Helios 05 UC Dual Beam FIB-SEM Standard Operating Procedure

Introduction

Instrument features:

- TEM Lamella preparation
- Site specific cross sectional imaging, 3D Slice and View
- HR imaging, Low kV HR imaging, SEM/STEM imaging, Ion beam imaging
- Nano-fabrication and Nano-patterning
- EDS- Area/Line/Mapping

Location :

SAIF/CRNTS IIT Bombay
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Facility Manager:

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If you encounter any problems with this facility, please contact the staff member listed above immediately.

Notice: Please **follow** the **SOP** strictly to keep the facility under good condition. **No** explorations on program allowed unless approved by core manager.

Initial System Status Check

Warning: Before sample preparation and loading, user should check the system status following the steps listed below:

1) Check the supply of **Nitrogen Air (N2)** pressure gauges as shown in the below image and make sure the pressure reads **~ 100 psi**. Report immediately if the gauge reads **zero**. **Warning**: **Do not** adjust the regulators on top of those gauges.

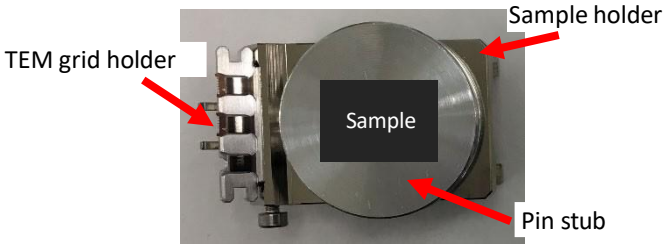
2) Sign into the **XT Microscope software**. The login ID and Password is Supervisor .The **chamber pressure** can be found on the **bottom left** corner of the window as highlighted below.

3) Check the **bottom right** corner of XT window above and make sure the chamber color is indicated in green. **DO NOT** proceed if the chamber color is in orange. This may indicate chamber leaking or outgassing. **Report** to the concerned authority immediately.
4) Check inside the CCD window (the bottom right quarter of above window) to make sure no samples are inside the chamber.

Sample preparation

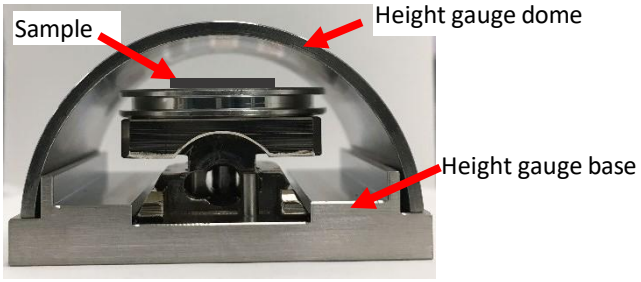
Note: the below steps are **ONLY** for sample transfer using the **Quick Loader**. For large sample loading we need to vent the main chamber.

1. **Always wear gloves** for vacuum sample preparation.
2. The sample for FIB should be completely **dried** before mounting.
3. The sample can be fixed using double sided carbon tape directly onto the pin stub as shown below:

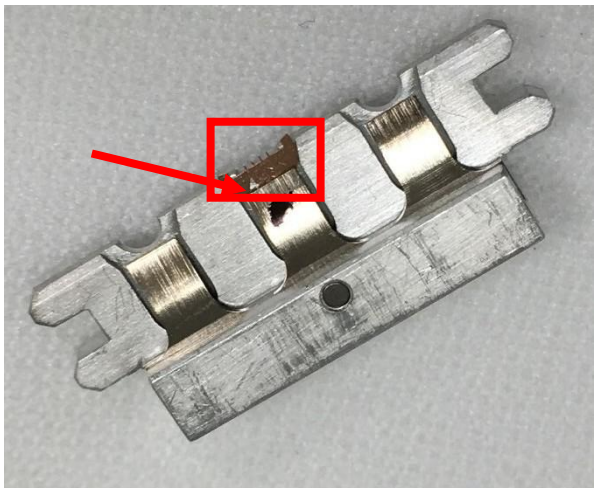


4. Sample height alignment: place the sample holder into the gauge as shown below and make sure the sample is **not touching** the inner wall of the height gauge dome.

Warning: misaligned/misplaced sample will hit the electron column and cause severe damage!



5. **Clean sample holder**: **blow off** loose particles on the sample surface (not on the grid) using the **blower**.
6. For TEM users, make sure the grid was mounted correctly on the holder as shown below.

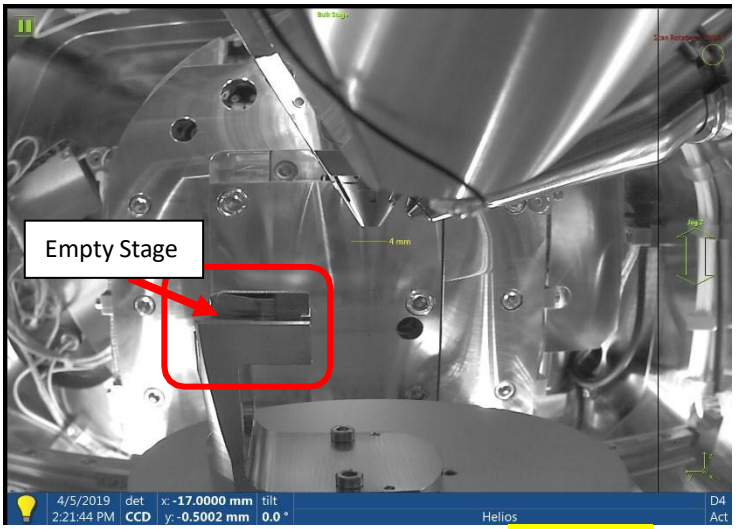


Sample Loading through Quick Loader

Note: if not familiar with sample loading steps, read the entire section before start.

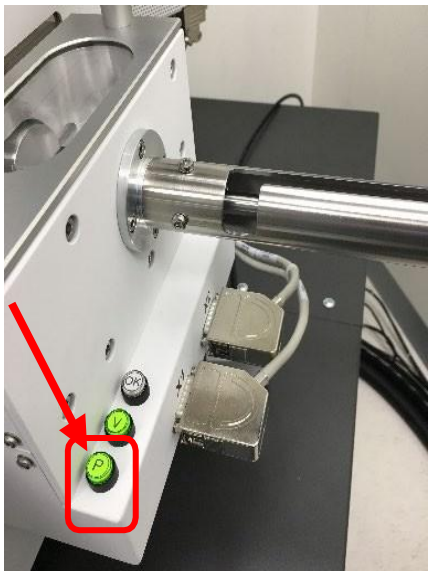
1. Click  button on **XT** window tool bar to open the **Sample Exchange** window below:

2. Click inside **CCD** window (bottom right window) to unpause/activate **CCD** view inside main chamber.
3. Click top menu **View> Large Image Window** to amplify the **CCD** window

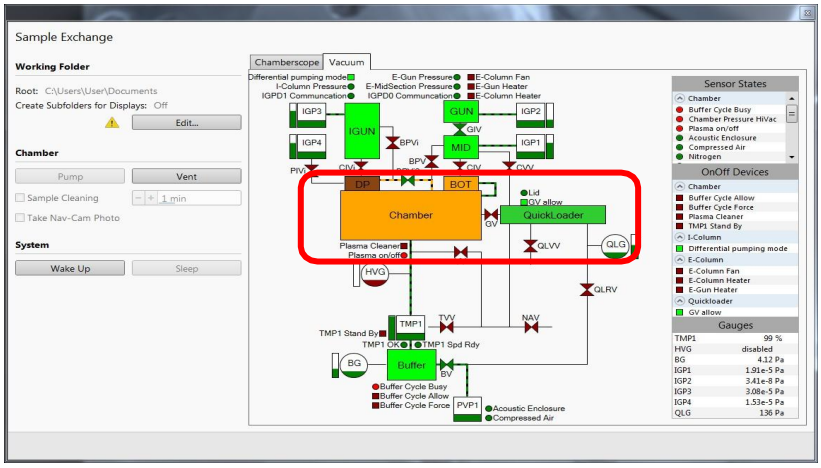


onto the computer monitor on the right side. **Make sure** there is no sample holder on the stage.

