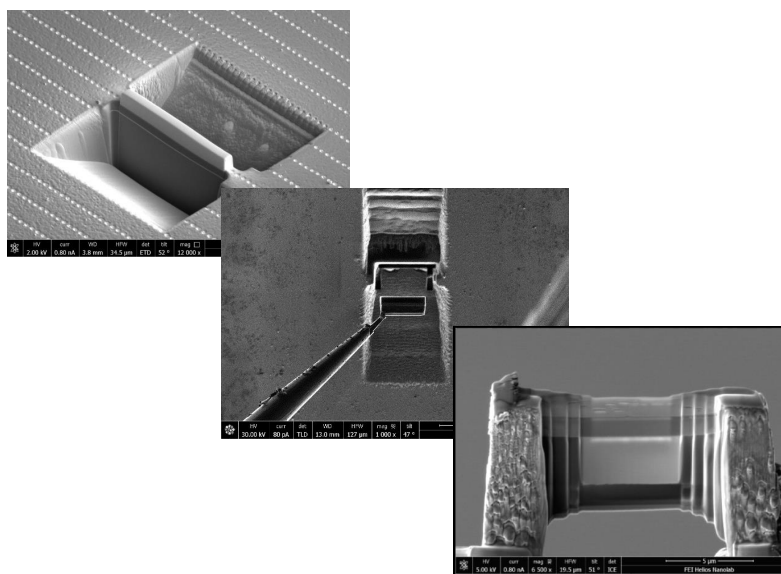


# TEM Sample Manual Preparation Procedure





## Manual TEM sample preparation procedures

### 1. Sample loading

- 1) Finish Z-link
- 2) Finish eucentric tilt correction.

### 2. E-beam Pt protection layer deposition:

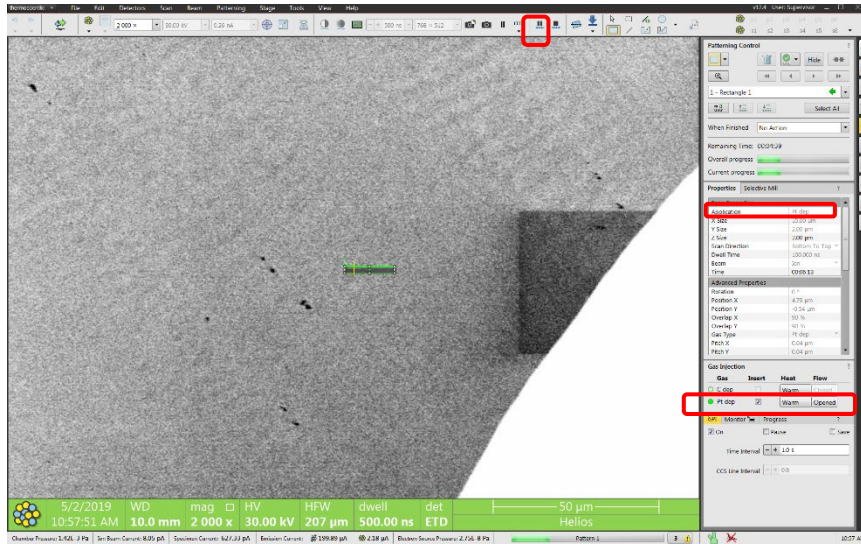
**Note:** carbon layer is recommended for less Ga and Pt damage to sample

- 1) Keep the sample tilt at 0°.
- 2) Set e-beam to **2 kV** and **0.21nA** or up to 1.6nA, and choose rectangle in Pattern Control panel. Typical size: **15  $\mu$ m (X) x 2 $\mu$ m (Y) x 0.2 $\mu$ m (Z: thickness)**. Dwell Time: **1  $\mu$ s**. Typical deposition time: **4 min**.
- 3) Insert the Pt GIS needle, open the gas flow and wait till the chamber pressure rise becomes slow, and start deposition/patterning.
- 4) Alternately if the sample surface is slightly charging, turn on reduced image area and change the scanning area to the same as deposition area. Keep the ebeam setting to **2 kV** and **0.21nA** and change the scanning time to 1  $\mu$ s, the same as patterning dwell time. Open the Pt gas flow and start e-beam scan/deposition. To finish deposition, close Pt gas flow.
- 5) Retract Pt needle and switch ebeam setting back to imaging conditions, typical 5kV and 96pA.


### 3. Ion beam Pt protection layer deposition:

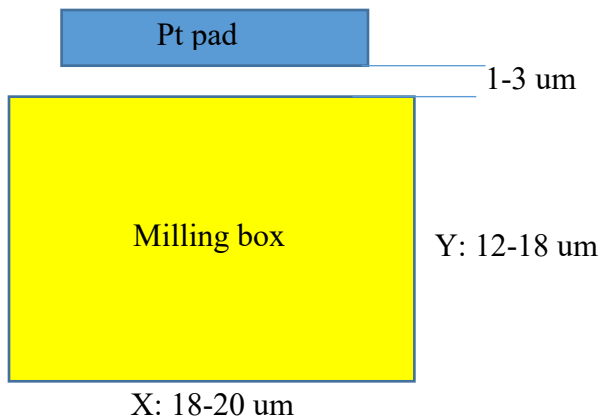
- 1) Tilt stage to **52°** and adjust eucentric height to center the e-beam deposited Pt layer in electron image.
- 2) Grab a screenshot in ion image window at typical imaging condition: 30kV and 30pA and use beam shift to center the Pt protection layer.
- 3) Draw a rectangle inside the e-Pt layer with Typical size **15 $\mu$ m x 2 $\mu$ m x 2 $\mu$ m**.
- 4) Choose **Pt dep** in Application and insert Pt GIS needle in highlighted areas below
- 5) Shift e-beam to re-center the e-Pt layer in electron image as affected by Pt needle movement.
- 6) Open the Pt gas flow and monitor the chamber pressure increase.
- 7) Wait until chamber pressure rise gets slower.
- 8) Choose typical **30 kV** and **0.10 nA** or up to 0.43nA on ion beam setting (smaller current favored for ion beam deposition).
- 9) If necessary, shift ion beam (use Shift key and left click to shift beam) outside e-Pt layer to adjust focus and stigma.
- 10) Click Pattern Start button to start deposition, typically takes ~ 8 mins.

- 11) After finish, **close** the Pt gas flow, **uncheck** the Insert box after deposition to retract GIS needle.



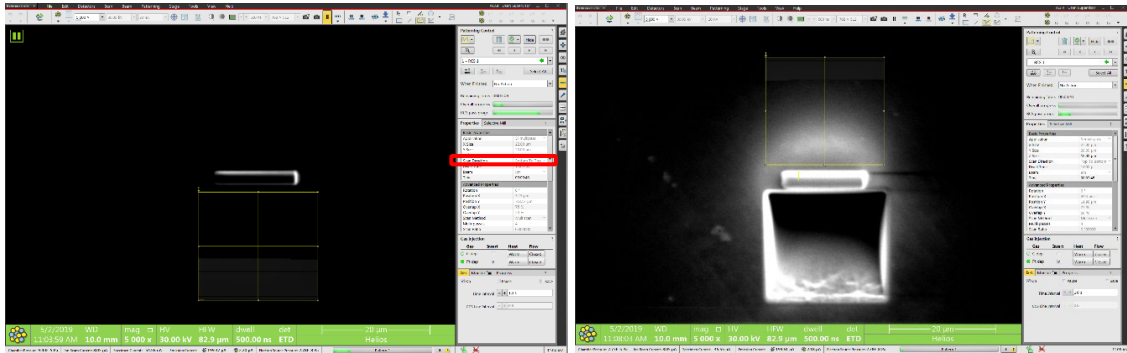
#### 4. Bulk milling:

- 1) Keep sample tilt at **52°**.
- 2) Use the  **Regular Cross Section** mode to define milling area below the Pt pad with a 1-3 um gap in between and the X slightly longer than Pt pad length, typical X: 18-20 um, Z: 8-12 um and the Y: 12-18 (typically **1.5-2 times the Z**), e.g. 20x20x10 um.



