

Materials Characterization Core at Drexel University

Training Library – Standard Operating Procedures

FIBSEM – TEM Lift Out Procedure

Manager: Craig Johnson
Director of Operations

Contact: Drexel University
Bossone 212
3141 Chestnut Street
Philadelphia, PA 19104
Office: 215-895-5900
Mobile: 610-400-7627
Zoom: 896-243-5909
clj46@drexel.edu

Latest Update: 2020-06-22

These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.

Before using the MCC, please review the MCC User Handbook available through our website.

TEM LIFT-OUT Technique using DualBeam and Omniprobe

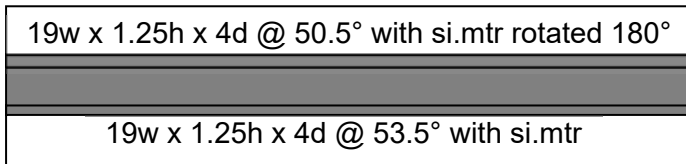
1. Deposit 1 um Pt using 100 pA, pt_tem.mtr centered on your area of interest. Use ebeam Pt or carbon/Pt sputter coater first if surface material is critical (e.g., a thin-film sample).

20w x 3h x 1d with pt_tem.mtr

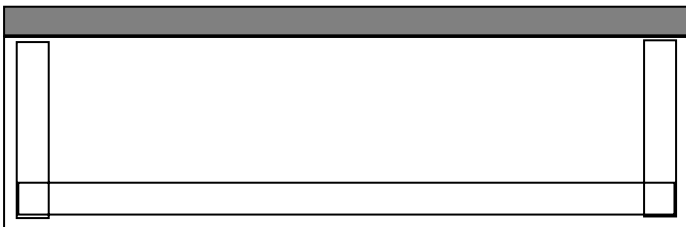
2. Mill a regular cross section on the front about 20 x 8 x 4 using 7000 pA, si.mtr, rotate the pattern and mill on the backside.



3. Mill regular rectangles in series near the Pt using 3000 pA and si.mtr. On the front side, tilt the stage to 50.5 degrees. On the backside, tilt the stage to 53.5 degrees. This will give the sample parallel faces (less wedge shaped).















4. Tilt the stage to 7°. Set scan rotation on ion beam to 180°. Draw regular rectangles (1.5 um wide) in the shape of a "U". Use parallel milling with 3000 pA. Monitor with 1E.



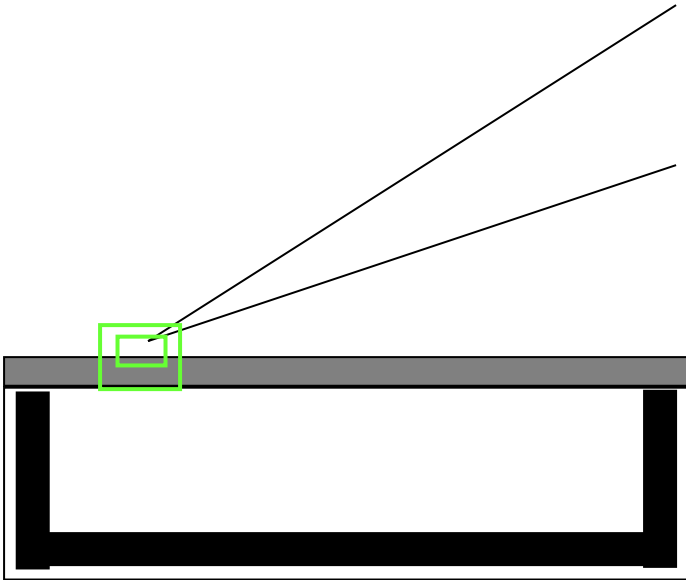
- Tilt sample to 0°. Lower stage 200 μm below eucentric height with Z knob. Insert the Omniprobe needle. Bring sample back to eucentric height. Move O-probe to the sample surface following instructions in the note below.