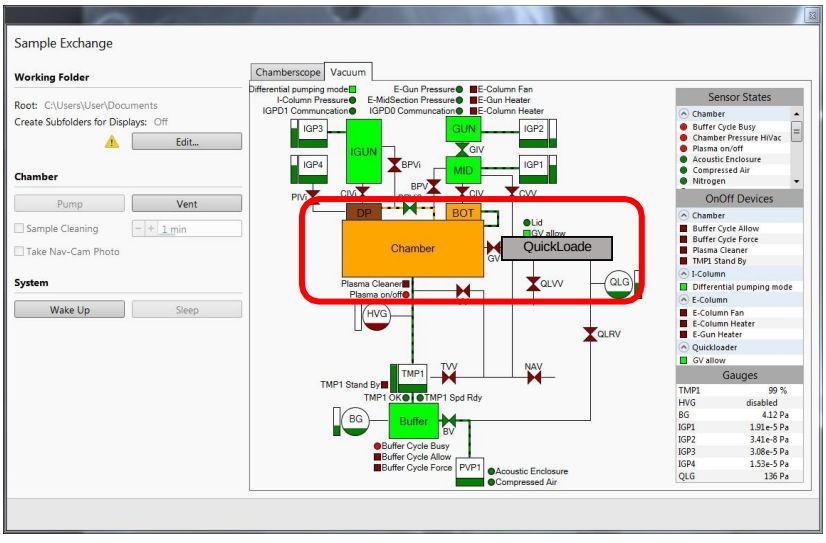
4. **Put on gloves**. Press and hold  button for one second on **Quick Loader** as shown below:



5. Watch the **Sample Exchange** window till the **Chamber** color changes to **orange** and **Quick Loader** to **green** as shown below:



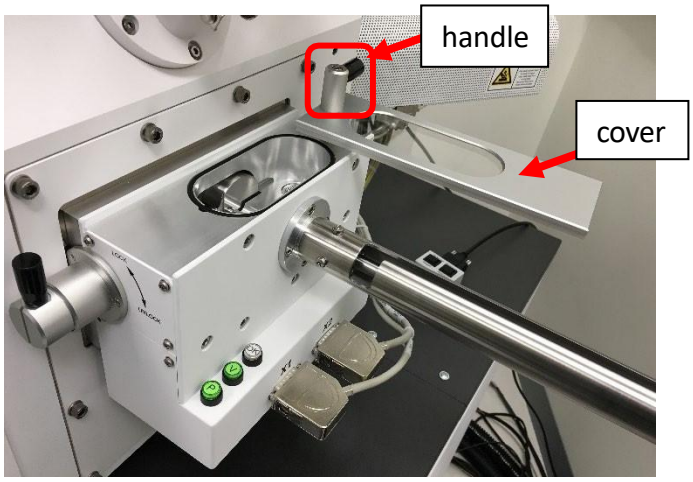
6. As soon as the **Quick Loader** turns into **green** while **Chamber** stays in **orange**, press and hold  button for one second on **Quick Loader** and wait to hear an air sucking sound, indicating the **Quick Loader** is being vented.
7. Watch the Quick Loader changing into **dark grey** as shown below, indicating **Quick Loader** venting is finished.



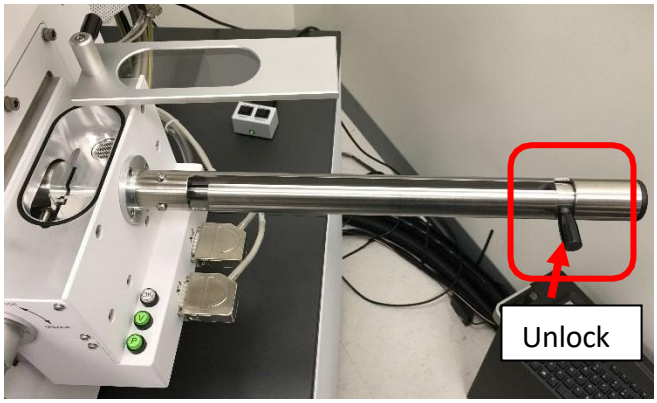
8. Grab the handle on top of the **Quick Loader** and **SLOWLY** lift the cover upward

and turn it **90 degrees** to expose loading area as shown below.

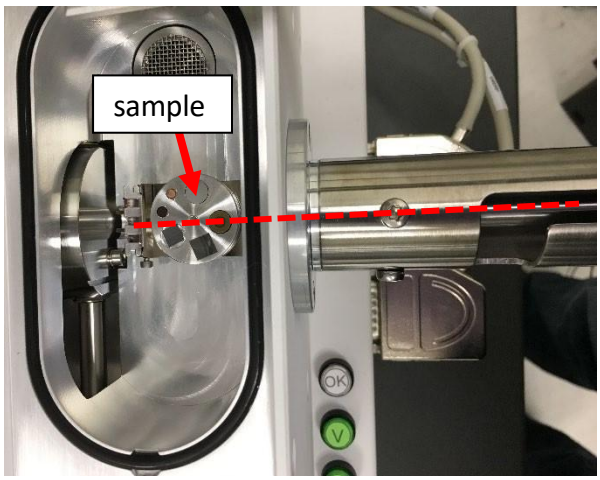
Warning: **Never force to break the vacuum** if the **Quick Loader** is not fully vented.



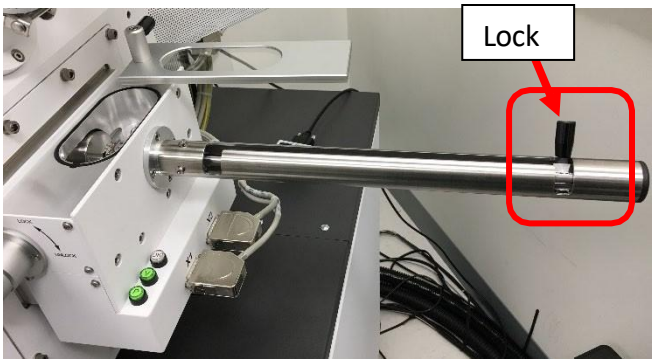
9. Pull the handle on the transfer rod downward to **Unlock** position as shown below:



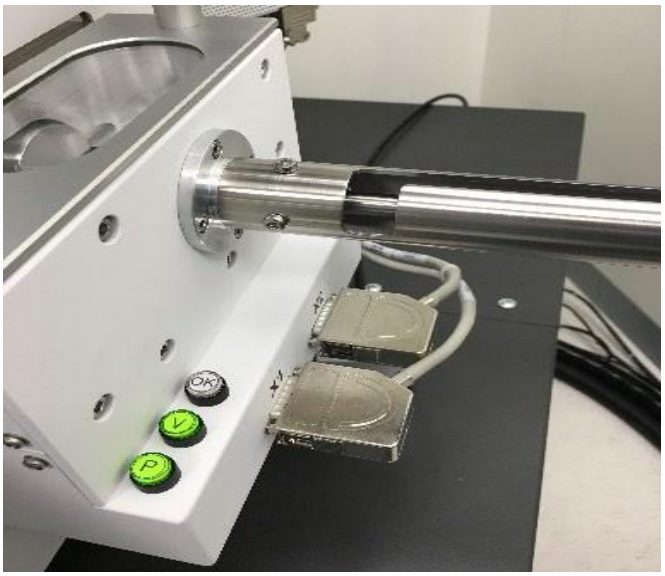
10. Pull the rod backward and meanwhile place the sample holder inside **Quick Loader** and align with the transfer rod as shown below:



11. **Left hand** holds and pushes the sample holder into the transfer rod meanwhile **right hand SLOWLY** turning the **Quick Loader** handle upward to the **Lock** position as shown below. Never force the handle to break parts.



12. Close the cover. Check if both the  and  buttons are now green.

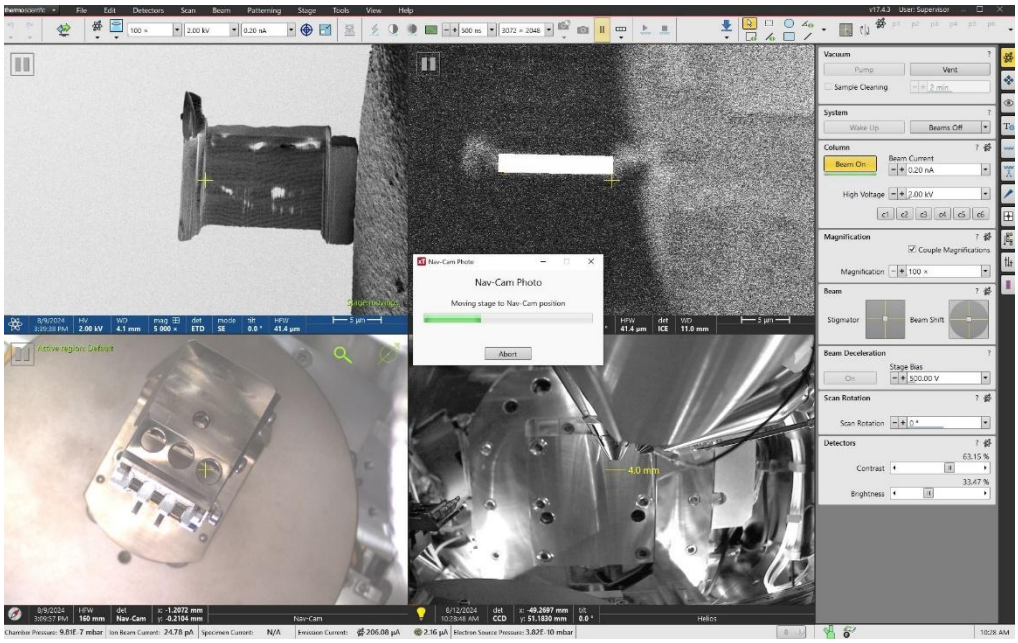


FIB-SEM OPERATION

The FIB-SEM utilizes two beams, an Ion beam (IB) and an Electron beam (EB), oriented at a 52-degree angle between them. The operation begins as below

Common Steps:

1. Check the system is ok and working.
2. Prepare the sample for loading to SEM chamber. Make sure the screw of the holder is well-tightened.
3. Select the correct sample holder.
4. Vent the loader chamber and load the sample clearly.
5. Take the Nav-Cam snapshot.



