TEM Lamella Sample Preparation

Preparations:

1) Place sample inside the SEM/FIB using the special TEM grids holder.
2) Make sure to check Compucentric Rotation at navigation tab. Check Compucentric tilt at the navigation tab only when moving along Z while stage tilt is not 0°.
3) Set eucentric height on the sample following the instructions in the main manual.

Stage 1 – Pt protective layer (Ebeam and/or Ibeam deposition)

Follow the Ebeam and Ibeam Pt deposition steps to add protective Pt layer on top of the sample surface. The procedure is briefly described below:

1) For delicate samples where protecting the top layer is important an initial Pt layer (0.1 um thickness) deposited by e-beam is recommended. Stage tilt is 0°.
    - Place Pt dep pattern in Ebeam quad (Application: **Pt e-dep surface**, usually the size is 20 x 2 x 0.1 um). Use e-beam settings 2 kV and 1.6 nA.
    - Inset GIS needle and start deposition.
    - This step takes several minutes. Take Ebeam snapshot to confirm that a well-formed rectangular layer is visible.
    - Retracted GIS needle and then proceed to next step on top of the prepared Pt layer.
2) For not so delicate samples, tilt the sample to 52° at eucentric height.
    - Place Pt dep pattern in Ibeam quad (Application: **Pt dep**, usually the size is 20 x 2 x 2-5 um). The rule for good Pt deposition is to use a current calculated by the area $\times$ **6 pA/um^2**. For the case above we use FIB settings of 30 kV and a current close to 0.23 nA.
    - Insert GIS needle. Deposit for as long as necessary (turn on iSPY to monitor the formation of Pt layer, you can stop the deposition at any time once you feel the thickness is good enough).
   - Retract GIS needle.

Note: Always take a quick snapshot for Ibeam whenever you change current to make sure no image shift in Ibeam. Check the focus and stigmator whenever changing the Ibeam current, if necessary adjust them.

Stage 2 – Rough cross-section (regular cross section)

1) The regular cross-section needs to be approximately twice the length of the Pt layer and y dimension should be twice the intended Z depth (X = 2*Pt_dep_length, Y = 2*Z_depth). For the Pt layer above use regular cross section pattern size 40 x 20 x 10 um. Z depth needs to be calibrated with your material according to Si.
2) Place the pattern below the Pt edge at a y distance of 2-3 um. Make sure scan direction (actually milling direction) is Bottom to Top. Mill at **21nA, 47nA or 65nA**.

   Note: if you turn the iSPY on in this step, do not pause the SEM until the milling finishes.

3) Reverse the milling direction (Top to Bottom) and place the pattern above the Pt layer. Repeat milling. The final result should look like the one below in Ebeam quad.
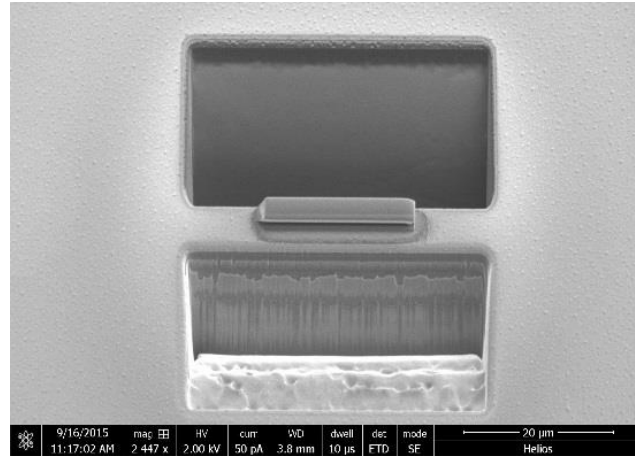
Stage 3 – wedge shape cut

1) Set Ion beam to viewing current (25 pA/40 pA).
2) Tilt stage to 53.2° (52°+1.2°).
3) Make a quick Cleaning Cross Section (CCS) on the bottom edge of Pt layer with **2.5 nA**. for example 41 x 1.5 x 15 um.
4) Tilt stage to 50.8° (52°-1.2°) and repeat cleaning cross section on the upper edge. Reverse the milling direction (Top to Bottom).
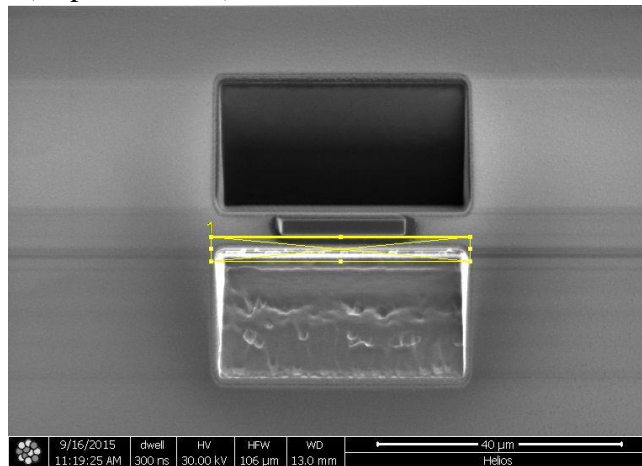

