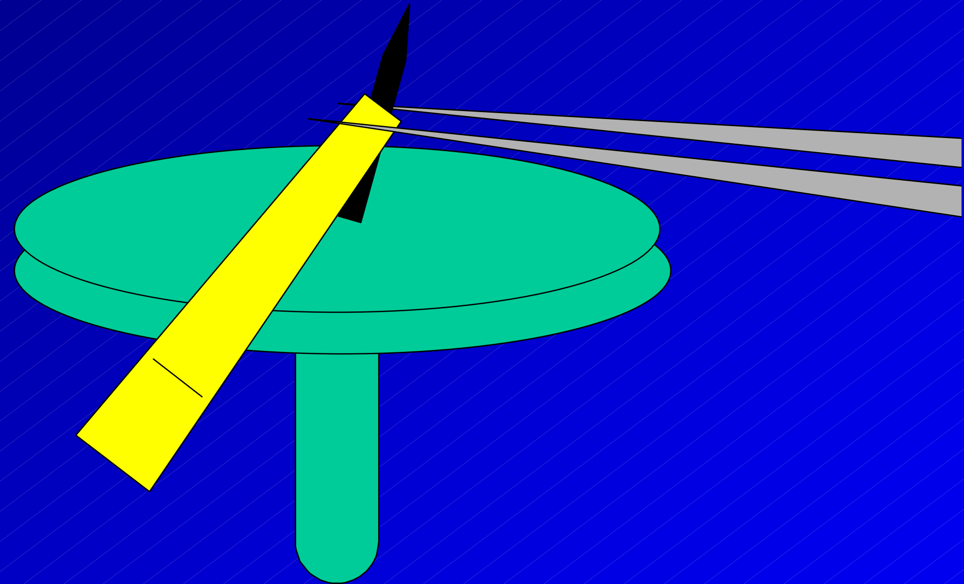
During the cleaving, a gentle sideways pressure is also exerted with the diamond scribe to push the sample away and protect the apex.

Notes: (i) Examine the fracture edge of strip before cleaving. If it is "jaggy" or not smooth, find and area that is. Try to intersect the cleavage line there.

(ii) The critical part of SACT is the determination of which cleaved samples are suitably thin, not all of them will be. Use the highest possible magnification of the microscope to image the apex of the cleaved sample. If the apex is sharp to the point where it cannot be seen, the sample is probably good. If a nice thin feathered edge can also be seen running up along the thickness to that apex, examine that sample in the TEM first.

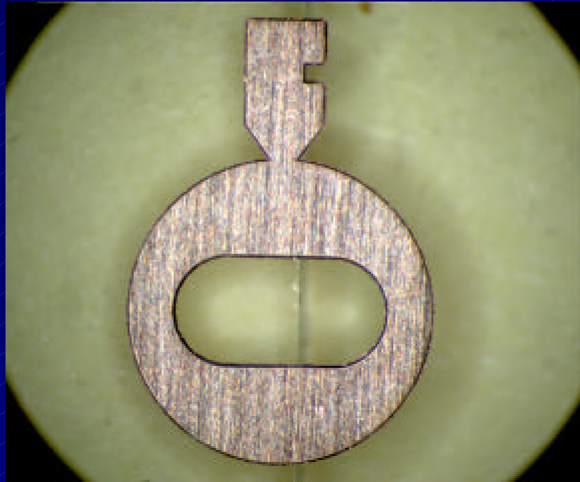
Mounting and Removing SACT/FIB Samples from an SEM Stub, Post-FIB Method



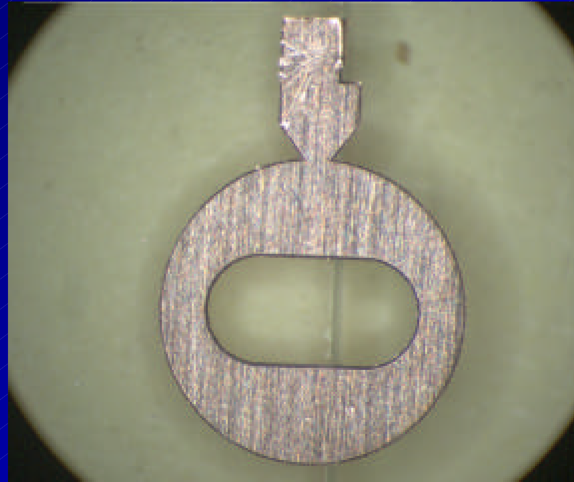


- Select stubs that are flat on the surface and do not have rounded edges.
- A step milled around the rim of the stub can protect the samples that project radially outward.
- Keep the epoxy that holds down the sample to the stub to a minimum. This makes it easier to remove the samples.
- A spot of epoxy is used as an index and to check when epoxy is cured.
- Use a Cut-down Post-it® and a sharp pair of tweezers to break the SACT/FIB sample from the SEM stub.
- Use a normally prepared SACT grid to mount the sample.