Omniprobe movements in electron and ion images

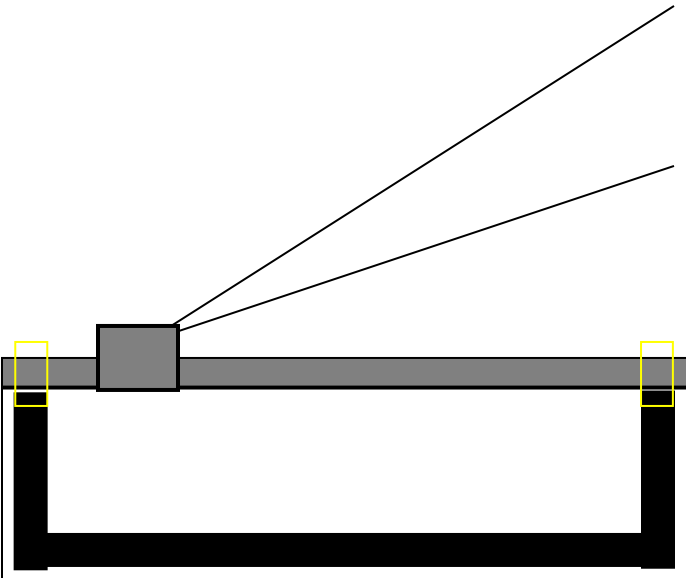
	X		Y		Z	
Imaging beam	e-beam	i-beam	e-beam	i-beam	e-beam	i-beam
Top button						
Bottom button						

Note on Omniprobe Movement: After insertion of the O-probe, bring it to the center of the screen using top Y button while observing in the e-beam. Next, bring the probe to the sample surface by toggling between Z and Y bottom buttons while observing in the i-beam. To move the probe back to the parked position, use the top Z button only, while observing the position in the e-beam.

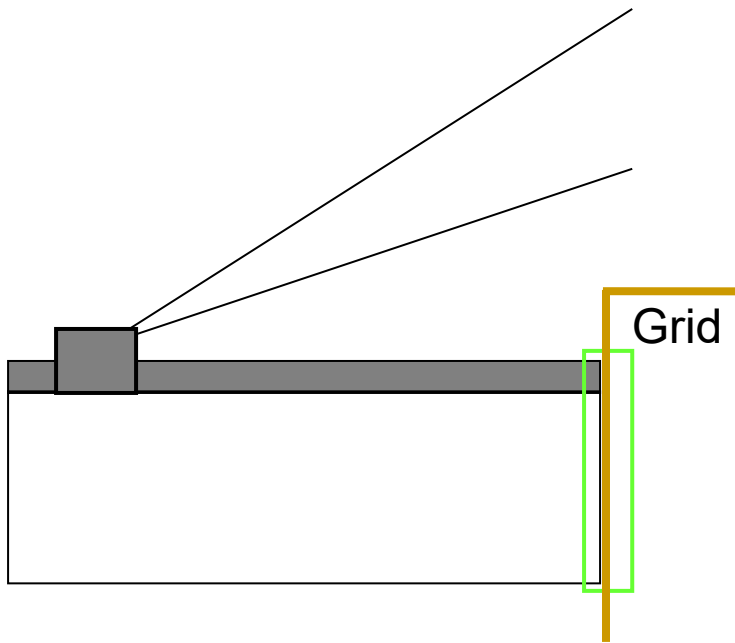
6. Leave a very small gap between the needle and the sample, then place your Pt box just underneath the needle and a smaller box covering the gap. Deposit weld in parallel with 10-30 pA Pt. Do not retract the Pt GIS.



