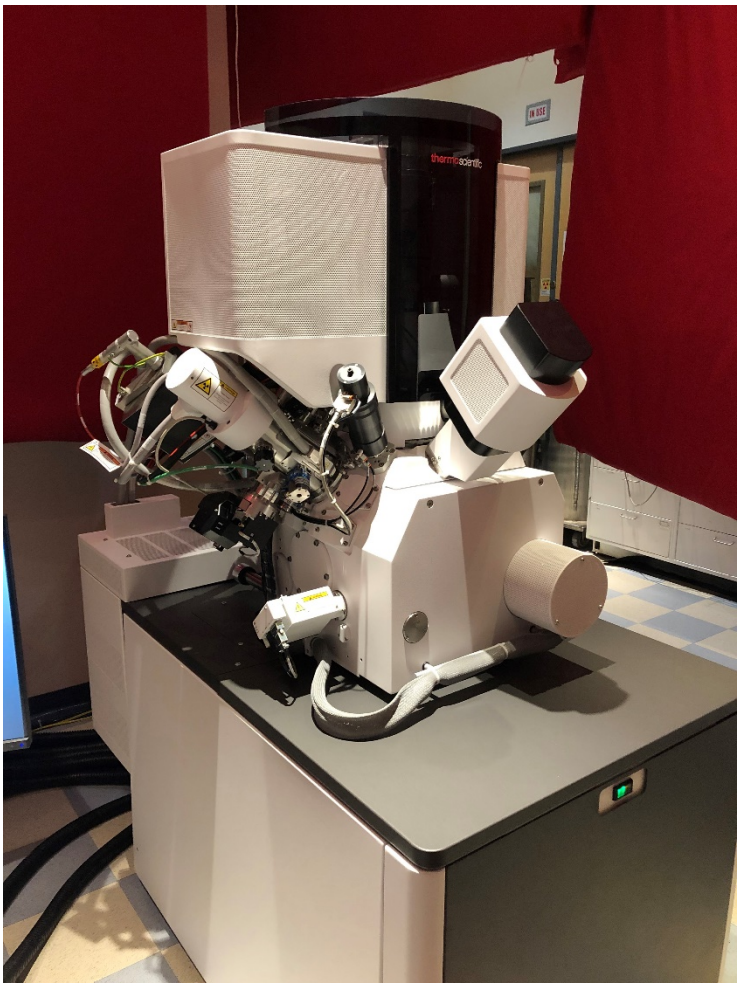
Helios FIB

part of the
CCMR EM Facility

Operating Manual

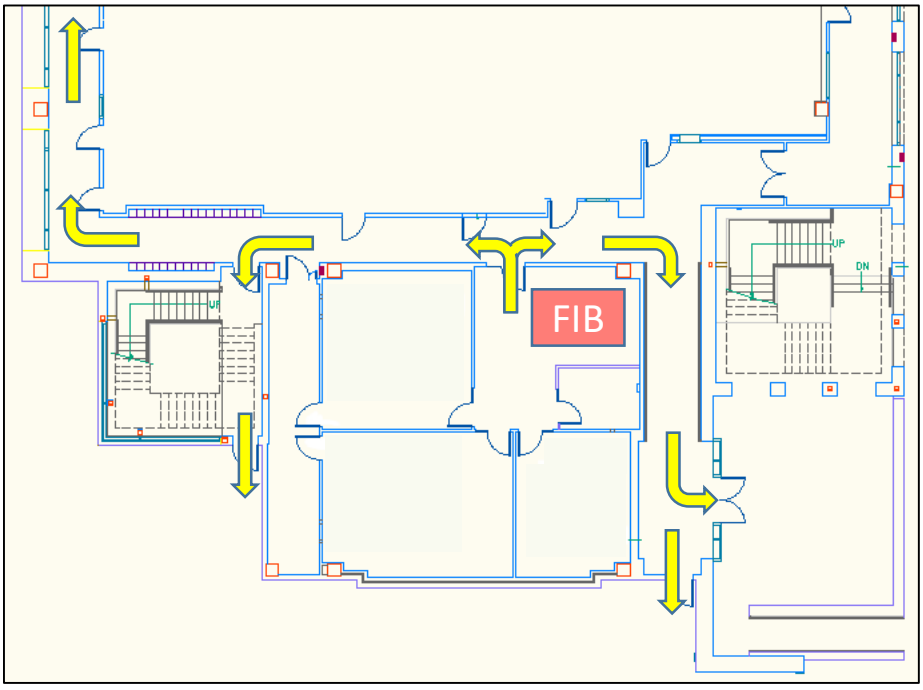


Laboratory Safety

- Users are required to have passed Cornell's on-line laboratory safety training courses particular to this lab before they can use the Helios FIB.
- Users must fill out a CCMR Shared Facility Access Form
- Do not bring any chemicals or hazardous samples into the lab.
- Take specimens, stubs, and raw material with you when you leave.
- Isopropanol and Aero-Duster are supplied for your use. Wear safety glasses and read the appropriate MSDS before using them.

Users failing to follow safety rules will be denied access to the lab.

In case of fire leave immediately and close the door behind you. Do not use the elevators.



Mick's Golden Rule

If ANYTHING does not even SEEM right, leave everything as it is and call Mick (607-592-5217). If Mick cannot be reached by phone then use text or email

Laboratory Policies

Do not attempt to repair or remove ANY part of the FIB

Do not add any software or hardware to the computer

Flash Drives (Memory Sticks) are forbidden

Data MUST be transferred via Cornell DropBox

Do not change the room temperature or bring food or drink into 150 .

Wear gloves when touching the shuttles and specimens.

All samples must be dry and very flat (details to follow)

If you sign up and then cannot use your time, please cancel your time via CCMR-FOM or contact Mick at least 24 hours before your scheduled time.

If you have to cancel your session less than 24 hours before it is scheduled to start you will be charged for your session. If you finish early (> 4 hours left) you must send a message to the Helios user group via FOM.

Failure to show up without canceling, unless due to illness or other emergency, will result in use charges