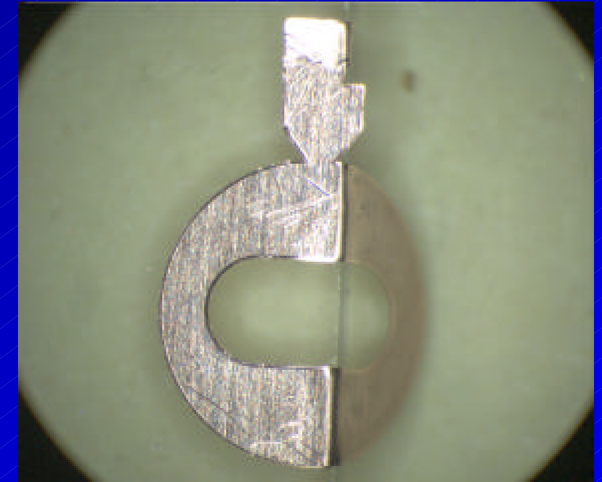
Bending SACT Grids for Post-FIB Method



Start with 2x1 Veco tabbed grids & align on the edge of a glass slide.



Bend the vertical tab down.



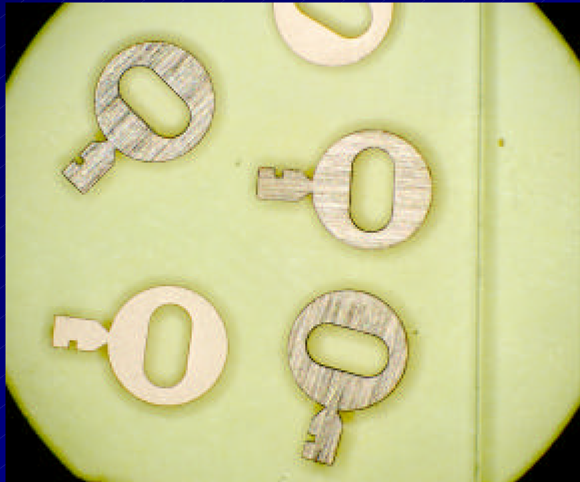
Bend the body of the grid on same line.

Continue fold and iron the body flat.

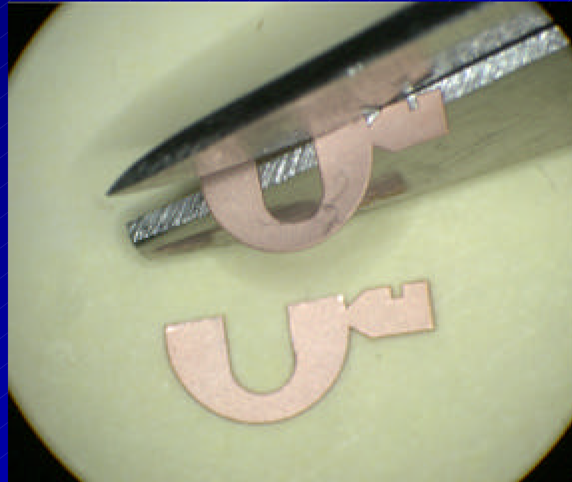
Align and bend the tab.

Ready for mounting. Use a dab of epoxy on grid to stabilize it.

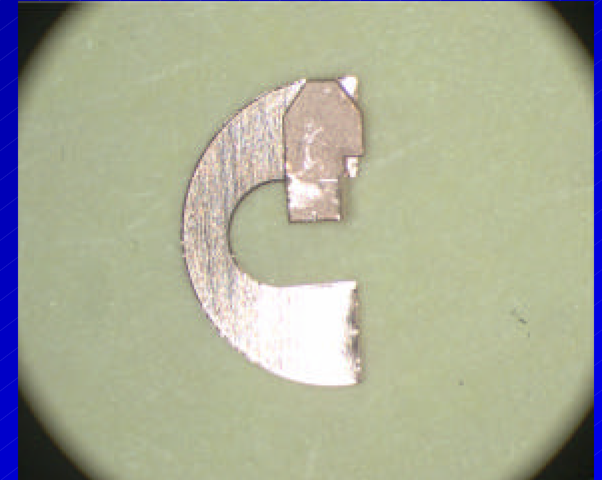
Cutting SACT Grids for Pre-FIB Method



Start with 2x1 Veco tabbed grids.