Stage 4 – undercut (J-cut) to separate slab

1) Tilt stage to 0°.
2) Set Ibeam to 25 pA/40 pA. In beam panel set scan rotation to 180° in Ibeam quad for convenience.
3) Set milling current to **9.4 nA**. Make patterns using **Rectangle** (Application: **Si new**) similar to the one shown below with Z depth of 10 um. Choose wider J-cut.
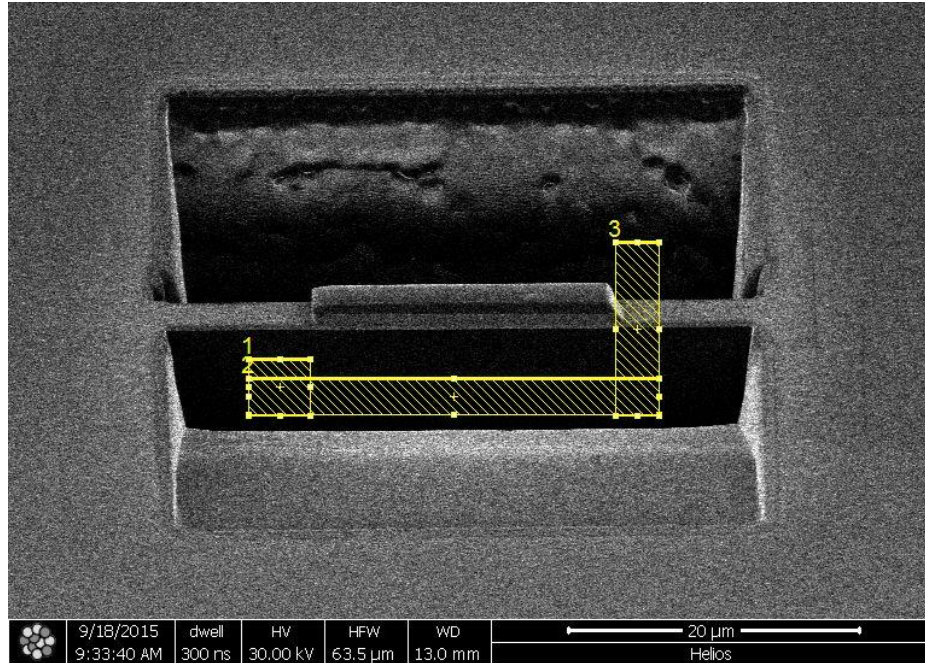
4) Set milling options to **parallel milling** and watch the cut progression in Ebeam quad until a full undercut is seen:
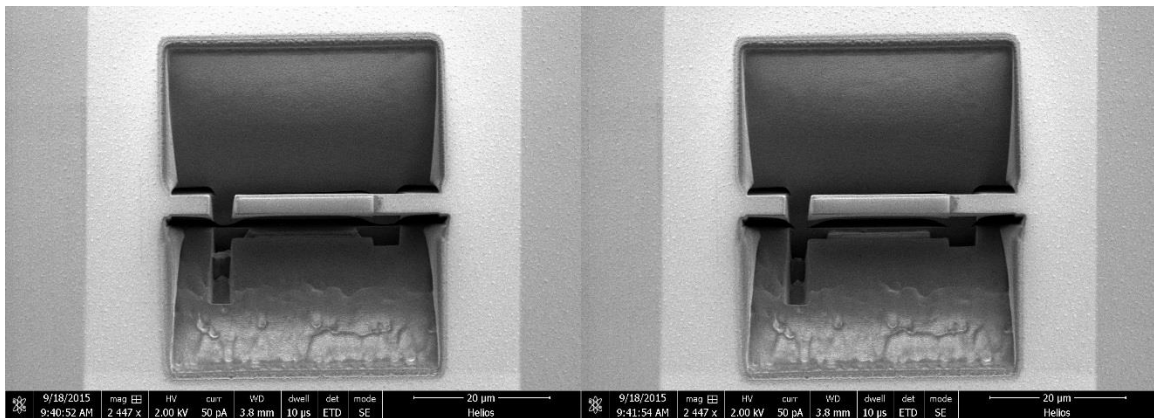

5) Tilt to 30° and examine the cut. You can also rotate the sample 180° (Relative in the Stage) to mill from the other side.
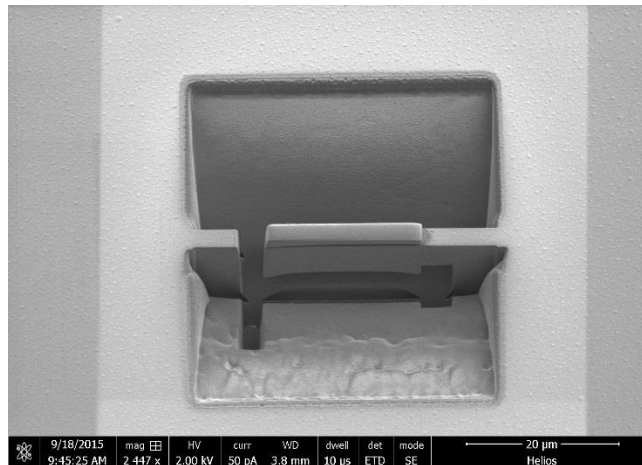
6) Set Ion beam back to 25 pA/40 pA. Set tilt to 0°.