If you are more than 30 minutes late then the FIB becomes available to other users.

If you feel sick, please reschedule your session. You will not be charged if you stay home due to illness.

Data Storage

Data storage and safety is not guaranteed – users are responsible for their data, and must copy their data in a timely fashion.

Data will only be kept on the hard drive for one year. Periodically the hard drive will be erased, leaving only one years' data there.

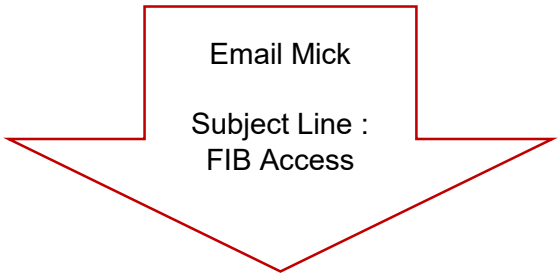
How to acknowledge CCMR in publications

When research is published using data from this microscope, the facility and the grant number must be acknowledged:

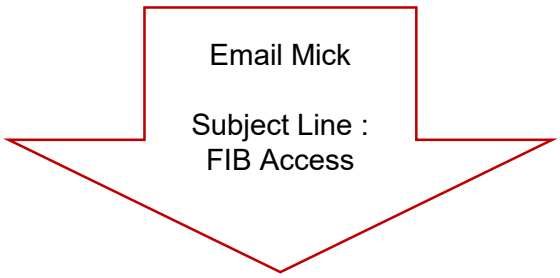
"This work made use of the electron microscopy facility of the Cornell Center for Materials Research (CCMR) with support from the National Science Foundation Materials Research Science and Engineering Centers (MRSEC) program (DMR-1719875)."

Laboratory Access

User Status	Signup	Room Entry	Access
Novice (Sessions 1 and 2)	Email	Manager	Mon – Fri 8am – 5pm



Trained (Sessions 3 and 4)	CCMR-FOM	Card Swipe	Mon – Fri 8am – 5pm



Experienced (session 5 and on)	CCCMR-FOM	Card Swipe	24/7

Problematic Samples

Some samples pose special problems to the Helios FIB and must be avoided or prepared very carefully.

Samples with irregular surfaces

Samples that are not very flat can pose a problem for the FIB due to the extremely short working distance (4mm at eucentric height). If a sample varies more than about 250um in height then contact Mick

Moist or wet samples

Moist or wet samples are not allowed at all in the FIB

Magnetic samples

Magnetic samples are not allowed in the FIB without consultation with Mick

Powder samples

All powder samples must be vacuumed and blown off.