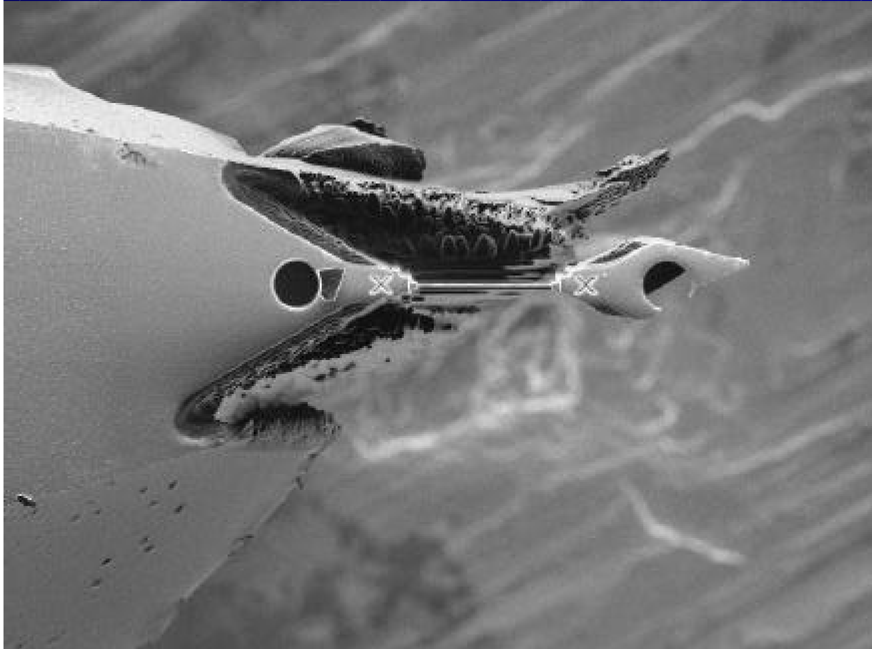


Cut the grids as shown, aligned with tab.



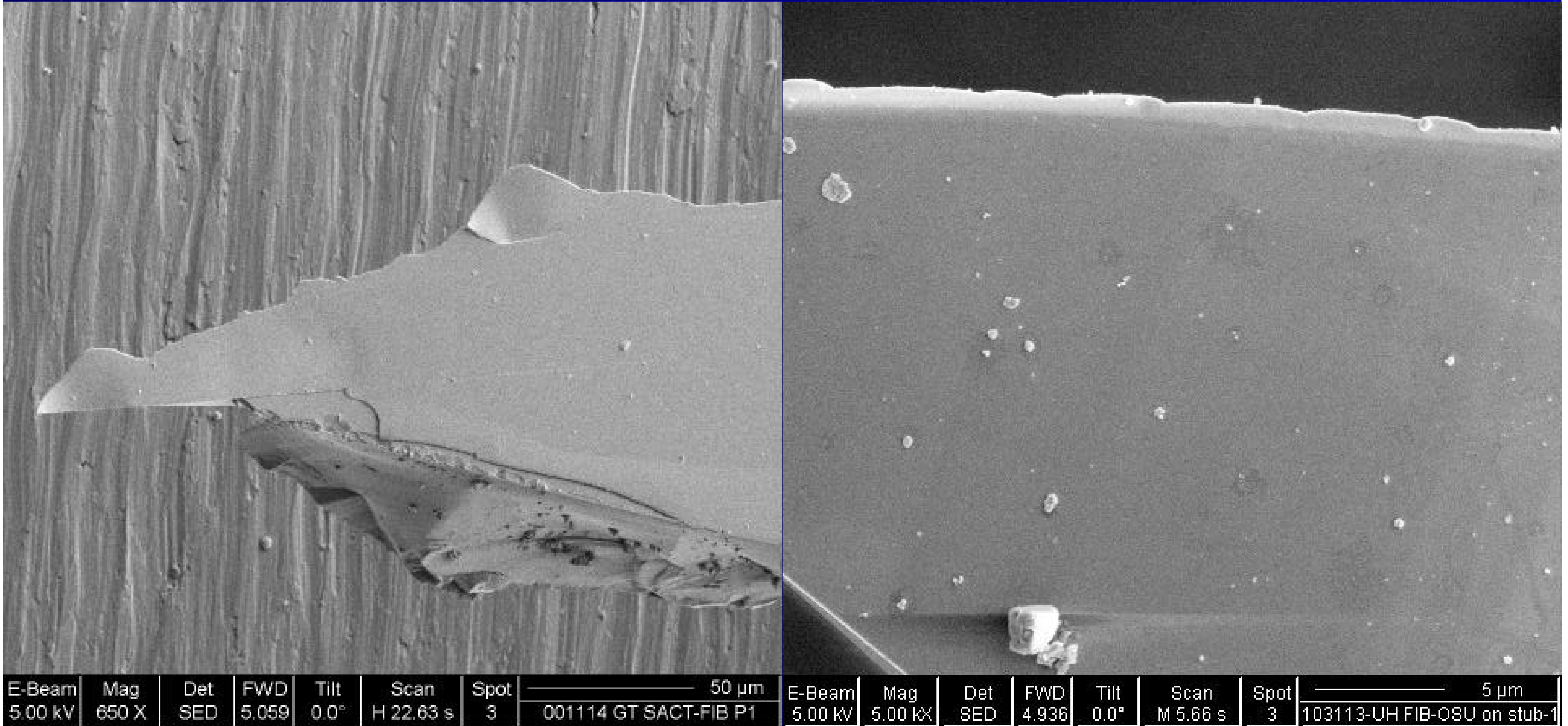
Bend the vertical tab and the handle normally. Use a dab of epoxy on grid when mounting sample to stabilize it.