Stage 5 – Patch slab to Easy lift needle

1) In movements tab select Easy Lift. Click Insert. Choose **Park position**.
2) In the SEM window zoom out. The needle comes in from the left side of the screen and needs to be manipulated to the long cut side of the slab.
3) Only manipulate X-Y by pressing left mouse button and dragging in the Ebeam quad. Never align Z in Ebeam quad. Align tip X-Y with the Pt rectangle
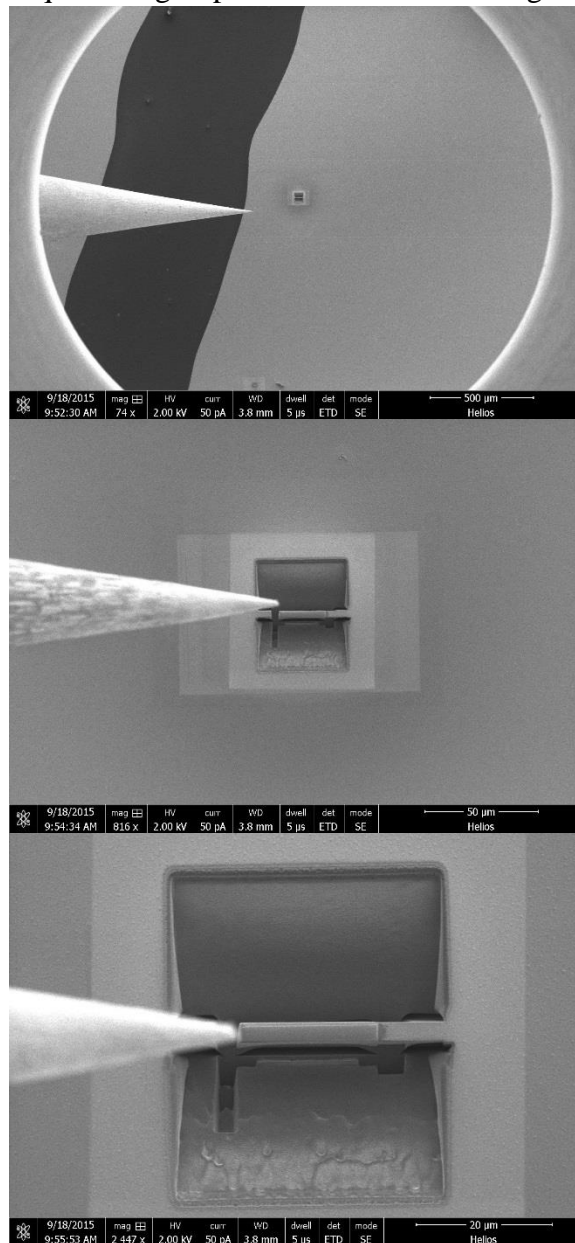
4) Align X-Y in the Ebeam quad and align the Z in the Ibeam quad until the needle is 1-2 um above the side of the Pt rectangle. (Scan rotation is 180° in Ibeam quad.)
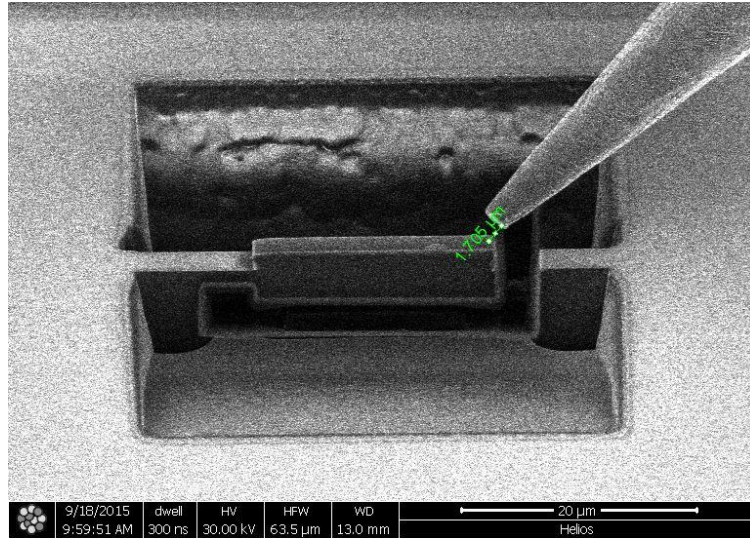
5) Bring in GIS needle.
6) Continue to manipulate the EZlift needle until it gently touches the edge of the slab. Go back and forth between SEM and FIB view until satisfied. Press ctrl+J to disable EZlift controls.
7) Make a 2 x 2 x 1 um patch for Pt dep with Ibeam. Recommended deposition rate 6 pA/um^2. I used **24 or 40 pA** with good results. After the deposition, retract GIS needle.
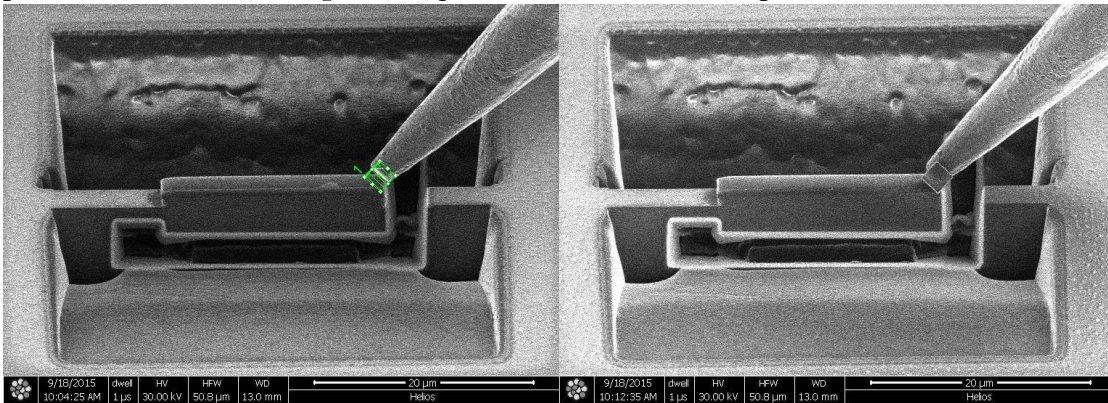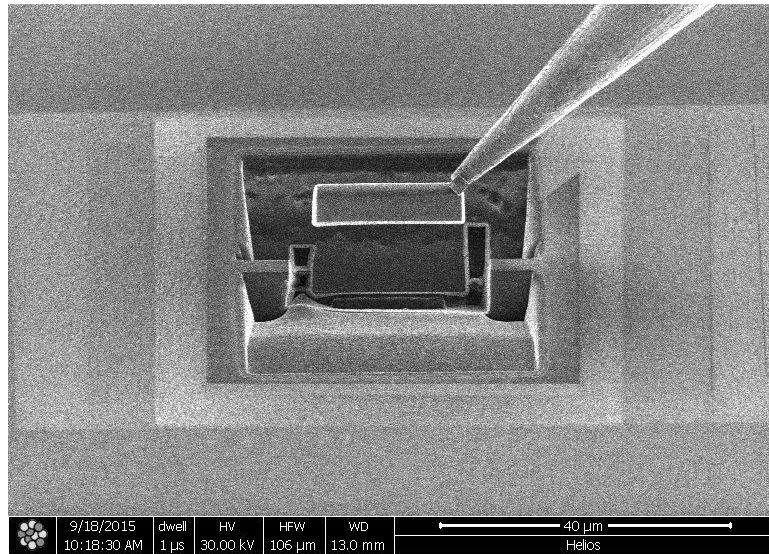