- 3) Choose **Bottom to Top** in Scan Direction for the milling box below the Pt pad
- 4) Select **Si-multipass** in Application as highlighted.
- 5) Choose typical **30 kV** and **7 nA** on ion beam setting to avoid over milling the Pt pad. Typically 9.3-21 nA can be used depending on material. For fast milling **20 nA** can be used. The rough milling time for each box should take 5-15 mins.
- 6) Adjust ion beam focus and stigma off the milling site if necessary and shift the beam back.
- 7) Click Pattern Start button as highlighted to start cutting.

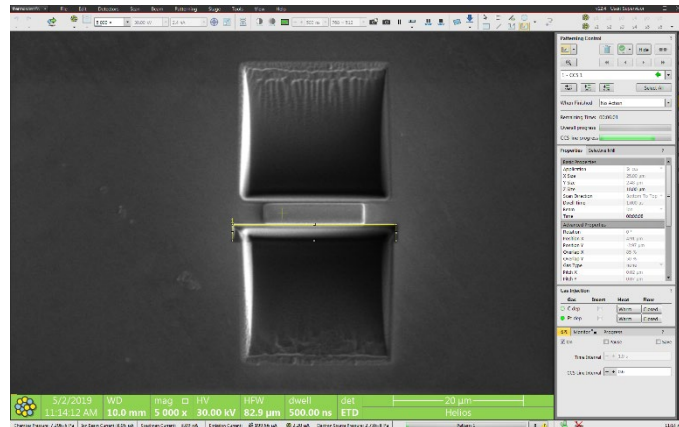
- 8) Bulk milling above the Pt pad: repeat steps 1) – 7) and choose **Top to Bottom** in Scan Direction.



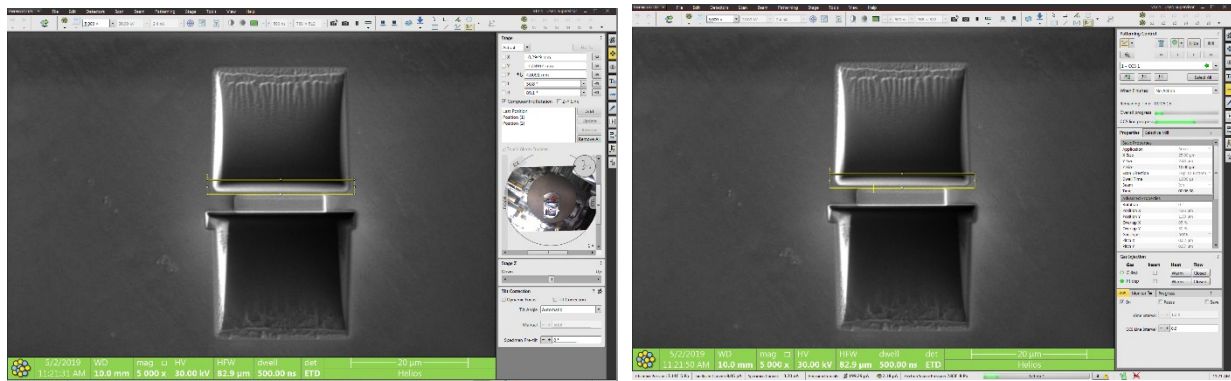
## 5. Intermediate milling

**Note:** this step is recommended to clean and correct the overcut from regular cross section milling so that the trench edge is perpendicular to the Pt pad edge.


- 1) Tilt sample **53 – 53.5°** (typically 1-1.5° over tilt from 52°, e.g. +1.2°) to clean the **bottom edge** of the Pt pad.
- 2) Choose **Cleaning Cross Section** mode and draw a rectangle inside the trench with a ~ 2 μm gap from Pt pad. Choose Z to be **50%** of regular cross section depth, e.g. 4-5 μm, e.g. 20 x **2.5** x 5 μm and start cleaning as shown below.

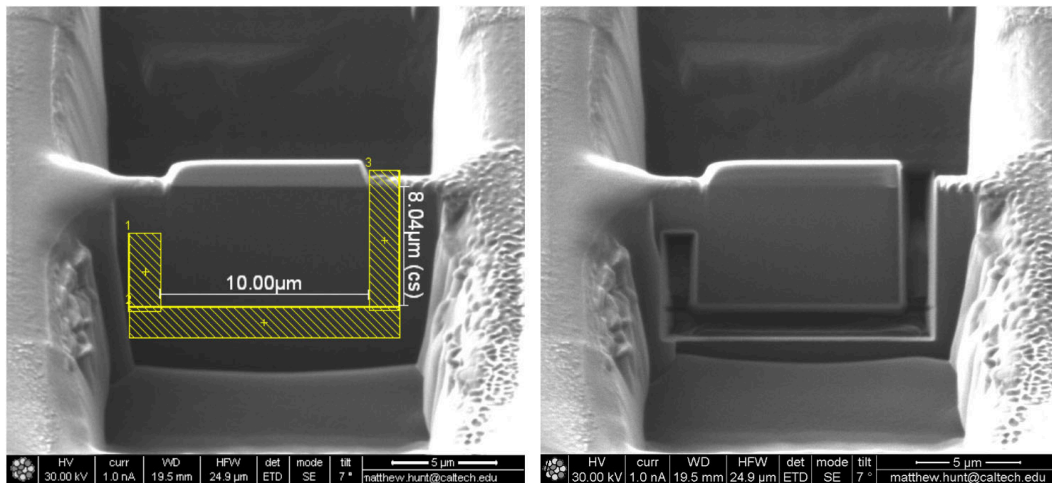


- 3) Choose typical **30 kV** and **2.5 nA** on ion beam setting. Can also choose 5 to 2 nA to avoid over milling the Pt pad.
- 4) Start milling on **bottom edge** of Pt pad. Typical milling time should be ~2-5 mins.
- 5) After finish, draw another **cleaning cross section** box **right underneath** Pt pad and start cleaning. The two step cleaning should avoid Pt layer damage. To save time, can just finish in one step.
- 6) Cleaning **top edge** of the Pt pad: tilt sample **50.8°** (-1.2° from 52°) and choose **Cleaning Cross Section** mode. Draw a rectangle of 25 x **2.5** (y) x 5 μm and start cleaning. The lamella thickness should be **800nm – 1.5 μm** after cleaning steps.