All powder samples must be coated with gold/palladium or carbon

About this manual

This is a ***limited use manual*** intended to help new users get started on the Helios FIB. It provides information on basic imaging, column setup, and saving and annotating images. It is not intended to be exhaustive, and there are many principles about Focused Ion Beam technology and many features of this FIB that are not covered by this manual. This manual will be updated and expanded as needed.

Starting the software

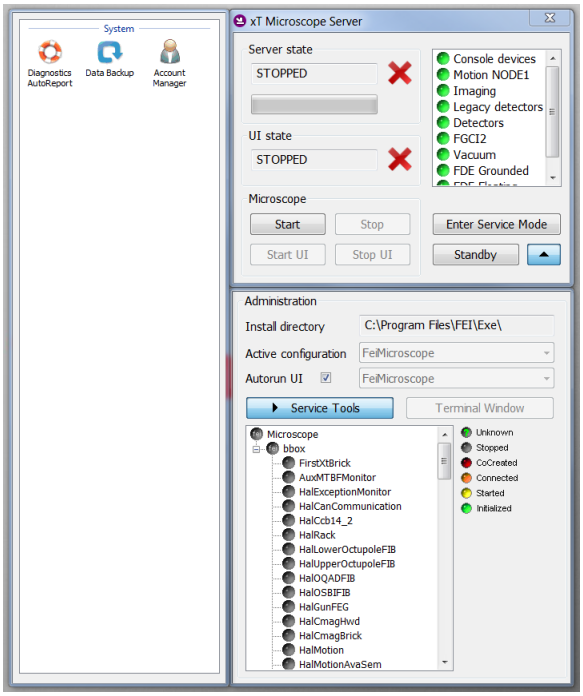
Log into CCMR-FOM

If the server IS NOT running:

Click on xT microscope Server

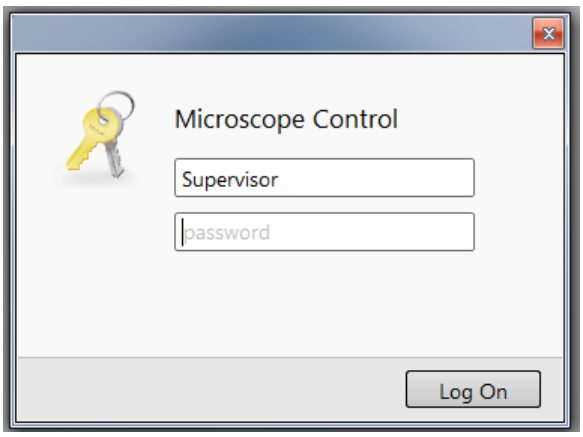
Click on Start

Continue below



If the server IS running:

Log into Microscope Control with your NetID and the Password you set up during training