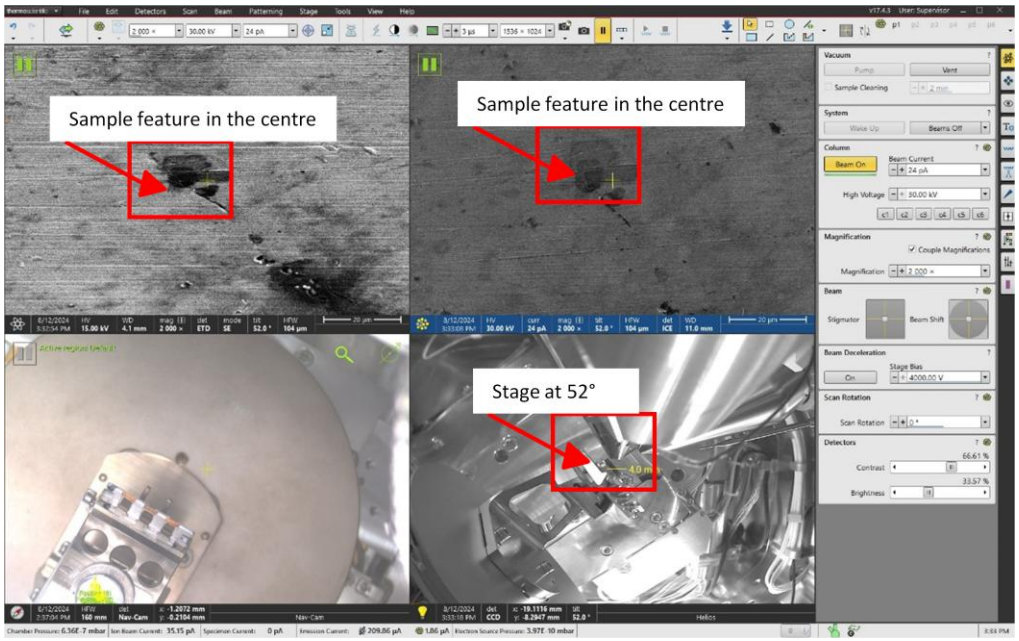
6. Focus on the sample (stigmator correction and other necessary steps), and then do the Z-link i.e. Couple the Z-axis and maintain it at 4mm height. If beam shift was there then reset the Beam shift for both I-Beam and E-Beam.

7. Now system is ready for the Eucentric height.

Procedure for Setting up the Eucentric Height:

1. Put Z- 4.0 mm then enter the key.
2. Select any mark/feature on the sample for our reference and make sure the Z-link is active.
3. Tilt the stage by increments of 10 degrees and adjust the Z-height accordingly.

4. After reaching 52 degrees, make sure it matches the mark/feature in the I-Beam/E-Beam window.
5. Go to 0-degree tilt by Control+E. for 52 degree by Control+I.
6. Confirm the Eucentric height setup properly.
7. Then do the Coincidence point adjustment. This is done by first double-clicking the feature in the I-Beam column window and then bring the same feature in the centre of the E-beam column window by moving the beam shift in the X, Y direction.



8. If it is OK, then the system is ready for further operations. If not, then do it again and make sure it is set up properly.

TEM Lamella Preparation

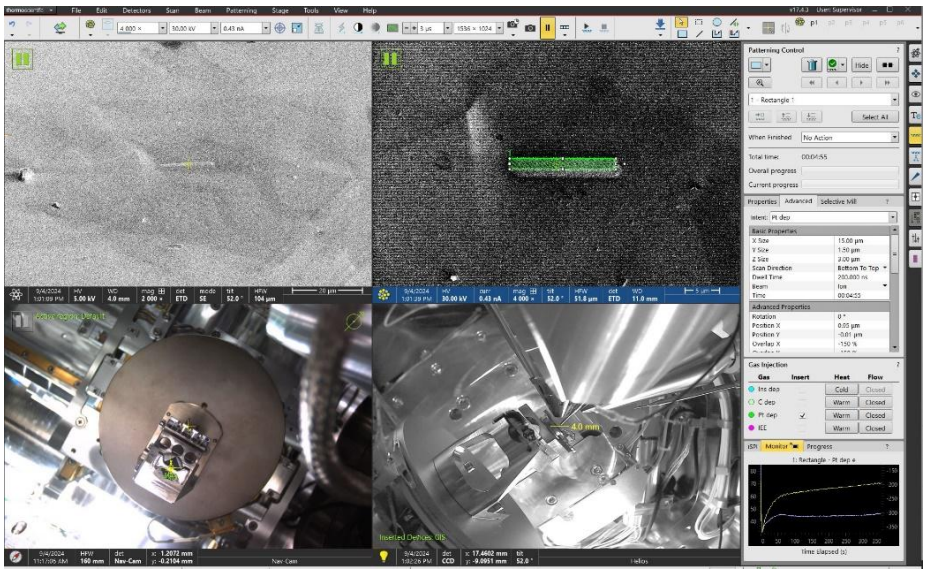
This detailed procedure outlines the step-by-step protocol for preparing and thinning a lamella using a Focused Ion Beam Scanning Electron Microscope (FIB-SEM). Screenshots (ss) should be taken at critical stages for documentation and analysis. Adjustments should be made according to the specific requirements of the sample and experimental objectives.

Platinum deposition:

This deposition process can be facilitated using a Gas Injection System (GIS), which should be inserted before the deposition begins. Remove the Gas Injection System (GIS) once the process is completed. *It is essential to ensure proper focusing and adjustment of the working distance to achieve the desired results on the deposited layer*

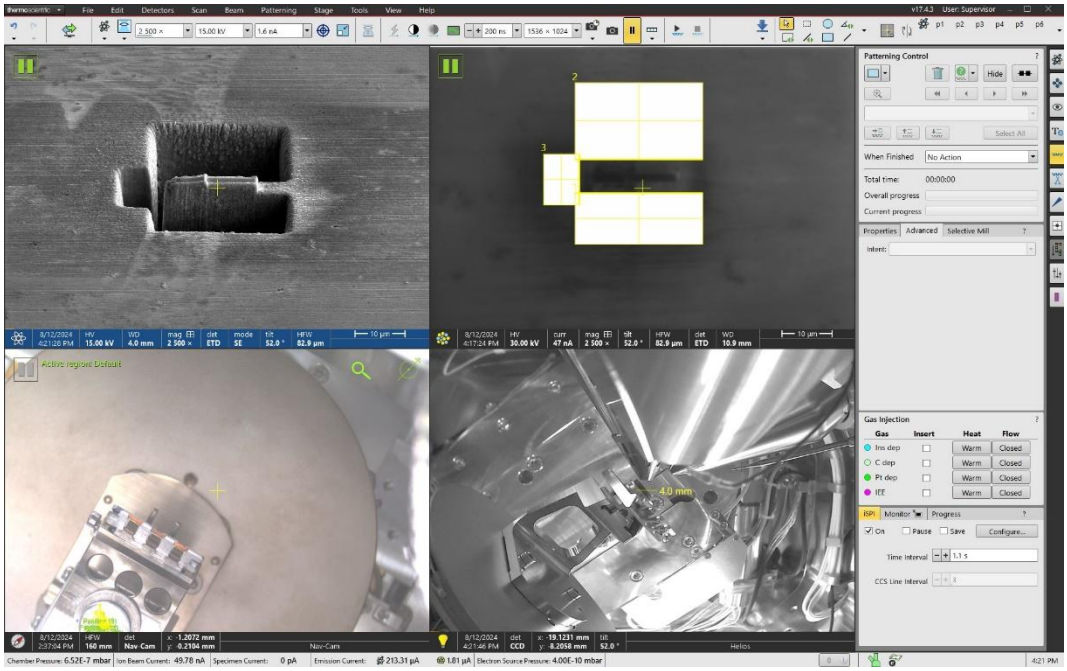
E-Beam and I-Beam Deposition procedure:

1. For E-Beam deposition, Sample tilt will be at 0-degree. Deposit e-beam platinum (approx. 15x2x2 microns).It is necessary when we want to preserve the sample surface feature from ion/electron damage. Draw the normal pattern and select the application as platinum deposition. *(Make sure drawn pattern turned into green one not the yellow one).* Set the x,y, and z parameters for deposition. Check one time pattern and its corresponding position on sample is correct. Then insert the GIS and do the deposition. **Remove the GIS needle after the deposition.**
2. For I-Beam deposition, Sample tilt will be at 52-degree. It is necessary when we want to preserve the sample surface feature from ion/electron damage. Draw the normal pattern and select the platinum deposition. *(Make sure drawn pattern turned into green one not the yellow one).* Set the x,y, and z parameters for deposition. Check one time pattern and its corresponding position on sample is correct. Then insert the GIS and do the deposition. **Remove the GIS needle after the deposition.**



RCS:

1. Select the 24 pA (System will be at 30KV till last cleaning procedure of the lamella preparation) for I-Beam to focus on the sample. If required do the stigmator correction.
2. Put the standard geometry of Regular Cross-Section (RCS) to do the rough milling to extract the lamella. *Select RCS in the menu and position the boxes away from the platinum deposit area to prevent damage. Focus, take screenshots, adjust, and start the RCS. Check for depth in e-beam and repeat if necessary (I = 47nA).*
3. Select the current (High current can be used here) according to the material *(In the default system, depth is according to the standard Si sample. For the different materials, the milling rate will differ. Adjust the Length (x), width(y), and Depth(z) with proper scaling factor with respect to the Si material)*