## 6. Tilt the sample back to 0° for U-Cut. (or 7°)

- 1) Center the sample in both electron and ion windows. May need to adjust Z in ion image window.
- 2) Adjust ion focus and stigma by shifting the ion beam to a nearby area.
- 3) In **ion image** window rotate ion beam **180°** so the ion image is the mirror of electron image. Keep in mind that after beam rotation, the Easylift probe will approach the sample from top right corner of the window.
- 4) Define three **U cut** boxes with typical z size of typical **1-3 um** and set as **parallel job**  in Pattern Control panel as shown below. Try not to put the bottom cutting box too deep in the trench to avoid Pt re-deposition and lamella attaching to the substrate.
- 5) Choose FIB scan direction: **Left to right** for side cuts and **Bottom to top** for bottom cut.
- 6) Ion beam setting: generally **1-3 nA** at 30 kV is recommended.
- 7) Refresh ion image window briefly (F6) to check for any drifts and start cutting.



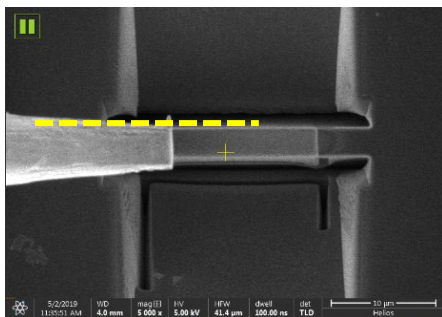
## 8) Check lamella clearance from substrate before lift-out:

- a) Tilt stage to **30°** and check in electron image quad if a clean cut was made,
- b) Tilt back to **0°** and make a compucentric **180°** rotation, and tilt back to **52°** again to check the reverse side. Repetitive bottom cut may be needed sometimes. Make sure the lamella is only attached to substrate through the micro-bridge before lift-out.

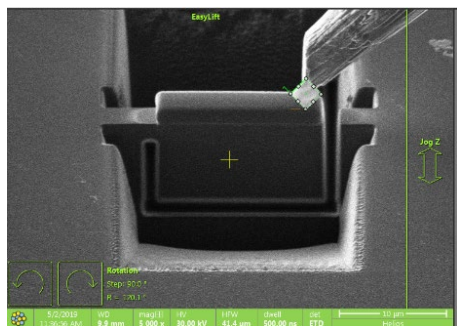


## 7. Sample lift-out:

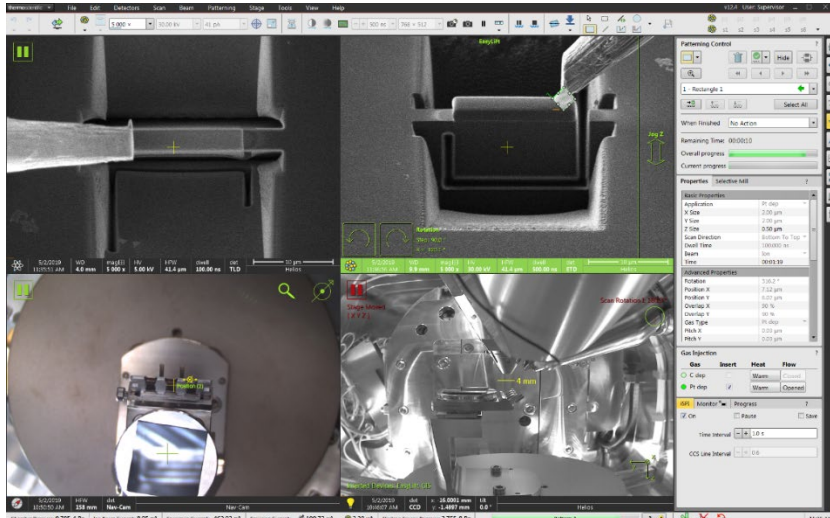
- 1) **Tilt stage to 0°**
- 2) Center the sample in ion image window. The micro-bridge should be on the left side of the image.
- 9) Adjust SEM image resolution to 768x512. Center the sample in electron image using beam shift, **do not** move the stage in X and Y. The micro-bridge should be on the **right** side of the image. If not, perform **180°** eucentric tilt.
- 10) In **Pattern Control > Gas Injection**, insert **Pt GIS** needle first before attaching EasyLift probe to the sample. Make sure the GIS is warmed.
- 11) In **Probe Control** panel set the EasyLift probe speed to **10um/s** and click **Insert** button. The probe should be at **Park** position.
- 12) Check if the probe is clean. Cutting to sharp the probe may be needed before attaching to lamella. **User line cut to save probe material**. EasyLift probe may need calibration if no use after long time.
- 13) Activate the **electron** image window, approach the probe slowly at **10um/s** and **align** the probe with the sample in and align the top edge of the probe to the top edge of the lamella as shown below. The welding Pt pad will be deposited on the same side.



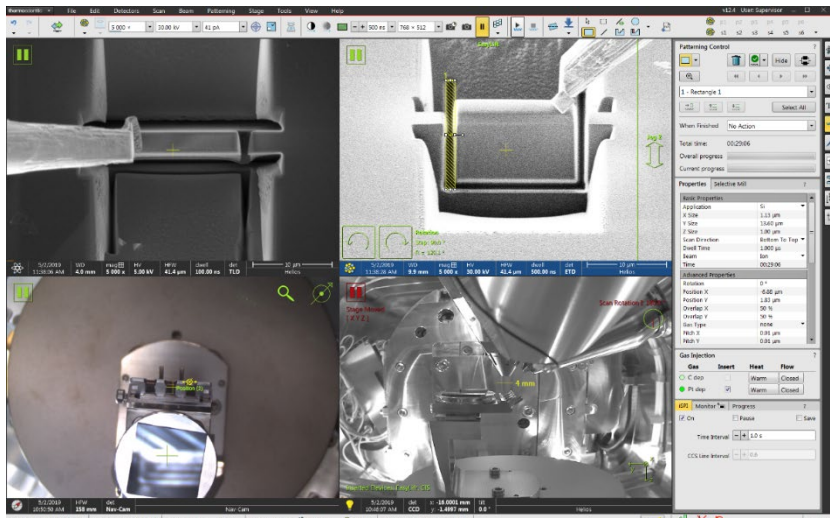
- 14) Activate **ion** image window, slow further the speed to **1um/s** to **0.5** or **0.1um/s** and approach the probe to the top right corner of the lamella as shown below. Then use the **Jog Z** in ion image window to drag further down to the corner. **Stop** if a sudden image contrast change is seen, indicating the probe is touching lamella. If contrast change is not noticed, try to tap the lamella slightly and watch if it shakes and pull back slightly.



- 15) Define the Pt welding pad at the top right corner of the sample as shown below in ion image window. Rotate the Pt pad **45°**. Typical Pt pad size: **2x2x0.5 um** or **0.1 to 0.2 um**. Ion beam setting: **30kV, 41pA**. Choose Pt dep in Application and open the Pt GIS flow. Wait to see the chamber pressure rises and start depositing.



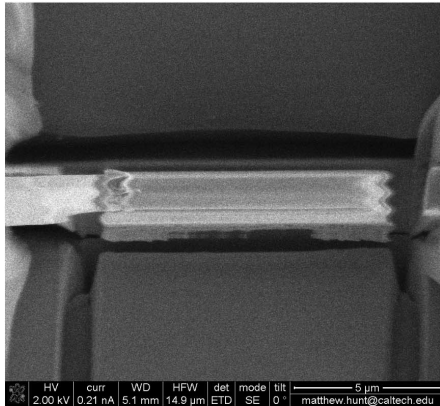
- 16) Meanwhile, if necessary and depending on the EasyLift probe shape, can try to weld the top part of the probe simultaneously using reduced imaging window in electron window. Typical e-beam setting: **2kV** and **0.21nA** and **1us** for dwell time.
- 17) Cut the micro-bridge (typical ion beam: **30kV, 41pA** or **1-3nA**): define a longer rectangle (for flag type mounting) close to the left side of lamella. Typical Z depth is 1-3 um to cut through the entire thickness. Make sure the cutting edge is straight and smooth for flag type mounting. To protect lamella and Pt pad readjusting beam focus and stigma may be needed before cutting.