AutoTEM Used With SACT/FIB



The first test of the SACT/FIB method on a Solarban® 55 coated glass sample showed a crystallite of NaCl at the glass/film interface (arrow). Although cubic holes had been seen in the film, this was the first time that they had been imaged in cross section in the TEM.

Examples of Suitable SACT/FIB Samples



Very thick Nb/Zr multilayer metal coating on Si. The tearing and deformation at edges prevents usable SACT Samples. Samples can be made further back from apex.

A large distribution of film defects on Sungate® 1000 coated glass is ideally suitable for the SACT/FIB method.

The electron beam is used to deposit on samples where film is very thin and a top layer would obscure details, but it is a slower process.

FEI xP — USER (Supervisor)

File Scan Detector DB Control E-Mag I-Mag E-Beam I-Beam Patterning Stage Tools Window Help Pages

To prepare a sample at the tip, trim the tip, perform deep rough cuts, tilt finishing boxes by half of the amount of taper desired. The sample should be tilted by ±1.2° for finishing.

I-Beam	Mag	Det	FWD	Tilt	Scan	pA	2 µm
30.0 kV	8.00 kX	CDM-E	17.0	52.0°	M 1.58 s	110	103284-2 Type II SB60-RH

I-Beam	Mag	Det	FWD	Tilt	Scan	pA	2 µm
30.0 kV	8.00 kX	CDM-E	17.0	52.0°	M 1.58 s	110	103284-2 Type II SB60-RH

E-Beam	Mag	Det	FWD	Tilt	Scan	Spot	2 µm
5.00 kV	6.51 kX	SED	4.979	0.0°	H 22.63 s	3	103284-2 Type II SB60-RH

I-Beam	Mag	Det	FWD	Tilt	Scan	pA	2 µm
30.0 kV	8.00 kX	CDM-E	17.0	52.0°	M 1.58 s	111	103284-2 Type II SB60-RH

Advanced Features

Use: I-Beam E-Beam None

µm Dwell: µs
µm Overlap: %
µm Time: Sec

Material File:
Rotation: deg

Center Position: X: 0.0 µm Y: 0.0 µm

Axis Scan: Long Axis X Axis

Close

Magnification Scan Rotation Zero
0.0 0.0

Stage

POS6
POS7
POS8
POS2

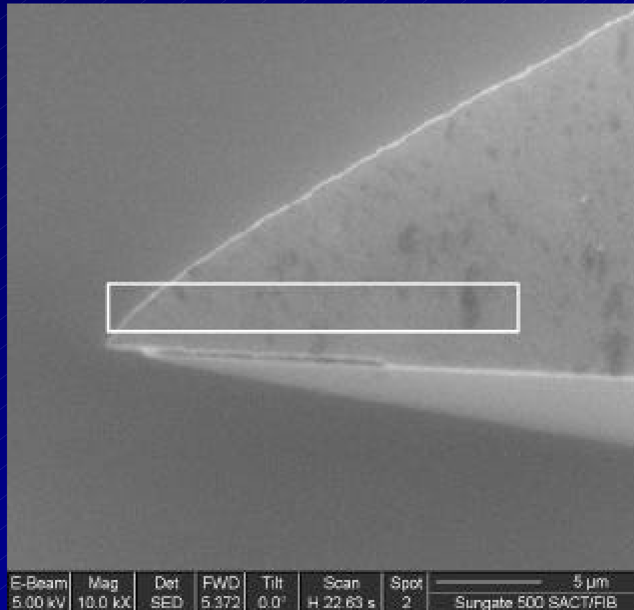
Use UU Z = FWD Absolute Relative

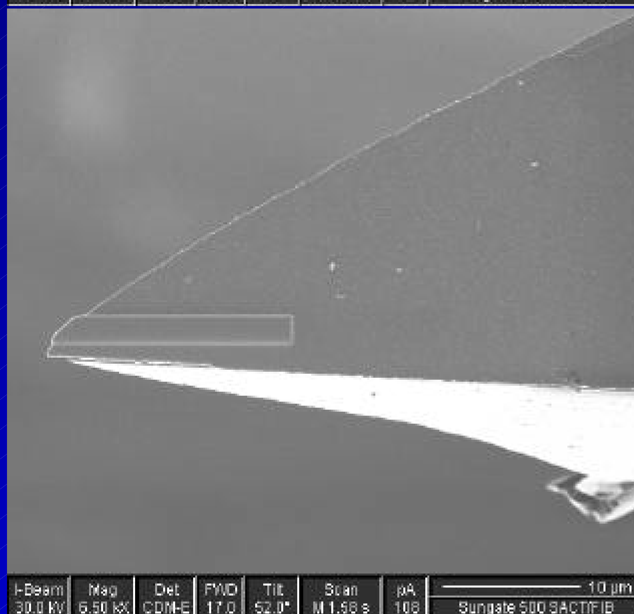
X: -5353.63 µm R: -171.052 Deg
Y: -186.24 µm T: 53.20 Deg
FWD: 4.903 mm