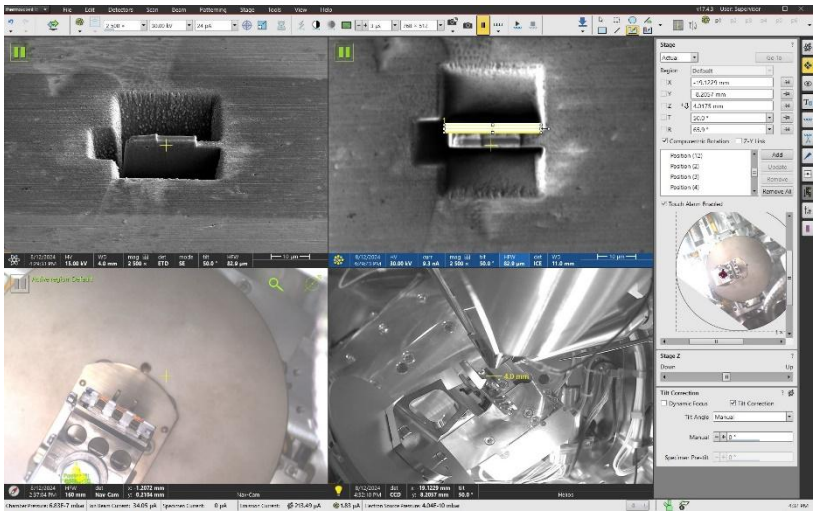
CCS Processing and undercut:

After the RCS, go to the cleaning cross-section (CCS) geometry (Select the current accordingly). Place it correctly and remove the burr/unwanted particle.

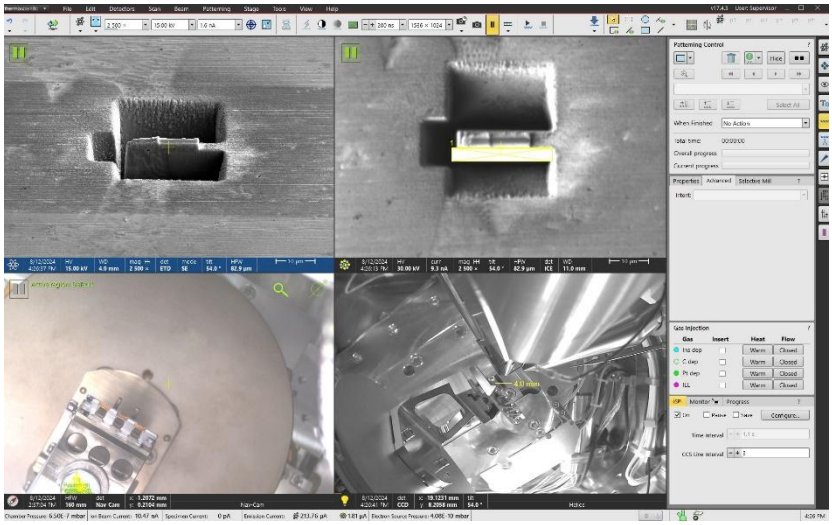
Make sure the RCS followed by CCS gives us a clean and smooth shape/geometry for Lamella.

If possible, do the little furthermore coarse thinning of lamella and then make sure that lamella is ready for undercutting.

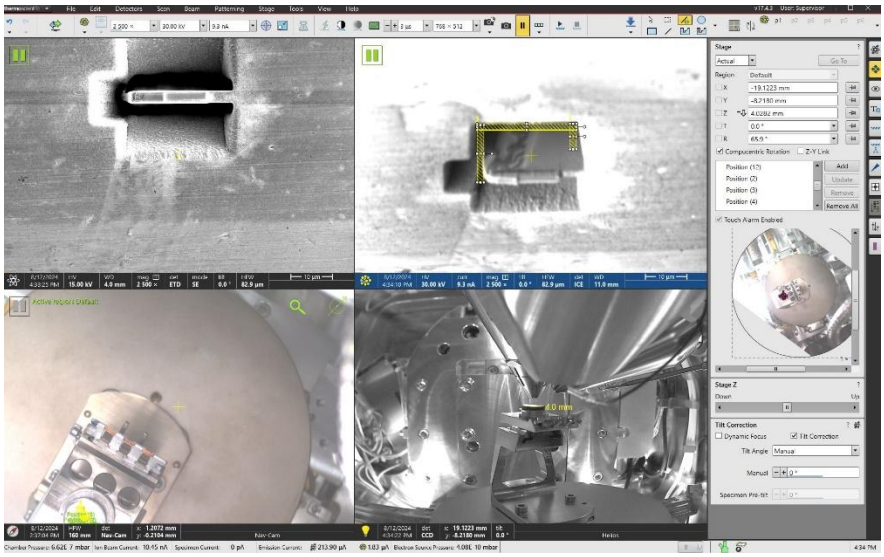
- Select CCS for top processing.



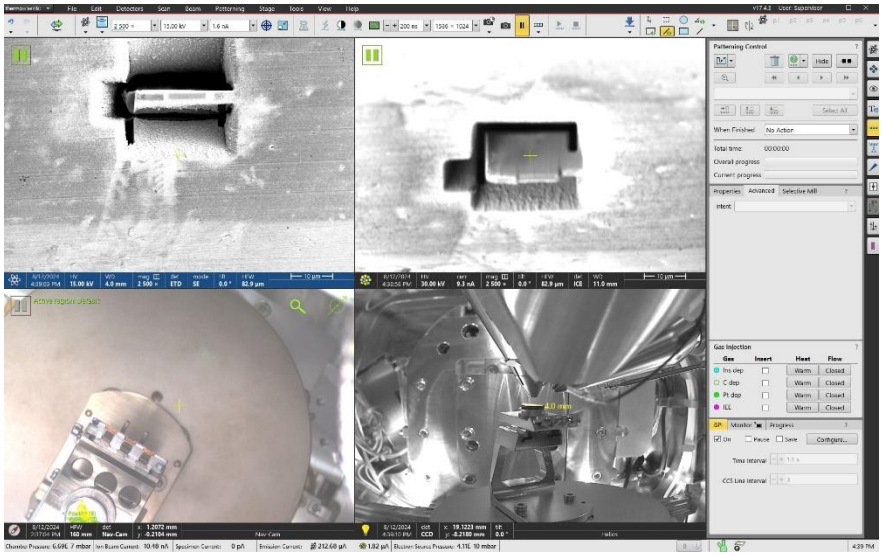
- Select CCS for bottom processing.



- Focus, take screenshots, adjust, and start the process.
  - At zero-degree tilt, select undercut, focus, take screenshots, and start for a fine cut.



- Once Undercut is done, the feature will look as below

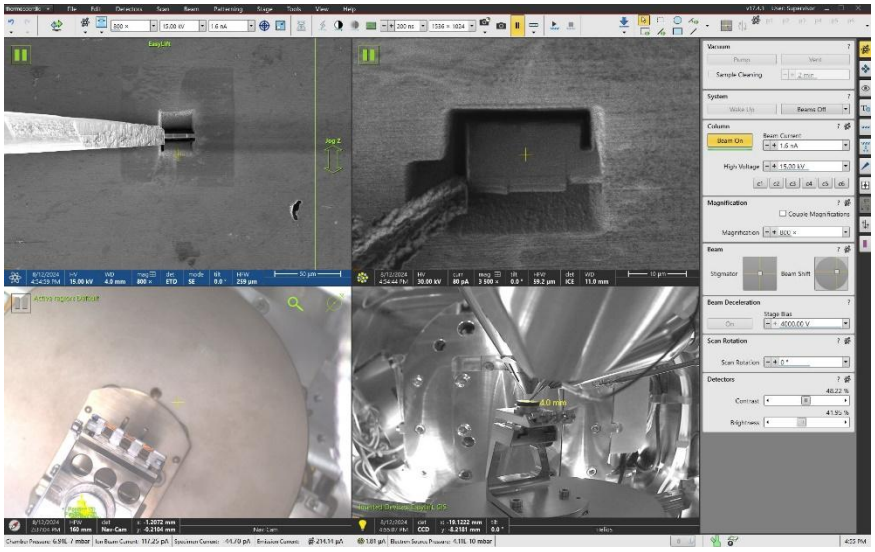


- After undercut is done, once again do CCS Bottom cleaning
- Now lamella is ready for the liftout procedure.

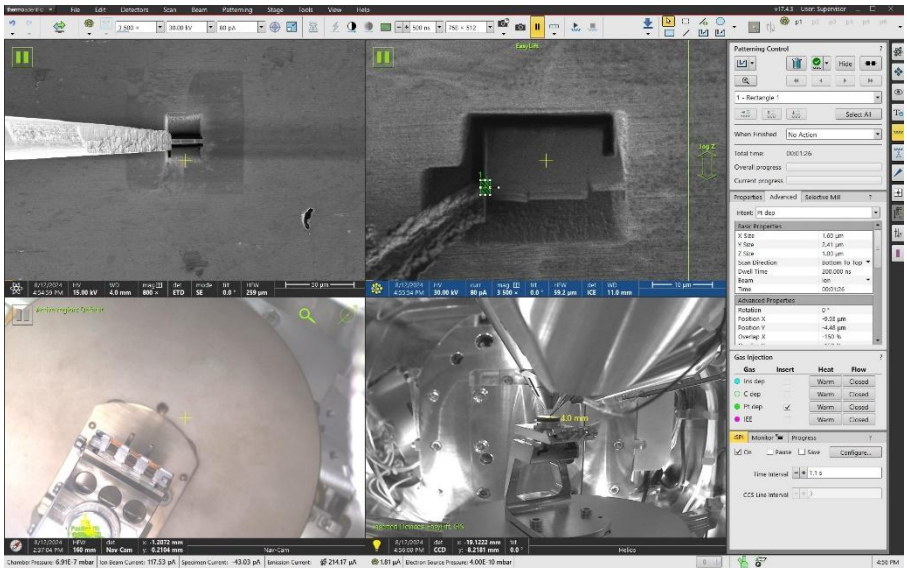
Liftoff and Attachment:

Go to the 0-degree tilt and insert the manipulator probe for attachment of lamella.
Make sure the eccentric height is correct. Slowly using the controls, approach the probe towards the lamella. You can see the probe view in both E-beam and I-beam window. Set

24pA current in I-beam for viewing. When the probe is a few microns away from the lamella then insert the GIS needle. Position the probe in such a way that it lightly touches the lamella and then attach the lamella to probe using the Pt-deposition.