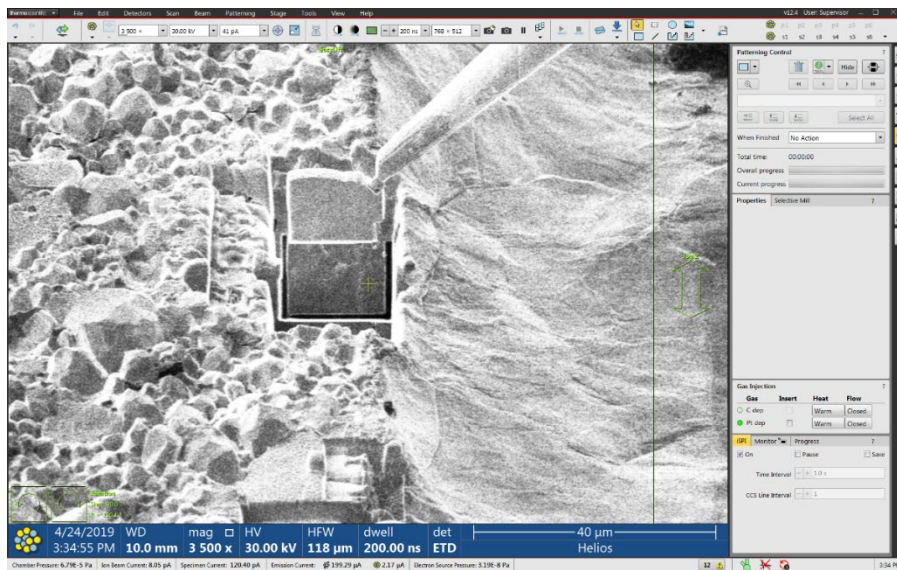
- 18) Test if the lamella is attached only to the EasyLift probe: tap slightly the FIB table and watch in electron image window if the vibration causes the probe and lamella to wiggle on the screen together as shown below. If not, try milling again on three sides.



**Warning:** **Never** do the lift out before confirming that the lamella is only attached to the probe.



19) Lift out: click and hold on **Jog Z** in ion image window and slowly pull the lamella **just above** the substrate plane as shown below:

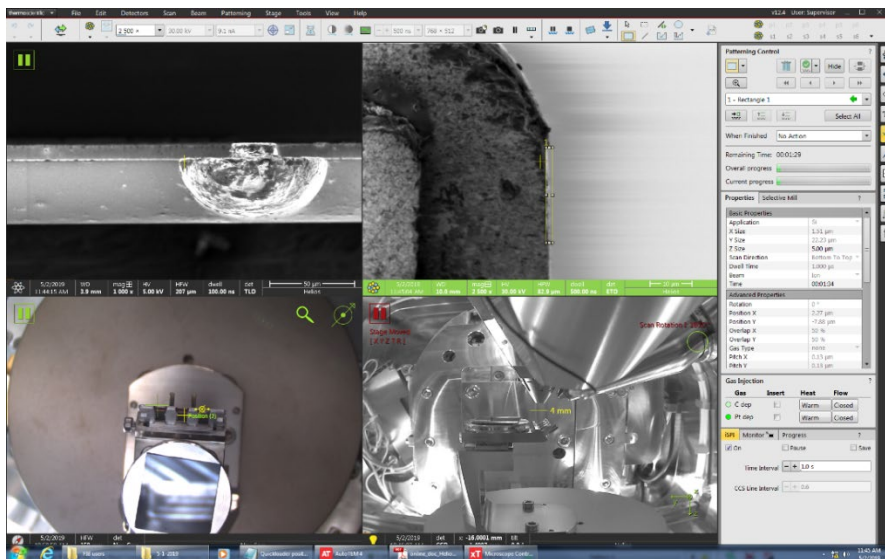


20) **Retract** Pt GIS needle.

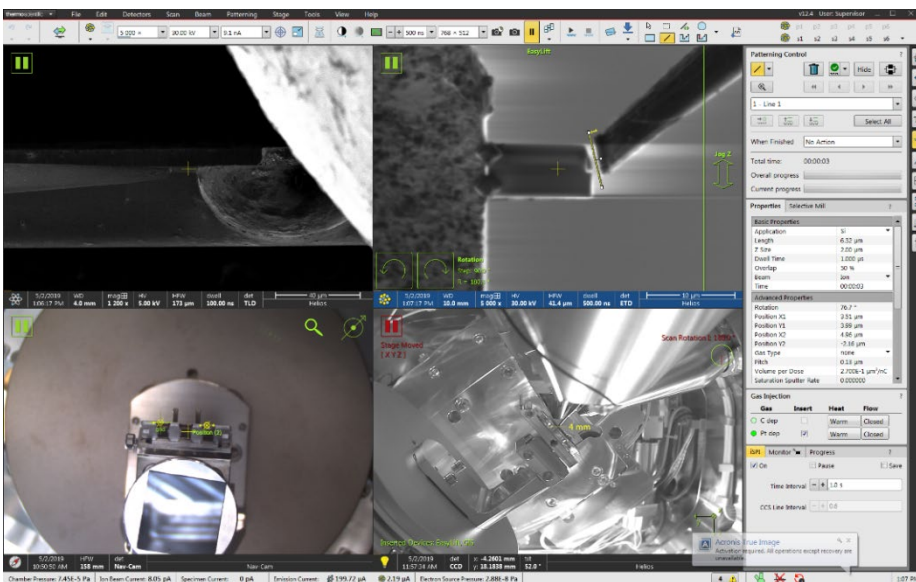
21) **Fully retract** the EasyLift probe with the lamella.

## 8. Lamella side mounting on grid post

1) Define grid position: rotate the stage **180°** and tilt to **52°**. The grids should appear in the side view in electron image as shown below:



- 2) Cleaning mounting area on the grid post as shown in above ion image window. Use rectangle mode and typical Z of **5  $\mu\text{m}$** .
- 3) Approach the EasyLift probe slowly at **< 5  $\mu\text{m/s}$**  and align the probe with sample in both electron and ion image windows as show below. Weld the lamella onto the grid post using two square Pt pad and use the **line** cut mode to separate the probe. Try to cut the probe on sample side to save probe material.



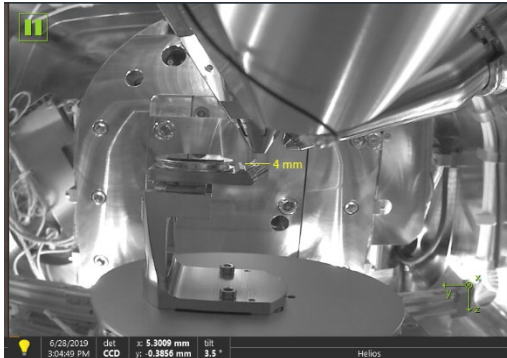
- 4) Retract both Pt GIS needle and EasyLift probe.