Status

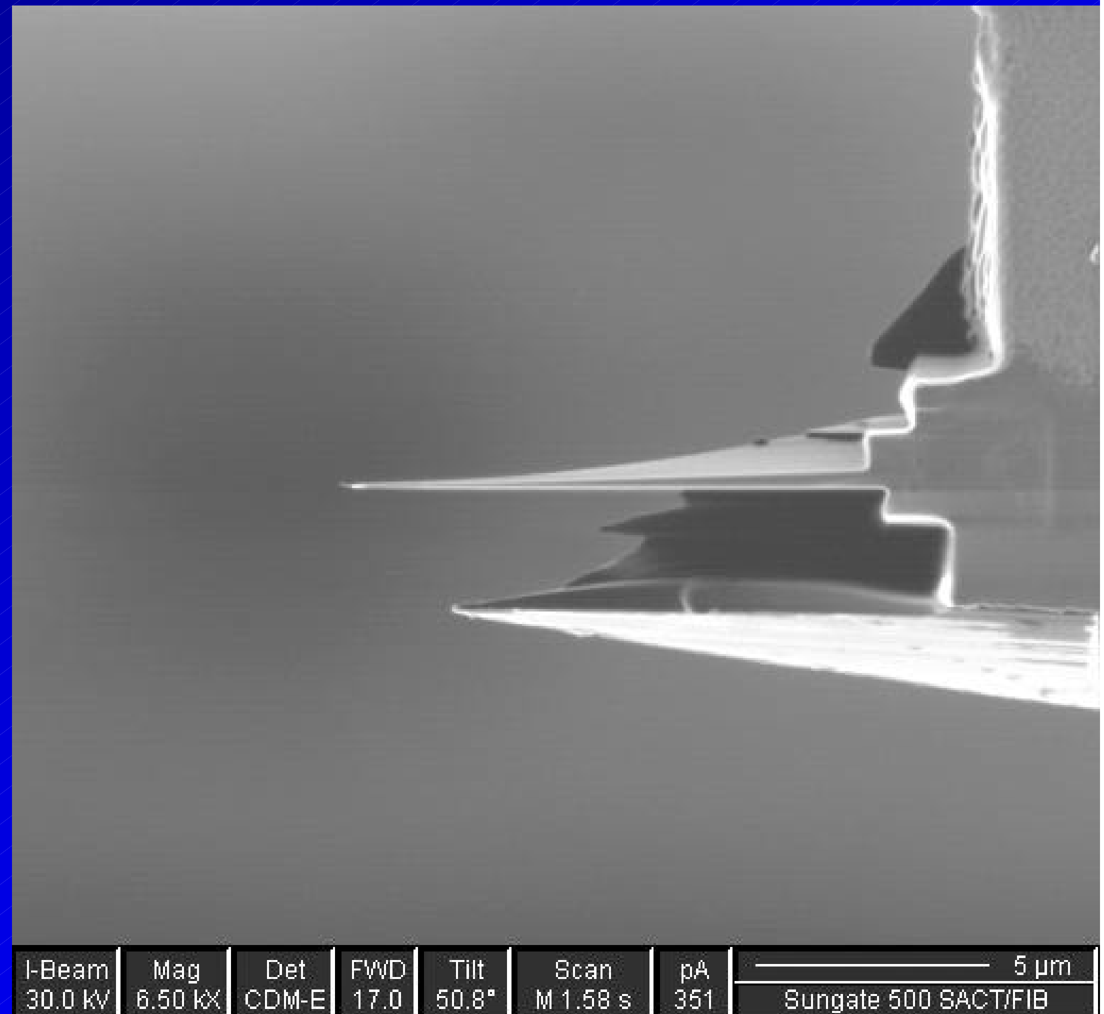
Ion Column Aperture:	100	pA
Ion Beam Current:	100	pA
Ion Emission Current:	2.2	µA
E-Column HV:	5.0	kV
E-Spotsize:	3	
Chamber Pressure:	1.0e-5	mbar