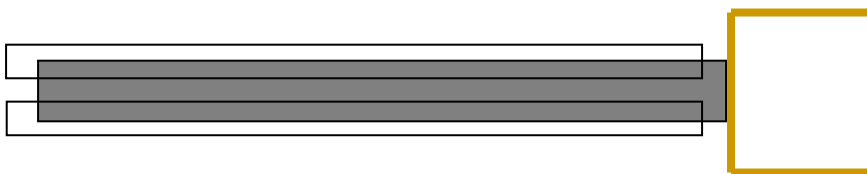
7. Cut the remaining "Pt bridges" free by serial milling with 100 – 500 pA and si.mtr. LOWER the stage until the sample is out of the trench. Put the Omniprobe in the parked location and retract the Omniprobe and the Pt GIS.



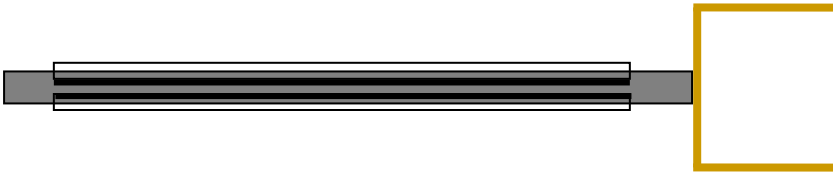
8. If necessary, exchange sample for grid.
9. Drive to the grid location. Make sure that the grid is parallel to the e-beam column at zero stage tilt. Bring the stage to eucentric height. Insert GIS and Omniprobe. Maneuver the sample down to the grid and weld it to the grid using pt_tem.mtr and 100 pA. Do not retract GIS. Cut the Omniprobe-sample weld with 300-500 pA. LOWER the stage until the needle is above the grid. Put the needle in the parked location and retract the Omniprobe and the Pt GIS.



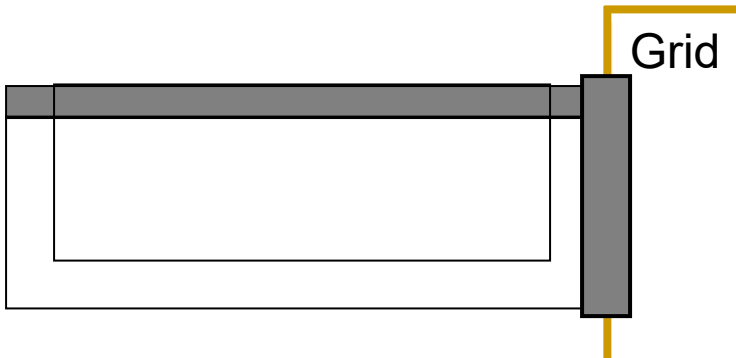
10. Tilt sample parallel to ion beam. Mill regular rectangles to cut lamellae thickness to $< 1 \mu\text{m}$. Use aperture that gives a time between 2 and 8 minutes. For the front side, tilt the stage to 53.5° . On the backside, tilt the stage to 50.5° and rotate pattern 180° . These are the top and bottom of the lamellae when viewed in the i-beam.



11. Cut window down to 100 nm using 100 pA aperture and cleaning cross sections with $z < 0.35$ μm (mill top first and check depth with e-beam image). Alternate sides with $\pm 1.5^\circ$.



Ion view at 52° .



E-beam view.

12. Reduce ion magnification to minimum. Get image increase mag and focus on the sample as well as possible. Start low kV mode (5 kV and 50 pA). Tilt the sample so that the ion beam is at 5° to the bottom of the sample. Mill a regular rectangle with time set for up to 5 minutes. Tilt sample so ion beam is 5° to the top surface (surface facing the e-beam) and mill for up to 5 minutes. Frequently observe the sample in the e-beam and watch for darkening that indicates electron transparency. If holes form or any Pt completely disappears, immediately stop milling.

NOTES:

- Before you begin welding with the Pt, take note of the chamber vacuum. During the welding the chamber pressure will increase slightly. After the Pt is done, the pressure will remain at this heightened pressure for a minute or two. You must wait for the pressure to come back to the initial pressure before you mill. Otherwise you will not get a clean cut because of the residual Pt in the chamber.
- Whenever you are using the Omniprobe, make sure the stage tilt is 0°. Liftout from the bulk and welding to the grid are always done with 0° stage tilt.