## 9. Lamella top mounting on grid post

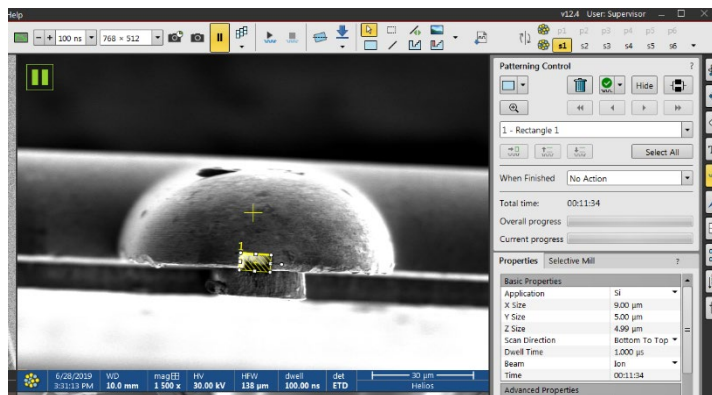
**Skip** this step for side lamella mounting. The top mounting is highly recommended to reduce the lamella bending or twisting during thinning. It is also a preferred orientation for EDS in TEM system.

- 1) Adjust eucentric height alignment on intended grid post.

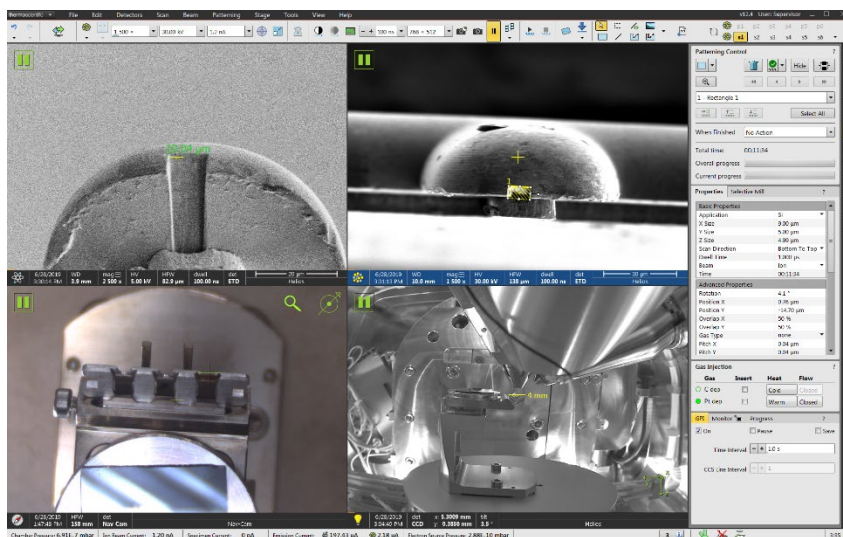
- 2) Rotate the stage **180°** and tilt the stage to **0°**. The grid (**38°** tilted from sample holder) post should be parallel to ion beam and on the right side in CCD window as shown below.



- 3) Align the post with ion beam carefully by adjusting tilt angle so the flat surface is parallel to the beam.

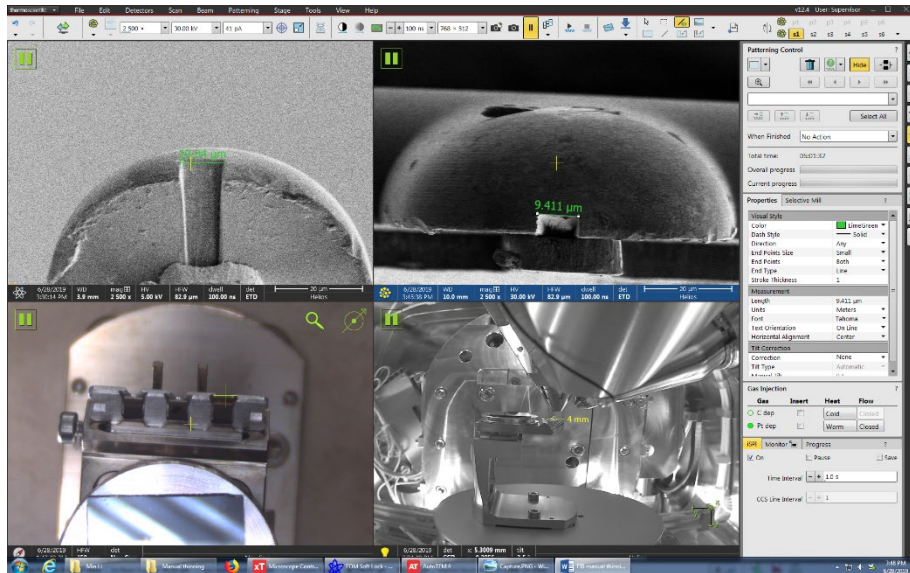


- 4) Cut a trench on the grid post top with the width slightly smaller than lamella width, e.g. choose X size: 9 µm for 12 µm wide lamella, Y: 5 µm and depth (Z) of 5 µm. Ion beam: 30kV and 1.2nA. Typical mill time: 10 min.