Apex FIB Cuts of SACT-Prepared Sungate® 500 Coated Glass Sample

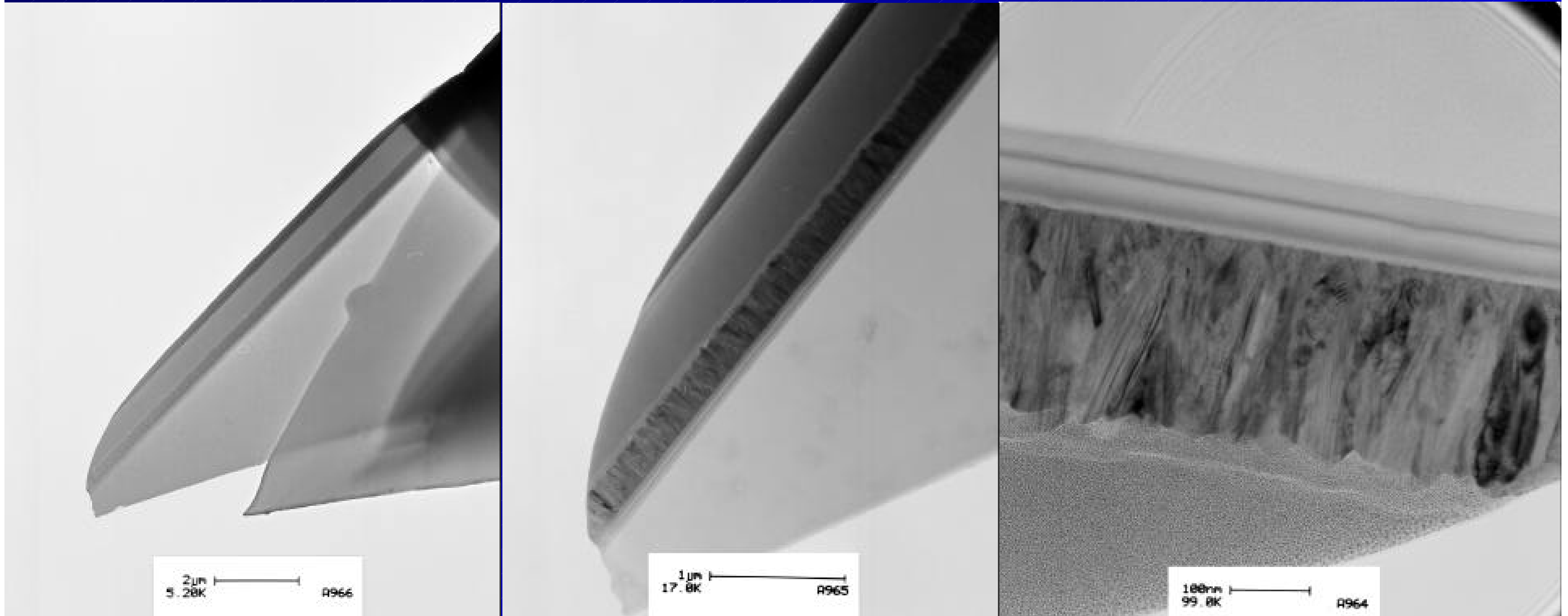




The sample is cut very quickly at the apex of the SACT sample with a taper angle used with the clean-up-cut boxes.