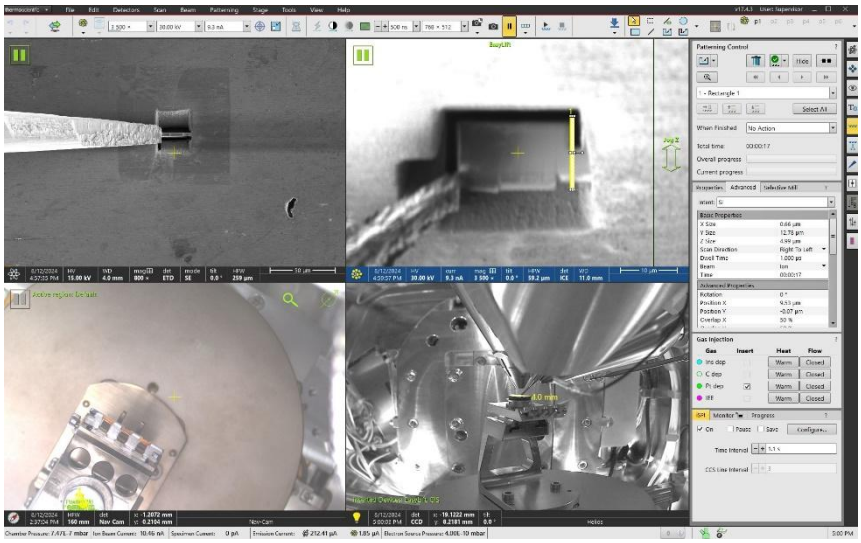


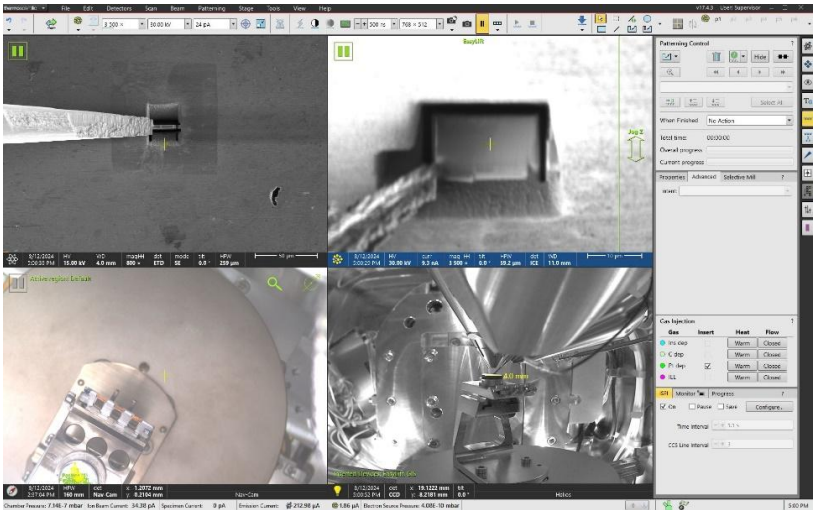
Attach easylift to the lamella at 80pA, z = 1 micron, focus, and start the process.



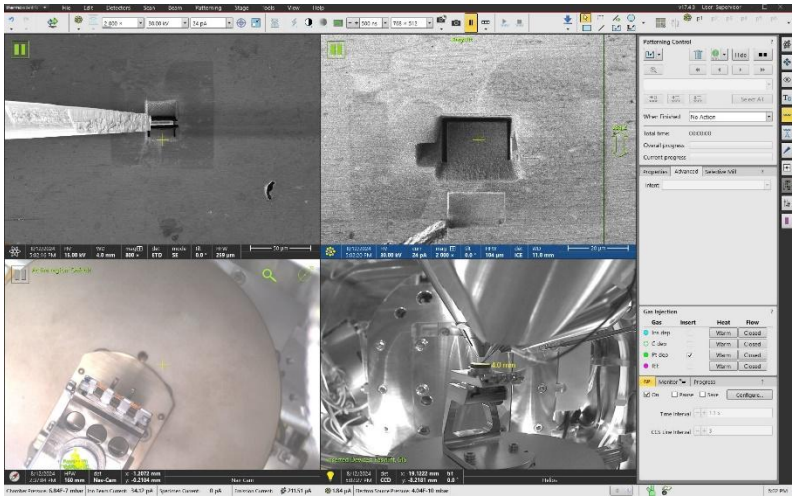
Break bridge:

- Break the bridge, retract gradually (1 micron, 10 microns), retract GIS, and then the manipulator needle.





After attaching the lamella, remove the GIS needle and free the lamella from the sample.



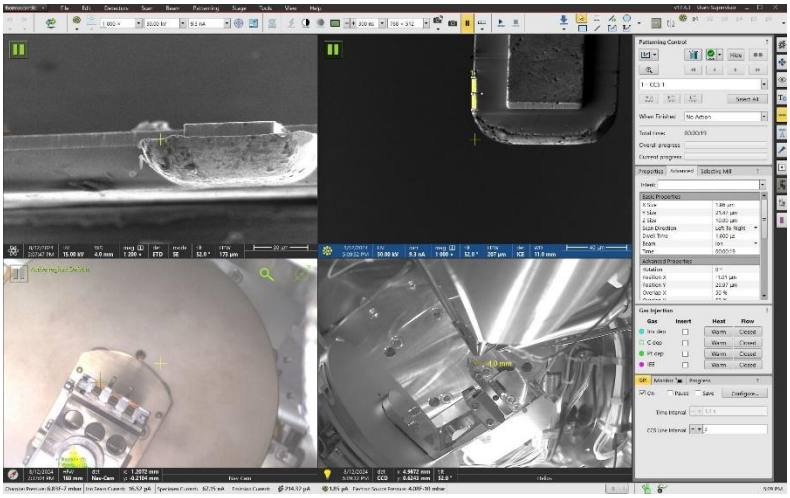
Retract the Probe to its parking position.

TEM Grid Viewing:

Go the grid and do the Compu-centric rotation to rotate 180 degree. Now again follow the setting eccentric height procedure on grid. focusing on the top of the grid, and adjust u-centric height to 4mm.After completing eccentric height procedure, select the area for the lamella attachment. Set current to 24pA, and view the TEM grid at zero degrees.

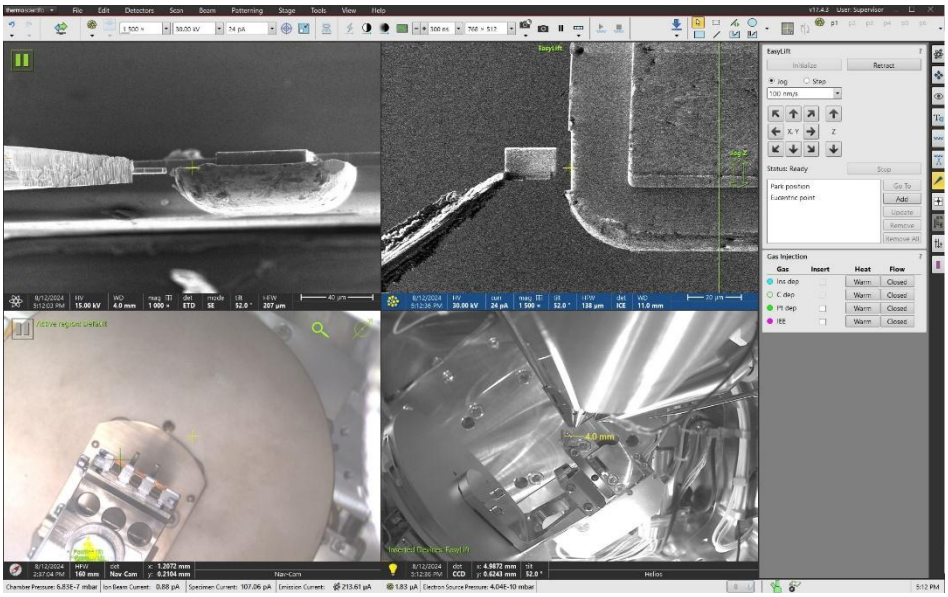
Finger Preparation:

- Do finger preparation to attach lamella to grid. Set the current to 1.8nA, focus, take screenshots, and start. Repeat if necessary.