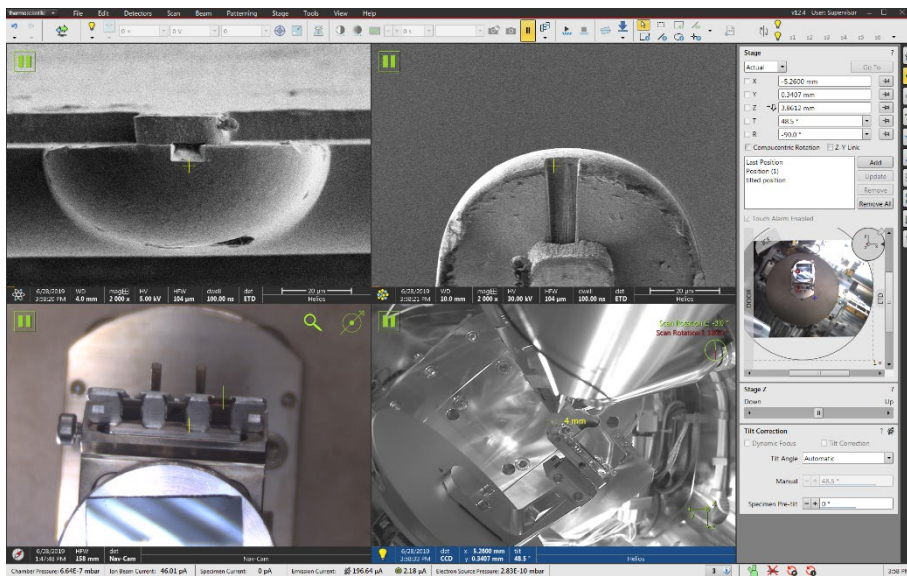




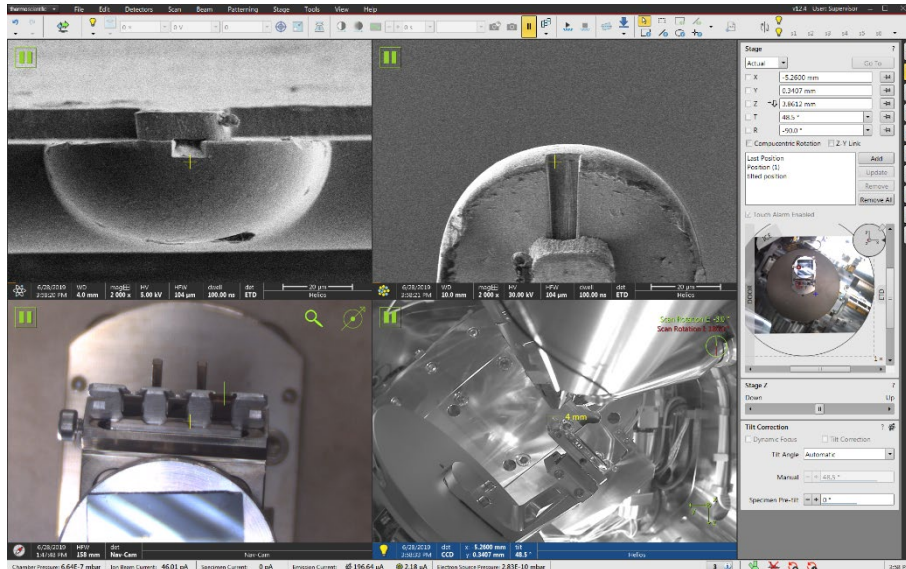
- 5) Check the trench in both electron and ion image windows. Keep milling if needed. The final trench is slightly larger than set value, in this case 9.4  $\mu\text{m}$  vs 9  $\mu\text{m}$  (set value).



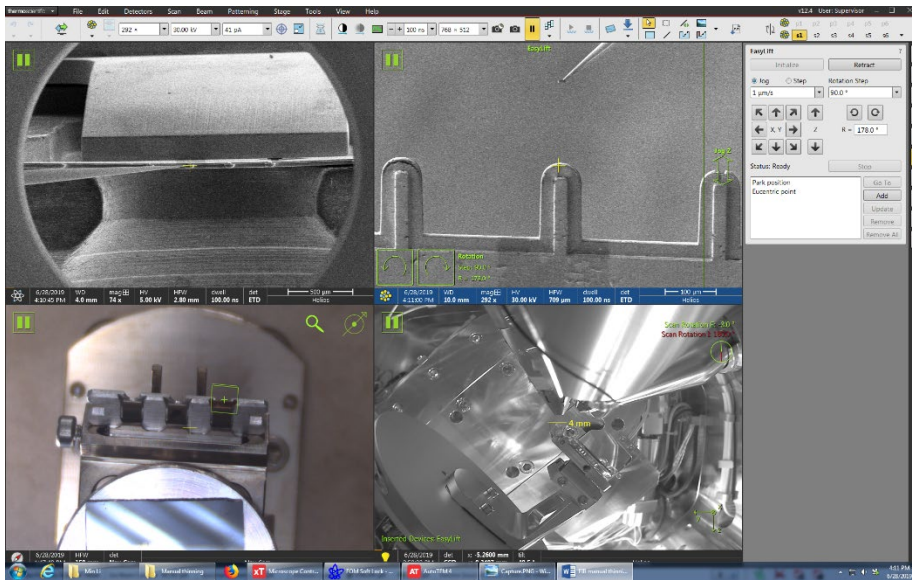
- 6) Rotate the stage **180°** back and tilt the stage to **52°**. The grid post should be on the left side of sample holder in CCD window as shown below:



- 7) Slightly adjust the tilt to align the trench surface with electron image (see below). Click to activate ion image window and change the **Scan Rotation** for to **180°** so the post is pointing upward in ion image window as shown below:



- 8) Insert Pt GIS needle and keep the gas warm before EasyLift probe approaching.  
 9) **Zoom all the way out** in both electron and ion image windows and insert the EasyLift probe to **Park** position:

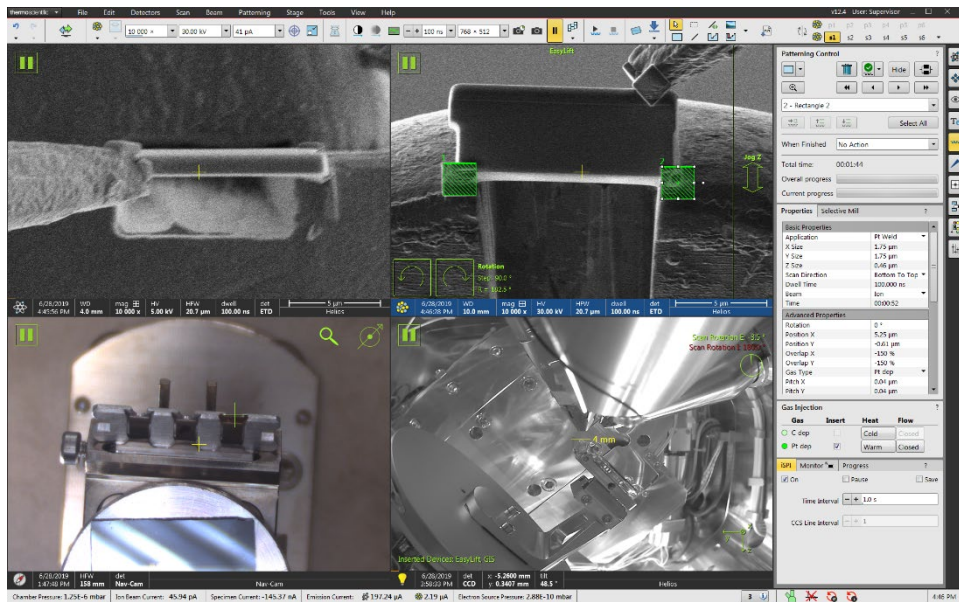


- 10) Activate ion image window, slowly approach the EasyLift probe with the lamella attached to the trench on top of the post. Align the lamella in both electron and ion image windows before touching the post. Align the lamella top edge with the trench edge as highlighted by the dashed line below. Stop probe if lamella starts to move laterally, indicating it is touching the post. Move up a slight distance.



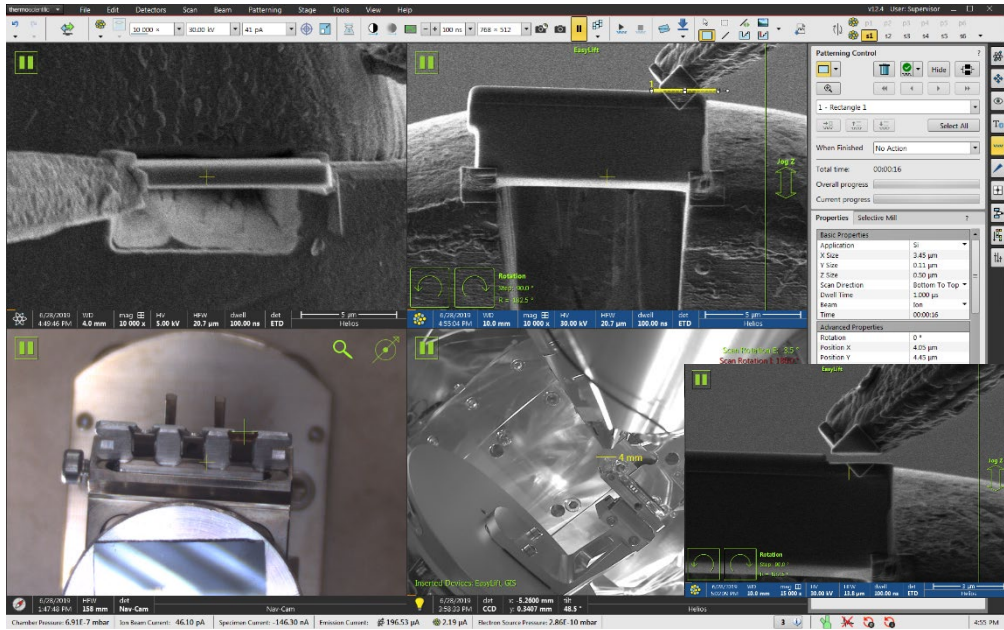


- 11) Weld the lamella by applying two **Parallel** small square pad Pt deposition ( $Z \sim 500$  nm) on two corners in deposition. Slice the probe after welding. The ion beam setting: **30kV** and **10-41pA**. Typical time  $\sim 2$  min.