8) Separation cut. Use **9.4 nA** draw a rectangle on the left side bridge to the slab. I used 3.24 x 6.15 x 5 um. Mill until slab is released.
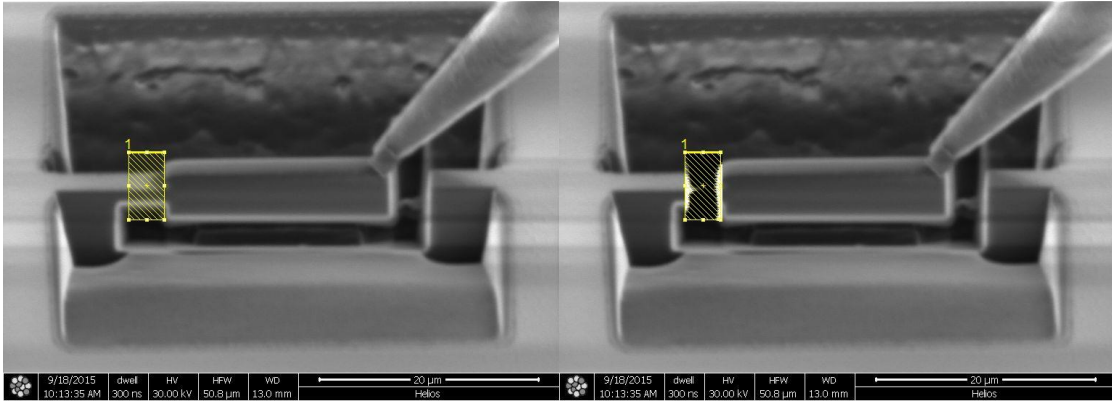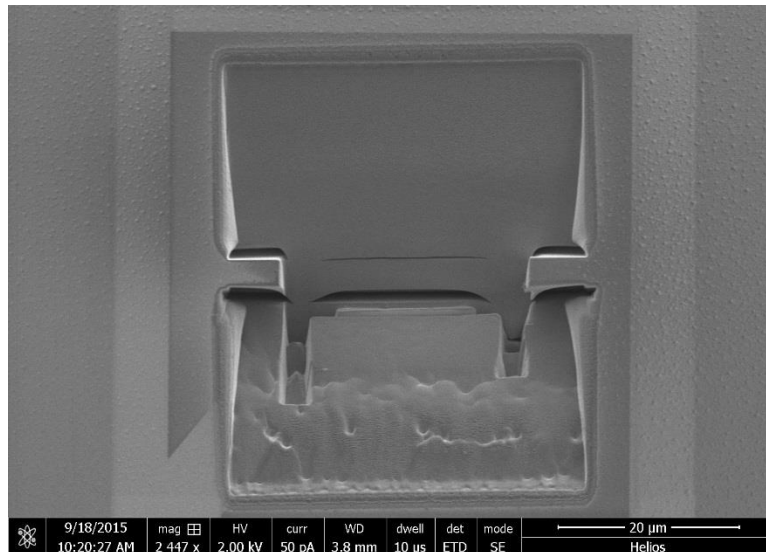
9) Change FIB current back to 25 pA/40 pA.
10) Use EZlift controls in Ibeam to bring up the slab in Z direction until it is hanging over the excavation area.