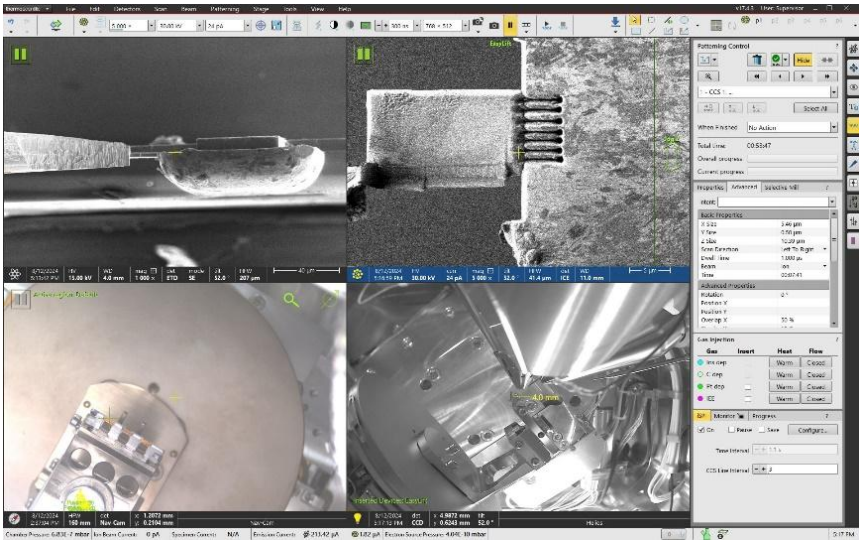
Lamella Attachment to the grid:

- Tilt to 52 degrees, insert the manipulator needle, align the lamella, and check in e-beam and I beam.



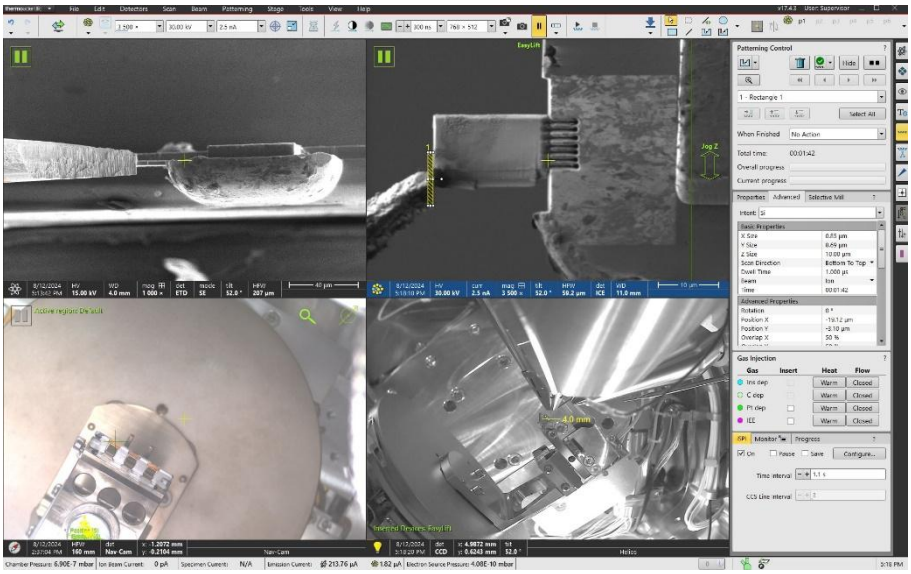
- Utilize GIS free attach, and align the boxes precisely on the lamella's border with tips touching it.

- Lamella is attached to the grid.

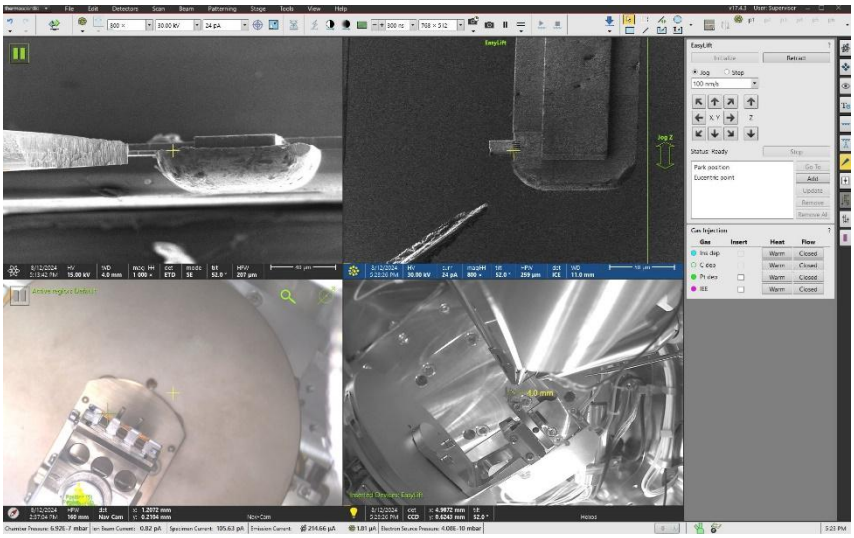


Needle Retraction and Realignment:

- Detach easy lift from the lamella, sharpen the needle, and retract it.

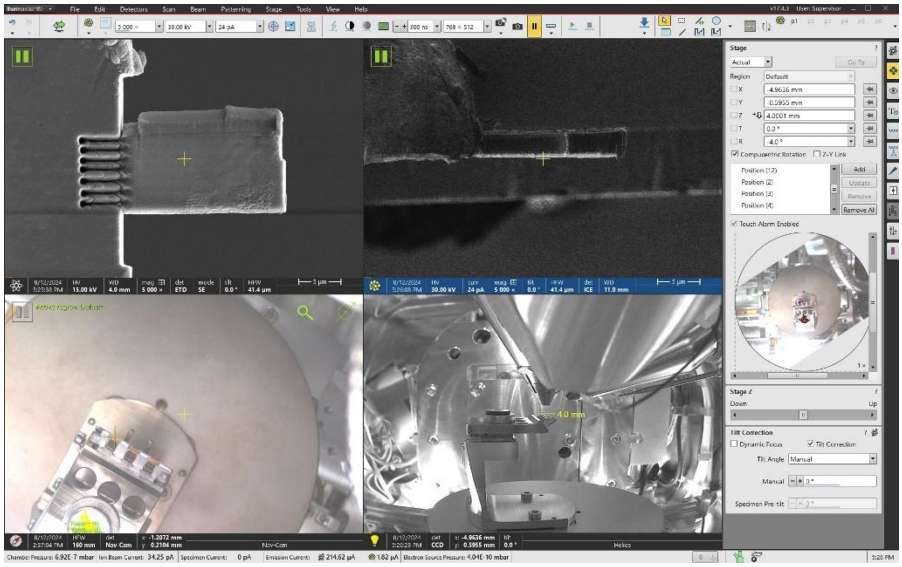


Retract the needle after lamella is attached



Final Viewing and Adjustment:

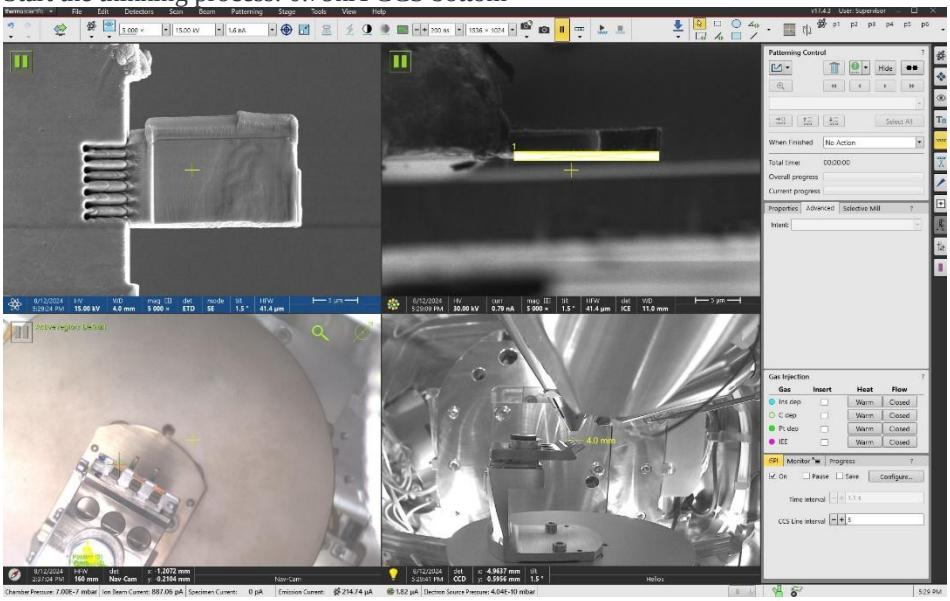
- Return to zero degrees, perform a 180-degree compucentric rotation, focus, adjust u-centric height, and tilt to 10 and 52 degrees for further adjustments.