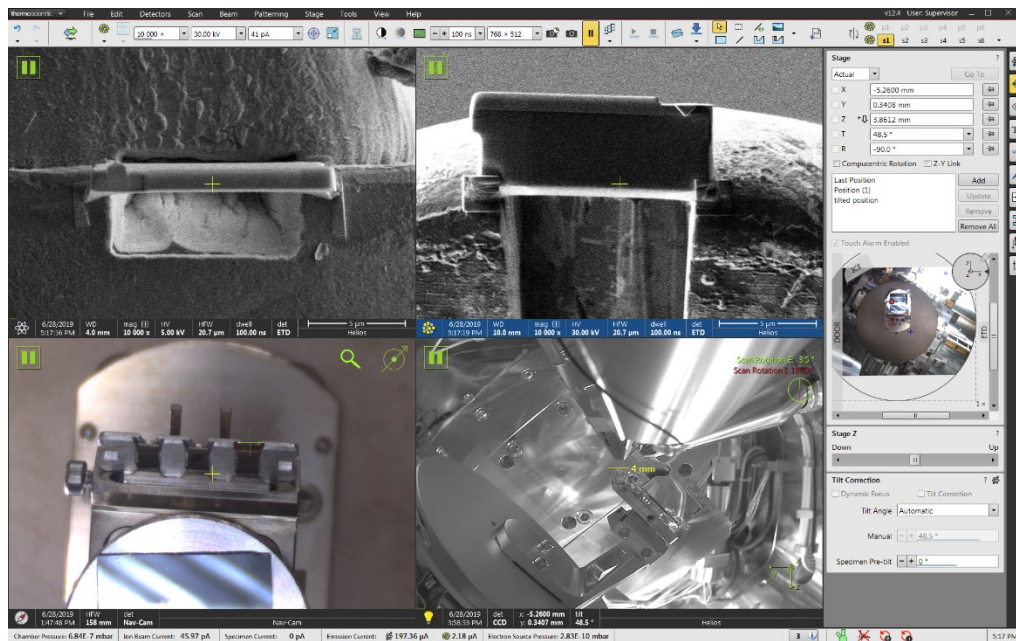


- 12) Check in both electron and ion image window to make sure the lamella was welded with Pa pads onto the post. Slice the probe along the top edge of the lamella and slowly retract the probe out just above post.



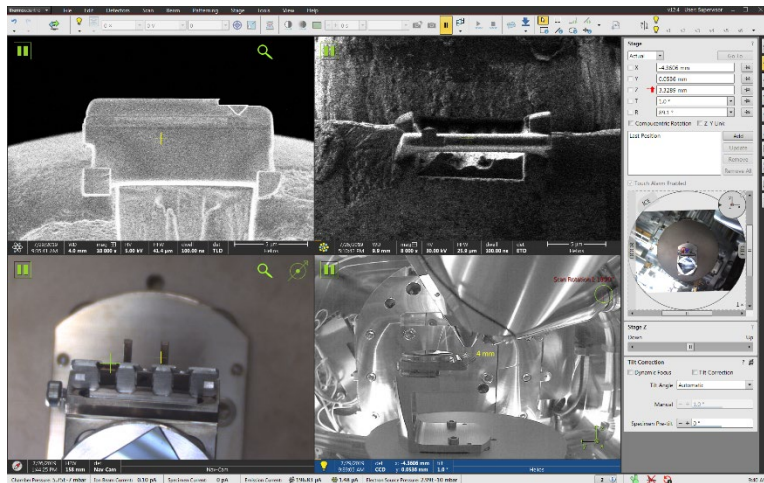


- 13) Retract the Pt GIS needle and **Cold** the gas.
- 14) Clean probe tip for next user.
- 15) Fully retract the probe.
- 16) Check to make sure the lamella was mounted safely on top of the post as shown below.



## 10. Lamella thinning

- 1) Tilt the stage back to **0°** and rotate the stage **180°** so the grid holder is on the **right side** in CCD window as shown below. Readjust eucentric height may be needed.



- 2) Adjust ion beam focus and stigma quickly on a nearby area using reduced area view before milling.
- 3) **30 kV Cleaning Cross-Sections**, tilt **1-2°** away from ion beam ( $\sim 0^\circ$  for tilted grid holder and  $\sim 52^\circ$  for vertical grid holder):
  - a) At relatively high current (e.g. **0.26-0.79 nA**), set **Z depth** just enough to mill all the way through the bottom of the lamella on both sides (e.g.  $1.5^\circ$  for front side,  $-1.5^\circ$  for back side, or for vertical grid holder  $53.5^\circ$  for front side,  $50.5^\circ$  for back side). Aim for **500-1000 nm** thickness when done with this step.
  - b) Select scan direction: Bottom to Top at  $+1.5^\circ$  tilt and Top to Bottom at  $-1.5^\circ$ .
  - c) At relatively low current (e.g. **0.23** or **0.43 nA**), set **slightly lower Z depth** (e.g.  $0.25 - 0.50 \mu\text{m}$  less) to leave some material at the bottom of the lamella on both sides, which will add mechanical stability to the lamella. Aim for **150-250 nm** thickness when done with this step.
  - d) At even low current (e.g. **80 pA**), set **slightly lower Z depth** (e.g.  $0.3 \mu\text{m}$  less). Aim for **80-100 nm** thickness when done with this step. Mill until uniform contrast in electron image.