11) In the EZlift menu go to park position first and then retract the EZ lift needle.

Stage 6 – Attaching to TEM grid

1) Use xT align feature in the Stage tab to align the TEM grid horizontal.
2) Make sure the stage tilt is 0°. Locate a fork or column to place your sample on.
3) Ion beam at 25 pA/40 pA. In Ibeam quad set scan rotation to 180°.
4) Set Eucentric height on the grid. Choose a proper stage tilt such that the TEM grid is upright. Should be very close to 0° but could deviate a bit.
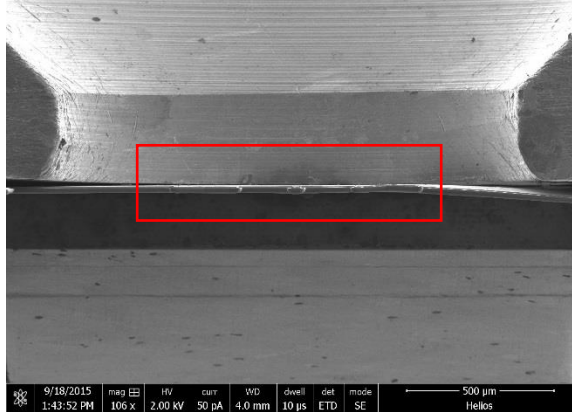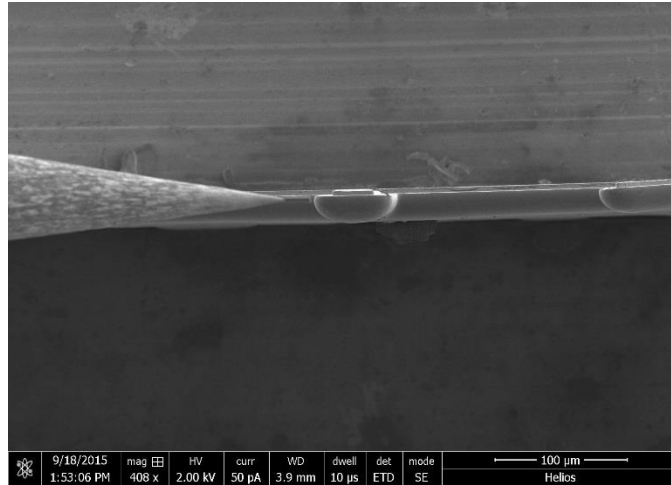

5) In Ibeam quad TEM grid should be visible from the side:
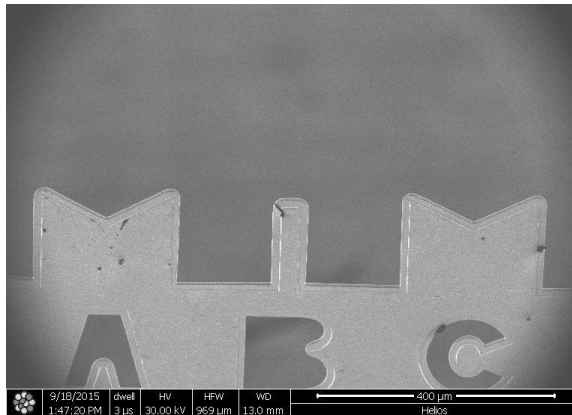
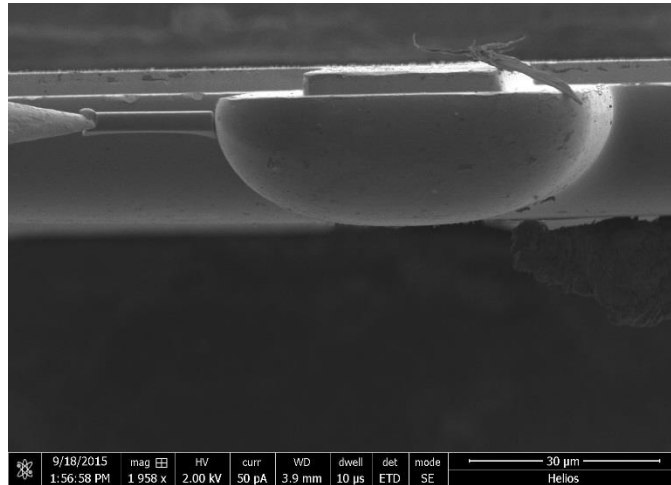6) Possible types of attachments to grid:
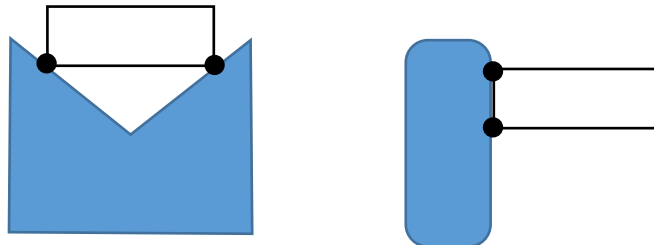
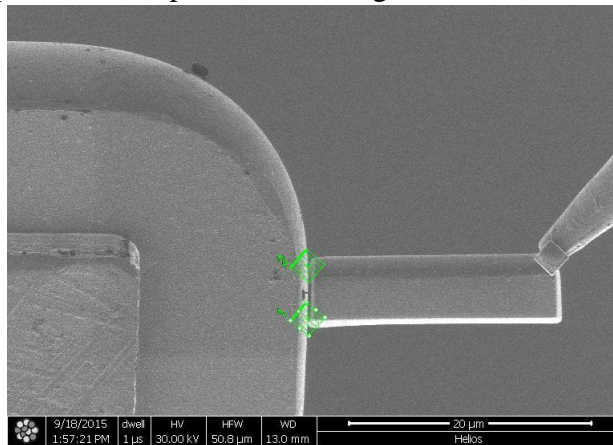7) Bring in EZ lift needle (same instructions as before).