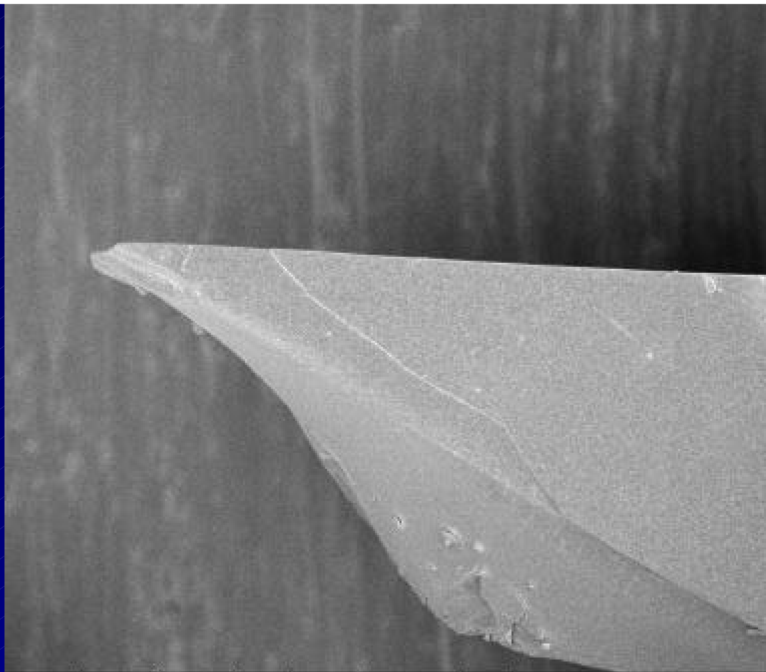


Apex FIB Cuts of SACT-Prepared Sungate® 500 Coated Glass Sample

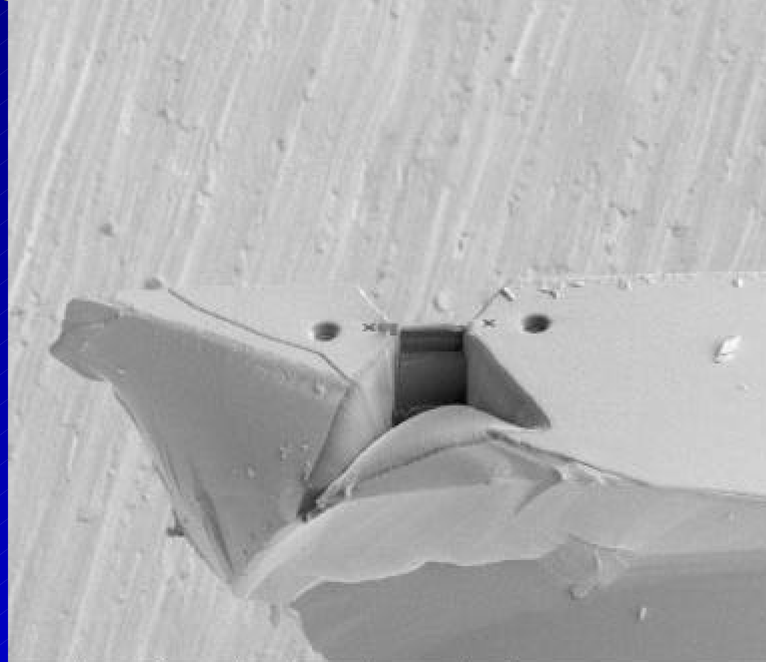


By using a taper, the sample is more robust and less likely to “wave in the breeze.” If the substrate is fully removed, then the sample can be cleaned up to remove FIB artifacts in a low angle ion mill.

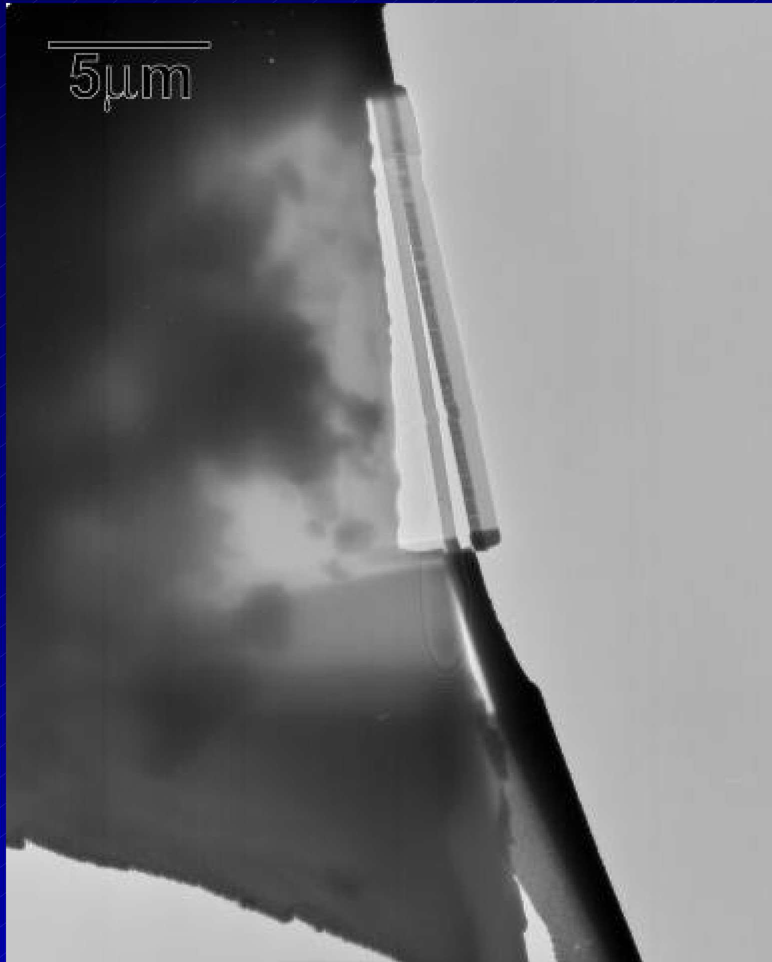
SACT/FIB Sample Cut Using the FEI AutoTEM Process