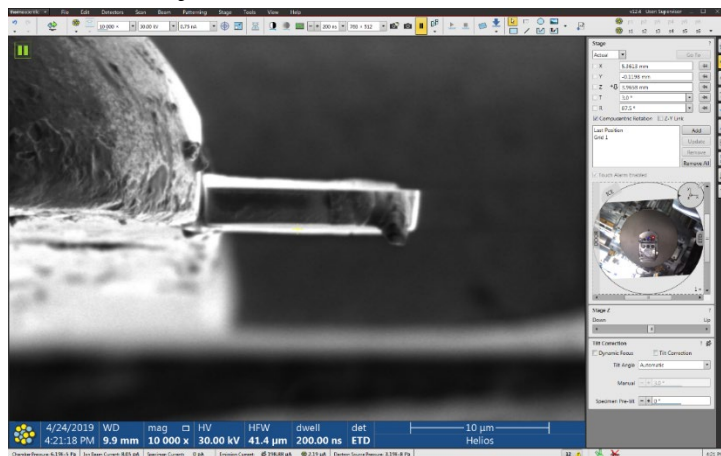


4) **Low kV thinning:**

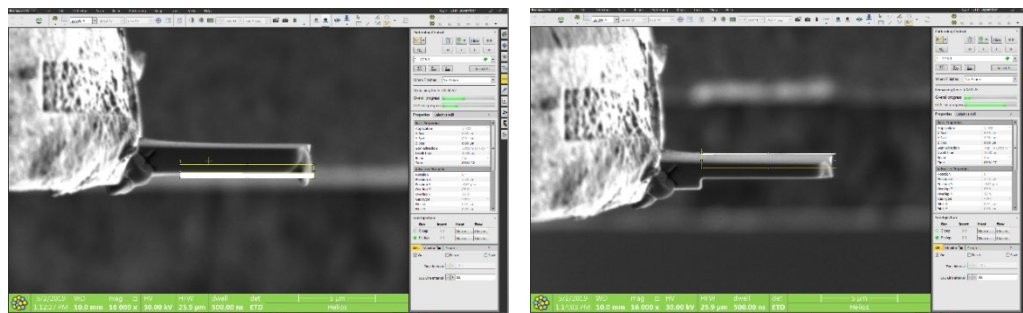
- 5 kV**, 70-100pA, tilt **4-5°** away from parallel (e.g. 56 or 57°:
- At ~42 pA, get good focus and fix astigmatism (try alignment rectangle technique on full frame). Place milling rectangle over cross-section face. Watch live in SEM quad as you mill, noting thickness of Pt protection layer. Allow Pt to reduce to ~half the thickness it had before you started this step, then manually stop it.
- Tilt to 0°, compucentrally rotate stage 180°, tilt back to 56 or 57° to see other side of lamella; you always want to be looking with the SEM at the side of lamella you are milling during 8 kV steps.
- Refocus and fix astigmatism (try alignment rectangle technique). Place rectangle over entire cross-section face. Watch live in SEM quad as you mill, noting thickness of Pt protection layer. Stop pattern when Pt is nearly gone, but some still remains. Final lamella thickness should now be 10-100 nm.

**11. For side mount lamella:**

- Align sample with ion beam: adjust stage tilt so that the white edges on both sides of the lamella (in dark) appears symmetric in ion image window. Typical 1~ 3° tilting might be needed for adjustment.



- Large mass cutting: choose **Cleaning Cross Section** mode and define cutting box on both sides of the sample to start cutting alternately. Typical beam settings: **30kV** and **0.75 - 1nA**. Tilt the sample **1.2° up** for bottom side cutting and tilt 1.2° (**-1.2 °**) to cut the upper

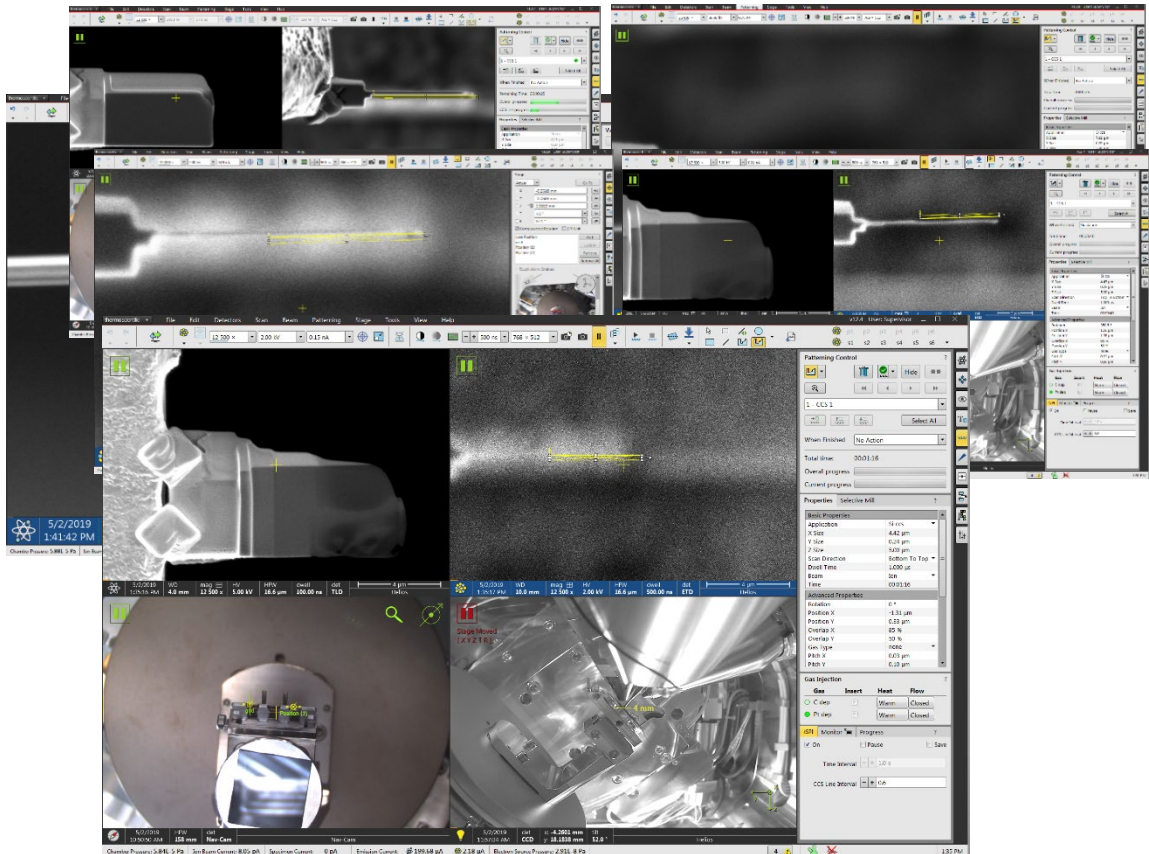




side. Choose **Bottom to Top** in **Scan Direction** for bottom side cutting and **Top to Bottom** to cut the upper side.

- 3) Initial thinning: choose **Cleaning Cross Section** mode and define cutting boxes on both sides of the sample to start thinning alternately. Typical beam settings: **30kV** and **0.75nA**. Tilt the sample **1.2° up** for bottom side cutting and tilt **1.2° (-1.2 °)** to cut the upper side.
- 4) **5kV thinning**: use **5kV** and **0.21nA** for ion beam. Choose **Cleaning Cross Section** mode and define cutting boxes on both sides of the sample to start thinning alternately. Typical Tilt the sample **1.0 ° up** for bottom side cutting and tilt **1.0 ° (-1.0 °)** down to cut the upper side.
- 5) **2kV cleaning**: use **2kV** and **0.15nA** for ion beam. Choose **Cleaning Cross Section** mode and define cutting boxes on both sides of the sample to start thinning alternately. Typical Tilt the sample **1.0 ° up** for bottom side cutting and tilt **1.0 ° (-1.0 °)** down to cut the upper side.

## 12. Thickness measurement



- 1) Align the sample with electron beam by tilting stage to **~52°**. The dark region in the middle should be the sample cross section as shown below:

- 5) The side view of final sample in electron image window: the thinnest part should be close to the interface between protection layer and sample. The stepped cutting should produce a step like cross section with the area close to mounting area the thickest.