8) Bring needle close but do not touch the grid.
9) Manipulate your sample slab to gently touch the side edge of the TEM grid (you can switch between Ebeam and Ibeam quads).
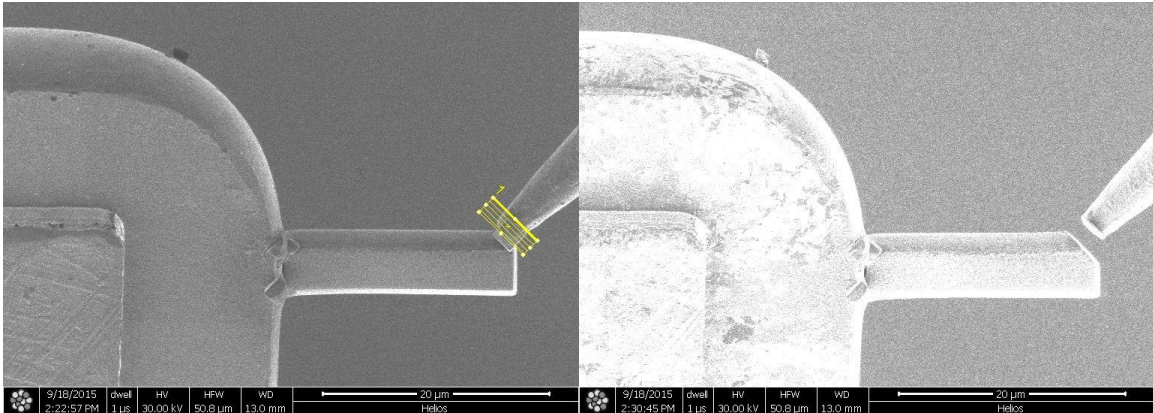

10) Insert GIS needle. In Ibeam quad make 2 patches for Pt dep 2 x 2 x 1 um and use **24 or 40 pA** of current to patch the sample to the TEM grid.
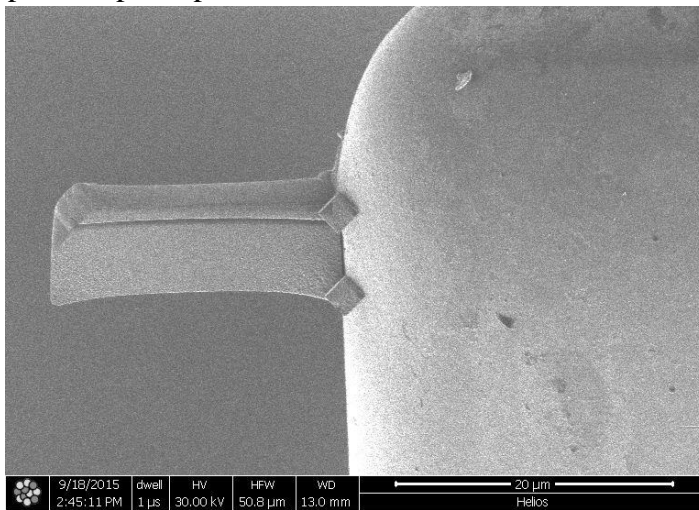

11) Remove GIS needle.

12) Separate the EZ lift needle. Use 9.4 nA current and make a rectangular cut as shown (5.5 x 1.1 x 5 um). Stop the milling once the cut is complete.


13) Move EZlift needle away from sample and then go to park position. Retract the needle.
14) The patches must be repeated on other side as well. Relative rotate the sample 180° (stage rotation).
15) Insert GIS and repeat the patch pattern.

16) Remove GIS needle. Relative stage rotate 180° to return to original position. Set scan rotation back to 0°.