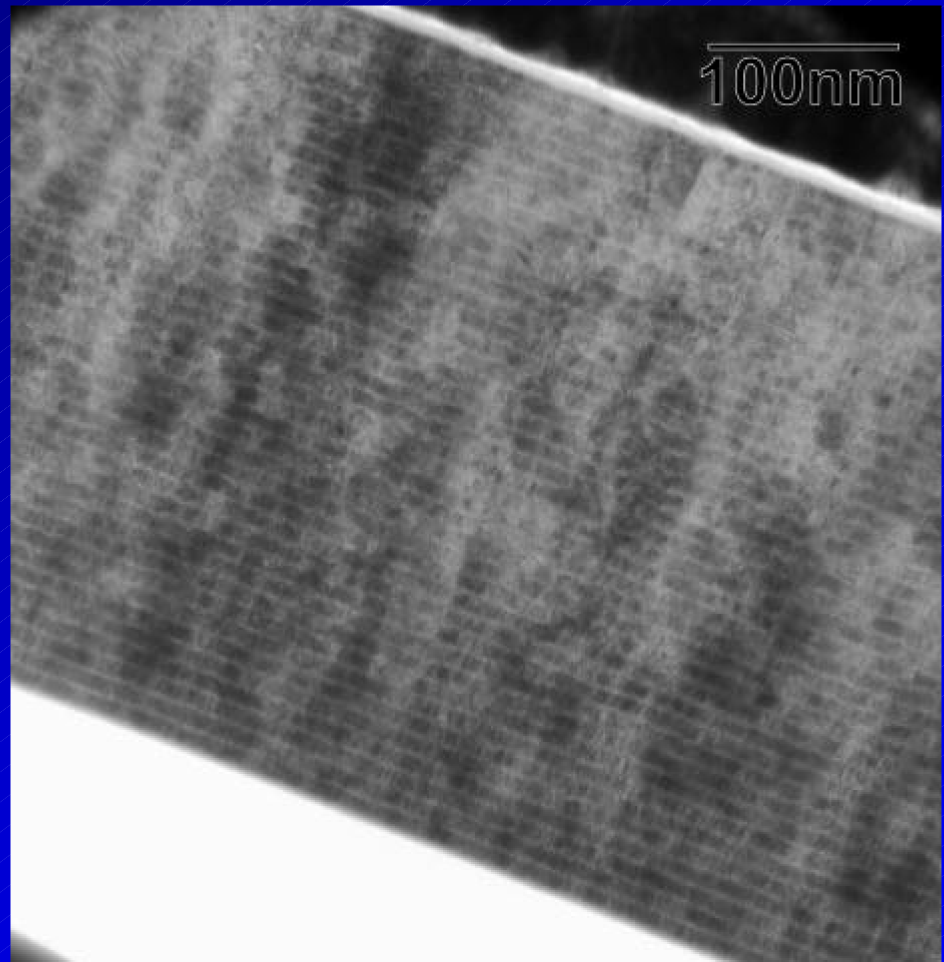


This is an example of a step in the film stack occurring during the SACT process. The sample is pre-thinned for the FIB.



Nb/Zr Multi-layer on Silicon



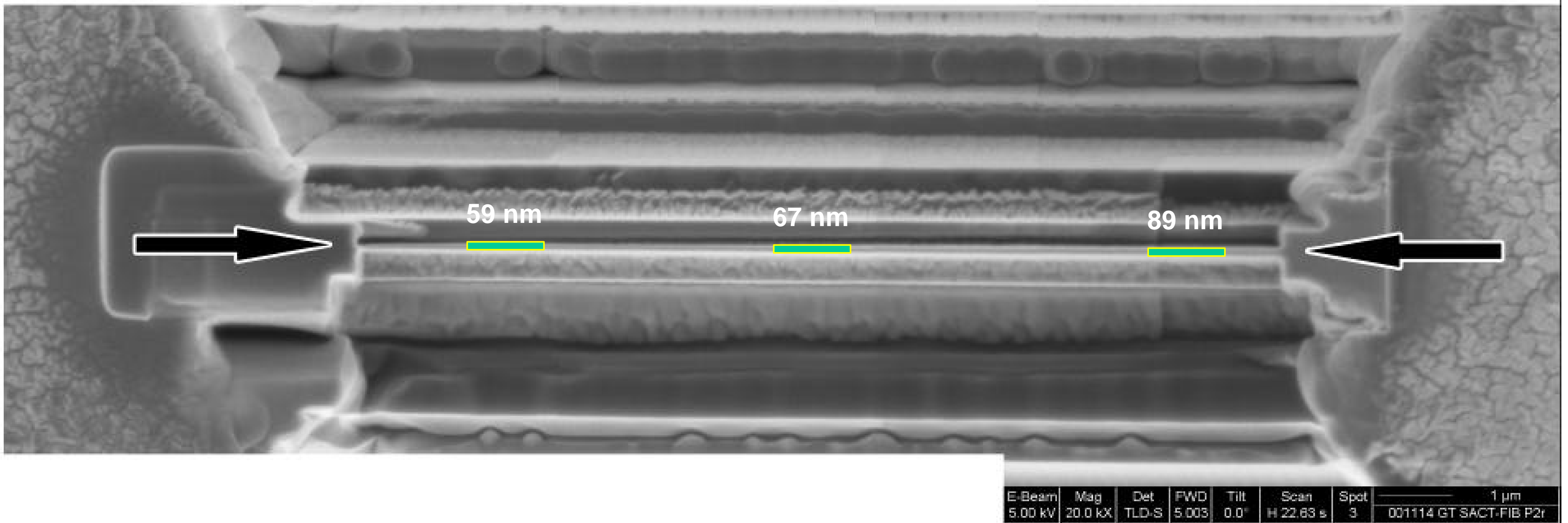


Even with a metallic thin film that has a tendency to delaminate, the SACT/FIB method was able to prepare a sample.

Conclusions

- The Small Angle Cleavage Technique (SACT) is an effective method for rapidly pre-thinning samples for FIB.
- The SACT/FIB combination can "salvage" material sample systems that can not be prepared by SACT alone.
  - Thick metallization layers that plastically deform or tear.
  - Weak interfaces or materials that give a jog.
  - Thick Oxides that blunt the tip of SACT sample.
- The Apex and the Pre-Thinned Near-Apex Methods give samples that are readily cleaned up by a low angle ion mill afterwards.
  - Care must be taken to ensure that the substrate material is sufficiently removed.

Add a Taper to Have One End of Sample Thinner for EELS and HREM Applications



SACT/FIB Tip Cut With Taper