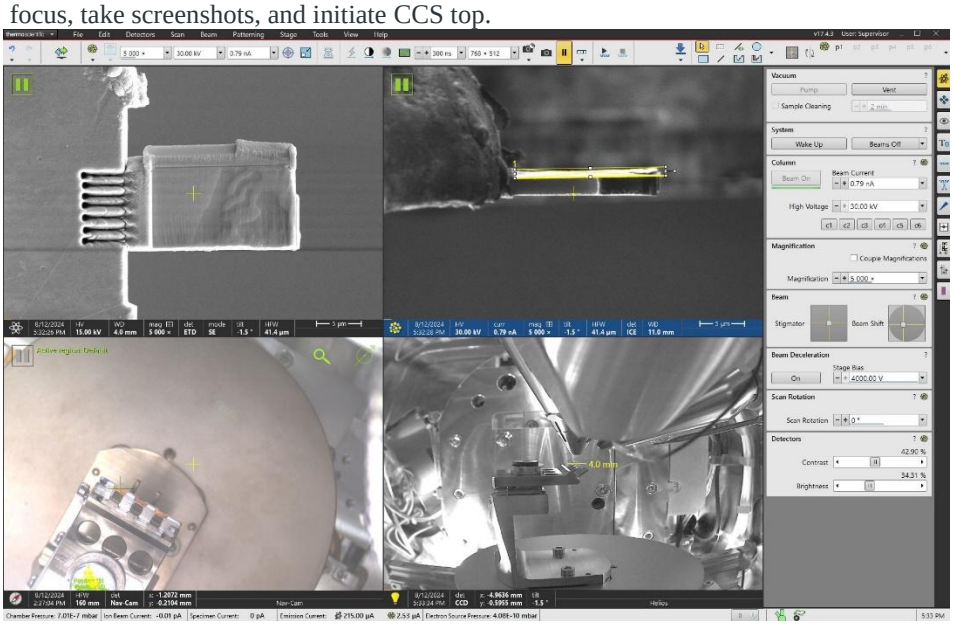
Thinning Initiation:

- 10KV E-beam and 30 KV (Current-0.79 nA) I-Beam thinning (Bottom and top surface with tilt of $1.5° \; to \; -1.5°$. Capture screenshots at the lamella in I beam (24pA). adjusting z = 5/10 microns.

- Start the thinning process: 0.79nA CCS bottom



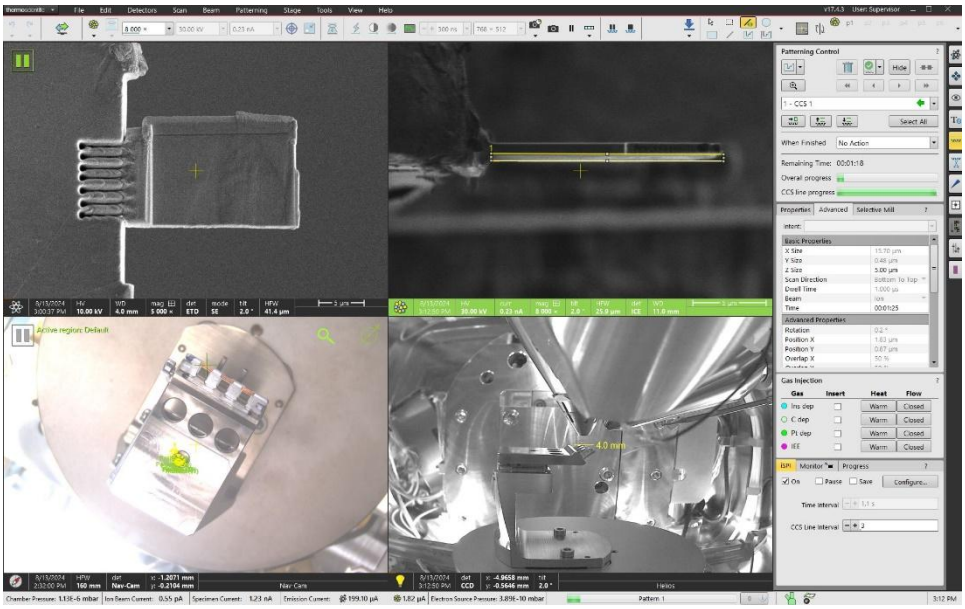
- focus, take screenshots, and initiate CCS top.



- Repeat for desired thinness. If the sample is looks (white) i.e electron can transmit through sample thickness then switch it to next step.

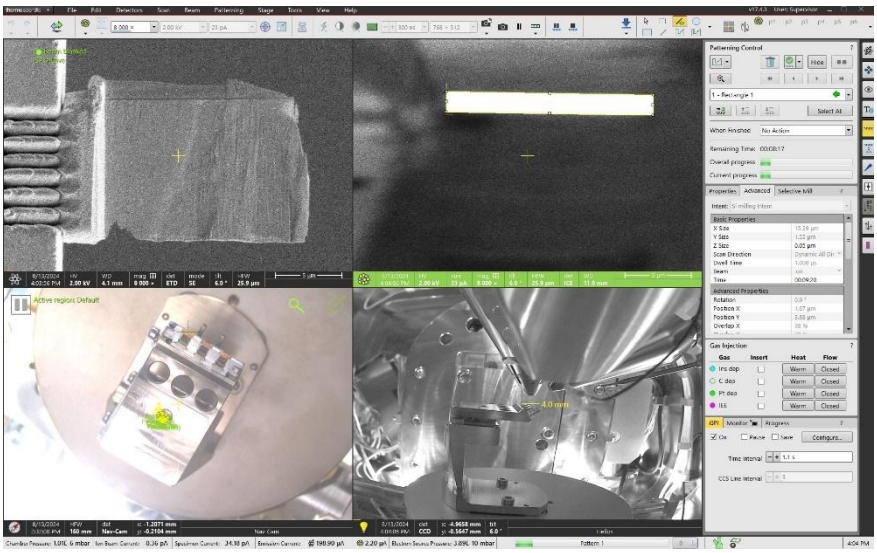
Thinning Progression:

1. Similarly select 0.23nA followed by 80pA for CCS bottom and top. 5KV E-beam and 30 KV (Current-0.23 nA) I-Beam thinning (Bottom and top surface with tilt of $1.5° \; to \; -1.5°$. If the sample is looks (white) i.e electron can transmit through sample thickness then switch it to next step.

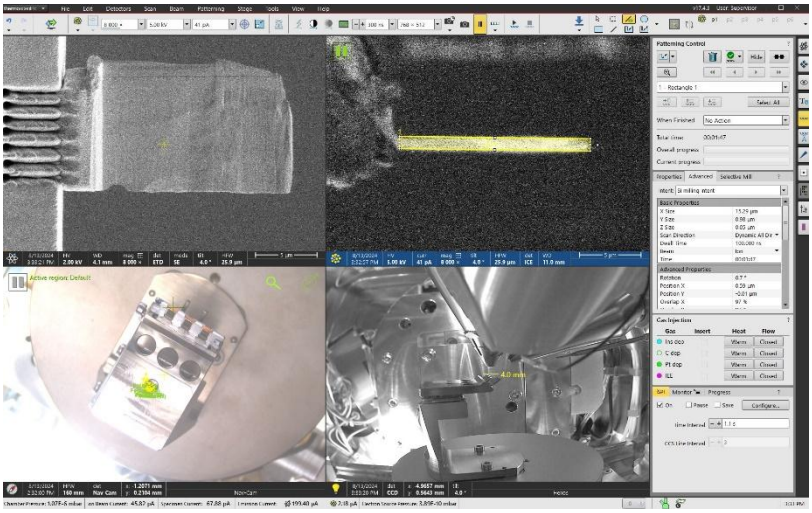


- # Final Cleaning:

2KV E-beam and 5 KV (Current- < 23 pA) I-Beam thinning (Bottom and top surface with tilt of $4° \; to \; -4°$. If the sample is looks (white) i.e electron can transmit through sample thickness then switch it to next step. Repeat till desired e-beam transparency is achieved. Do not pause in e-beam at this stage.



Final cleaning with 2KV I-Beam, 41pA to remove amorphization.

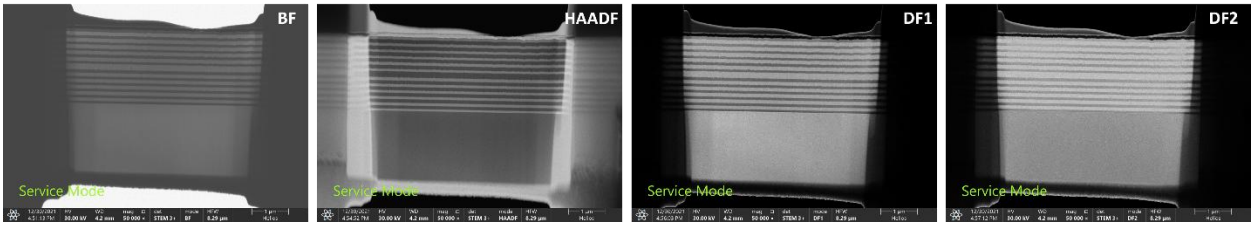


STEM imaging process

After the lamella is sufficiently tinned, one can view the same using the STEM detector in onrder to ensure the thinning. Once checked, the lamella can then be viewed under TEM.
The process of STEM imaging is as follows

1. Ckeck if Quick loader pre-tilted holder is selected.
2. Check if the Working distance= 4mm, z= 4 mm, HV= 30 Kv.
3. In Quad 4, keep unpaused, insert STEM holder, View the lamella in the following modes : bright field, dark field, HAADF
4. Retract the STEM holder once done.