Stage 7 – Thinning the lamella

1) Make sure that eucentric height is maintained on the sample.
2) Find a tilt angle (Theta) for which the sample is upright in Ibeam view, it may not be but should be close to 52°.
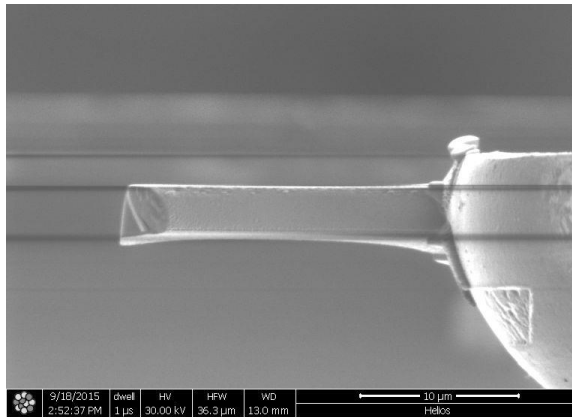
3) Tilt to Theta + 1.2°. Save position as "front".
4) Use **0.79 nA** current and make a cleaning cross section milling on the bottom edge (see picture below).
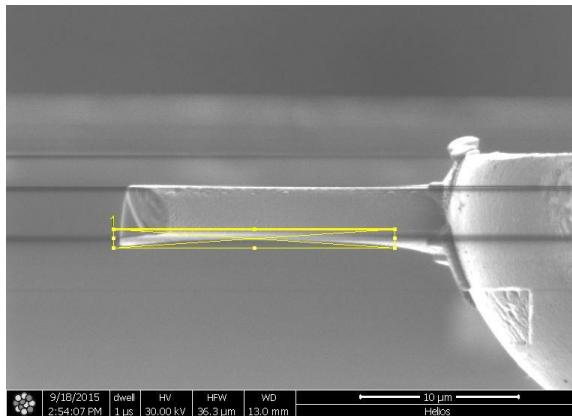
5) Tilt to Theta - 1.2°. Save position as "back". Repeat cleaning cross section on the top edge (remember to flip milling direction). After initial cleaning cross sections make sure the next is about half the slab width.
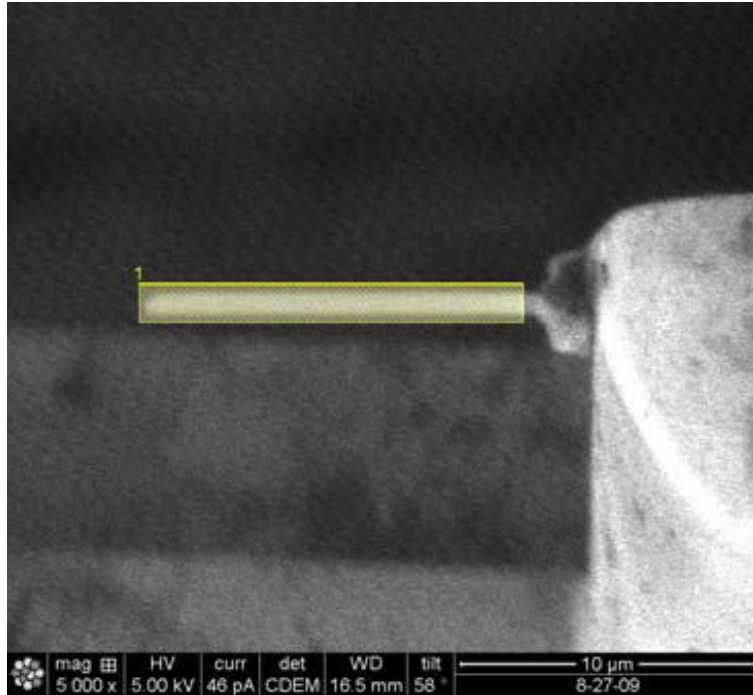6) Change current to **0.43 nA** when the thickness is below 750 nm and repeat the above steps.
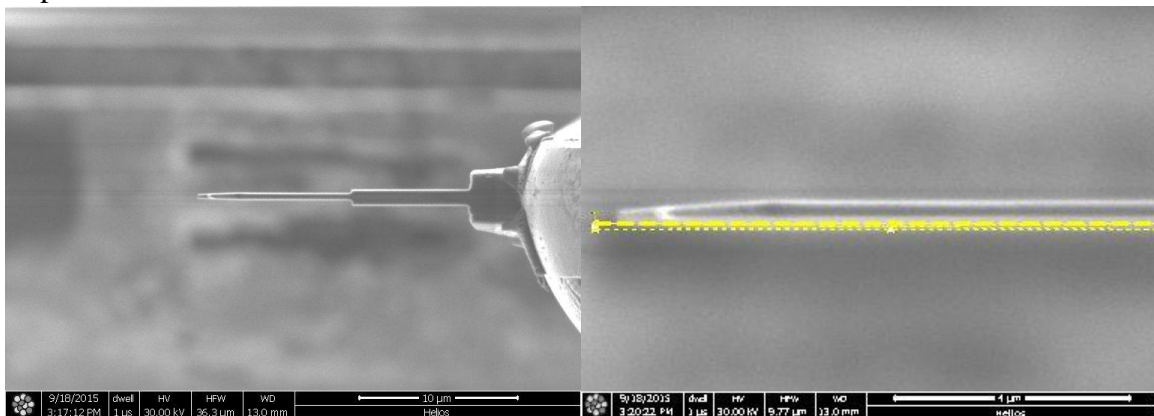
7) Decrease the current to **0.23 nA** as the sample thickness goes down to about 250-300 nm. The final thickness should be ≤150 nm (150 nm max). 50-100 nm is the ideal thickness.

| 9/18/2015 | dwell | HV | HFW | WD | 5 μm |
| 3:27:45 PM | 1 μs | 30.00 kV | 19.5 μm | 13.0 mm | Helios |
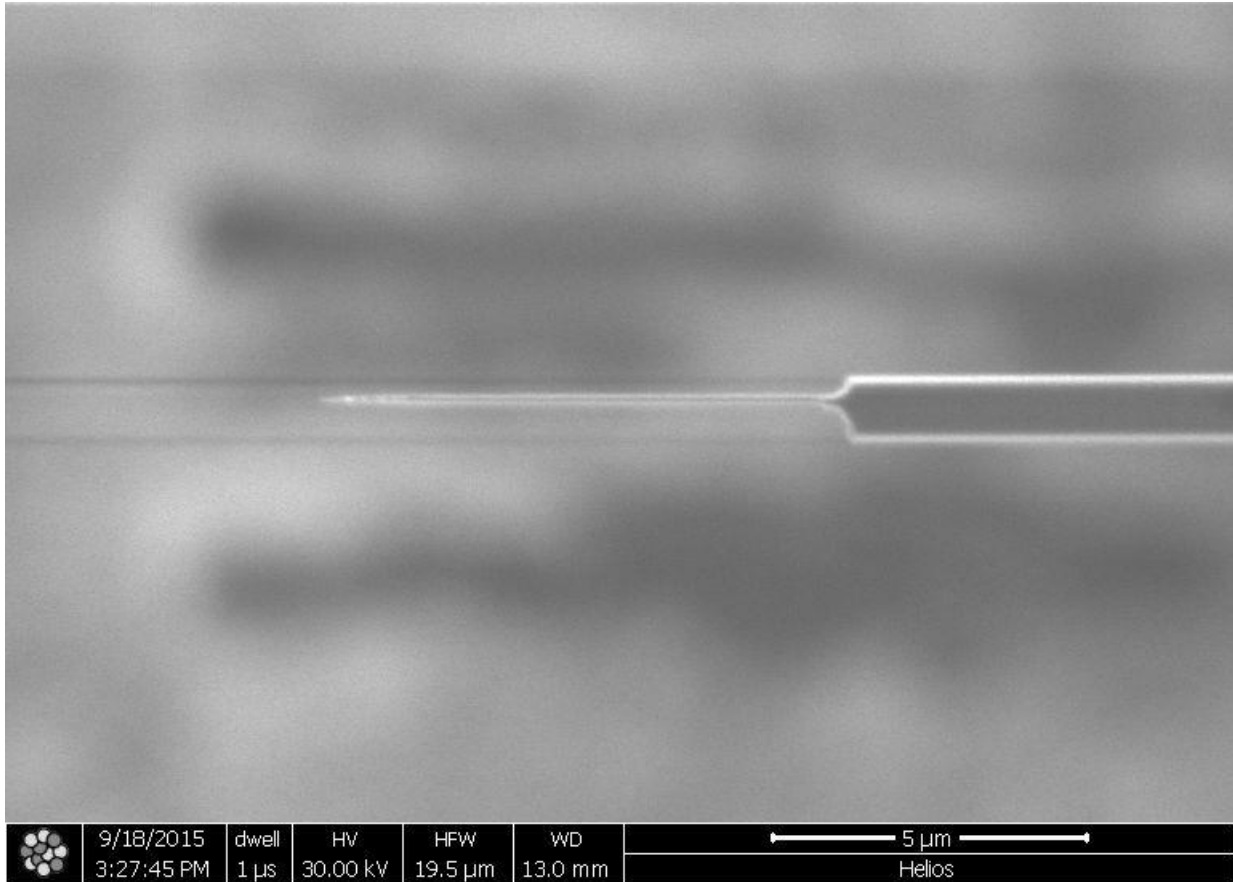
Note: Toward the ends of the thinning step use the magnify function in the pattern or measure tab to zoom in, which make it easier to setp up cleaning cross section position.

Stage 8 – Final cleaning

1) Tilt the stage to Theta + 5°.
2) Change the Ibeam HT voltage to 5 kV, current to 46 pA.
3) Pattern a rectangle box on top of the sample and mill 2 -5 mins, while monitoring the milling in the ebeam quad (current > 100 pA).
4) Tilt the stage to Theta - 5° and mill another 2 -5 mins, while monitoring the milling in the ebeam quad (current > 100 pA).
5) (Optional) For minimizing Ga+ damage, use 2 kV and 28 pA of Ibeam to mill both sides for 1 -3 mins at Theta +/- 7°.

| mag ⊞ | HV | curr | det | WD | tilt | ⊢————— 10 μm —————⊣ |
| 5 000 x | 5.00 kV | 46 pA | CDEM | 16.5 mm | 58 ° | 8-27-09 |

Stage 9 – TEM Imaging

1) Load the sample to TEM holder and image it in (S)TEM. If the thickness of sample needs further thinning, reload the TEM grid back to FIB.