STEM imaging



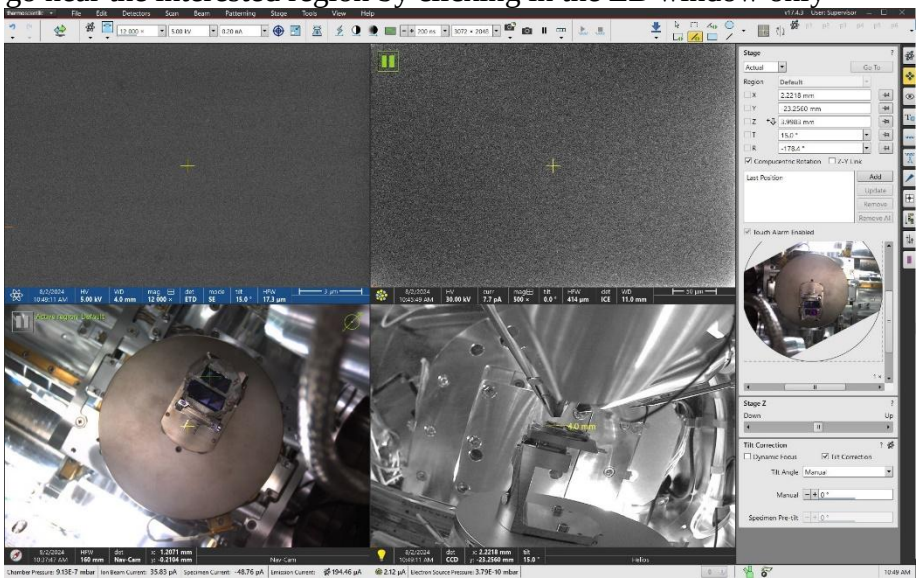
FIB Patterning

Patterning in Heleos 05 needs a digital pattern file in the following formats,

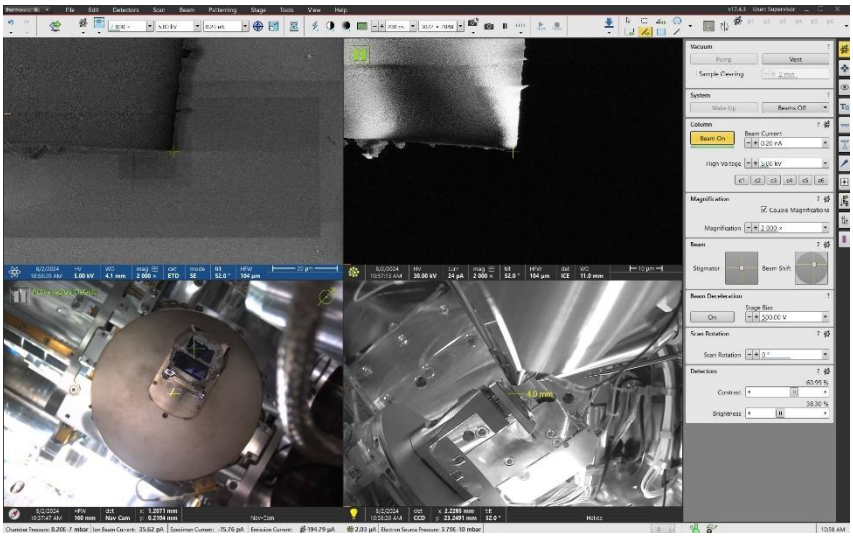
A. Bitmap file (.bmp format)
B. Stream file
C. Regular shape patterns are given in software (not preferable for nanostructure because of some glitches when small patterns are in large number).

To make a pattern,

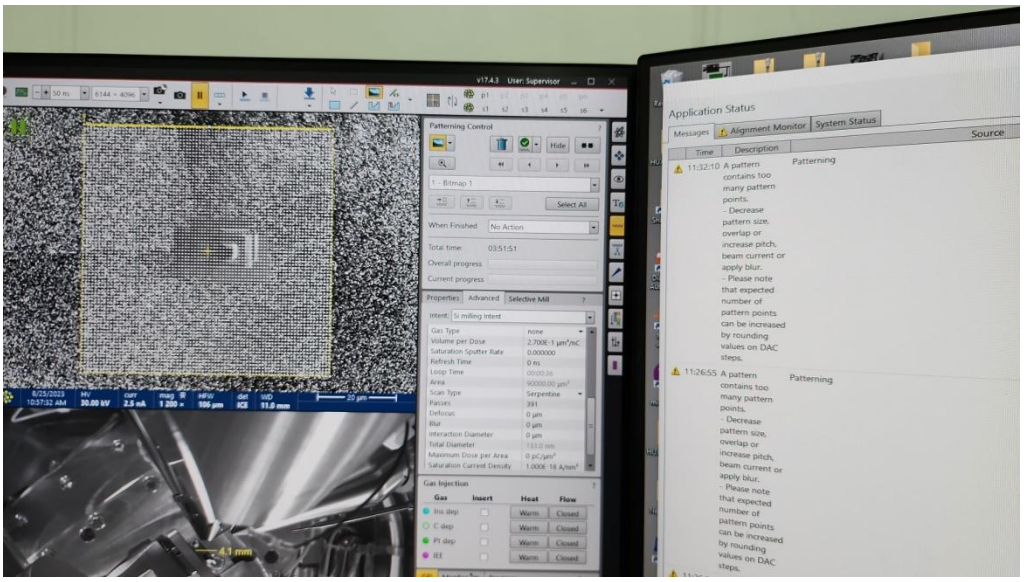
a. First, go near the interested region by clicking in the EB window only



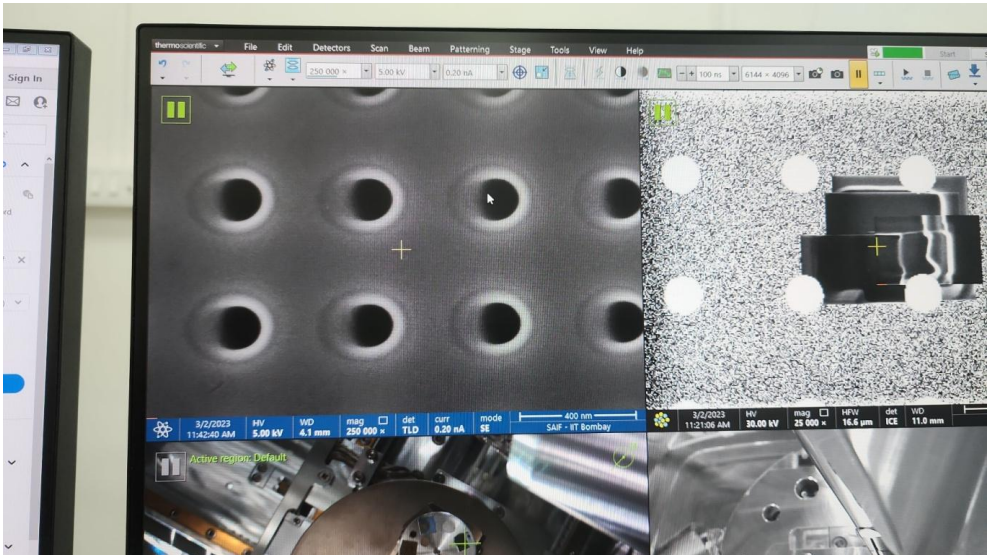
b. Click on the IB window, *FOCUS, ASTIGMATISM*, on the selected current which will be used for patterning. (Doing it properly is very crucial in patterning)

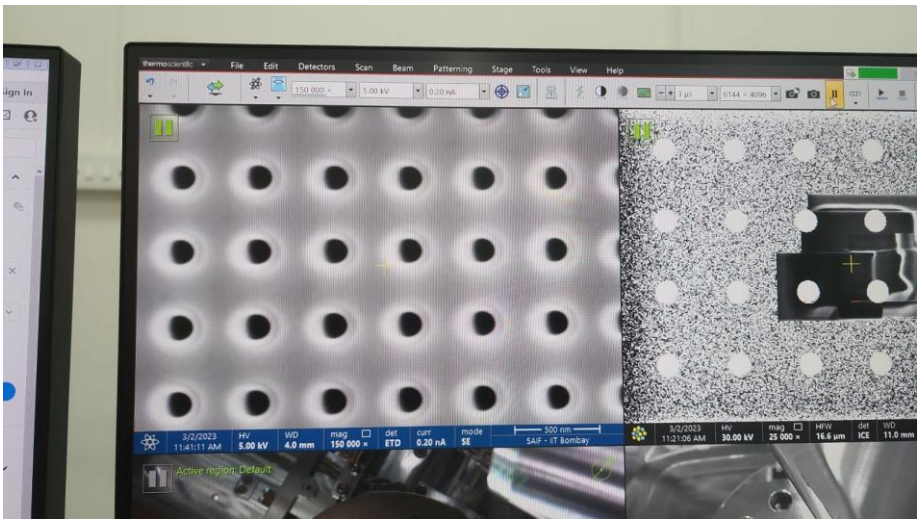


c. Go to the patterning window, choose the *BITMAP* option and draw a rectangle. choose the BITMAP file and adjust the x and y dimensions. Also, choose the z-depth of the pattern.



d. Start the patterning. After patterning, take the SEM images




File
Edit
Detectors
Scan
Beam
Patterning
Stage
Tools
View
Help
3/2/2023
11:41:11 AM
HV
5.00 kV
WD
4.0 mm
mag
150 000 ×
det
ETD
curr
0.20 nA
mode
SE
500 nm
SAIF - IIT Bombay
Active region: Default
3/2/2023
11:21:06 AM
HV
30.00 kV
mag
25 000 ×
HFW
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det
ICE
WD
11.0 mm