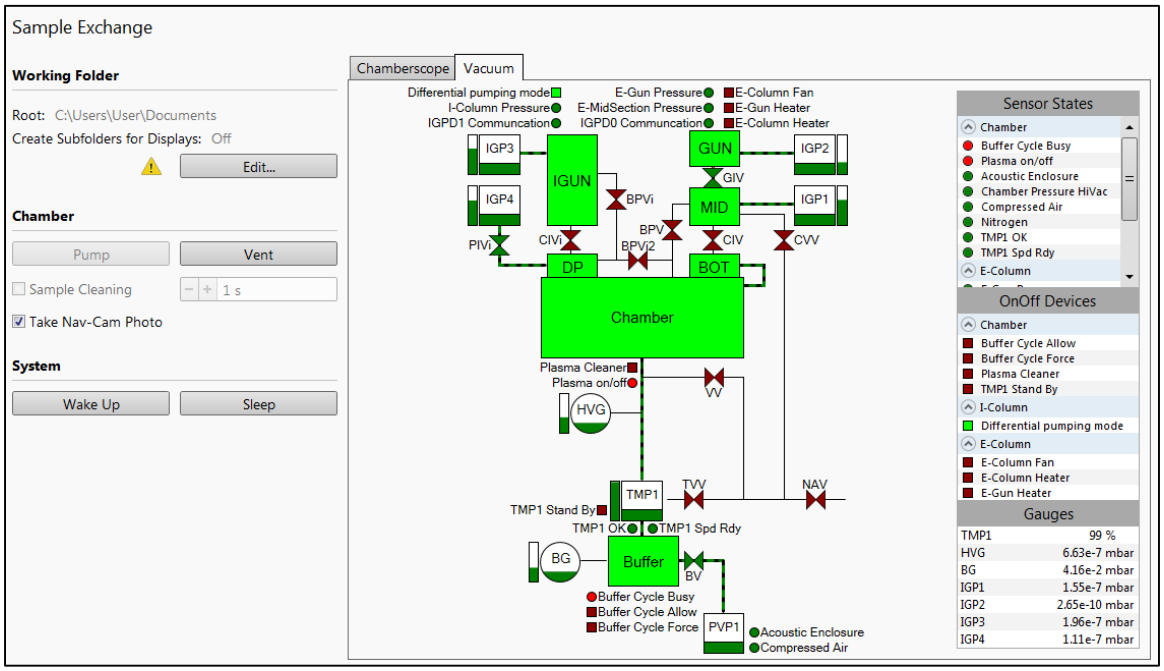


Loading Samples

Click on 

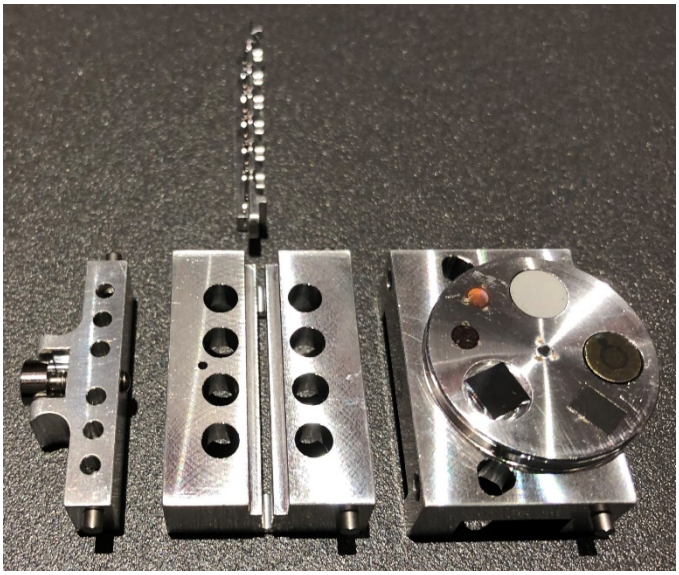
Note vacuum layout – all boxes should be green and most valves are dark red

Click on Chamber -> Vent



Loading Samples

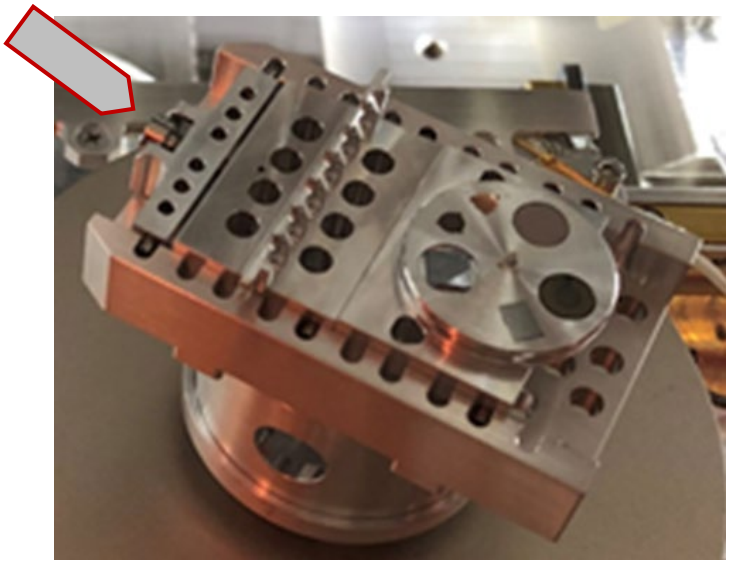
The stage comes apart into several sections as shown at right. However you generally do not need to remove these pieces.



<u>One-sided (SEM type) sample:</u>
Leave the parts in the stage and just mount your stub.

<u>Two-sided (TEM) sample:</u>
Loosen the screw as shown at right. Remove the TEM row holder and mount the grids you need.

Mount your sample and stub into the stage.

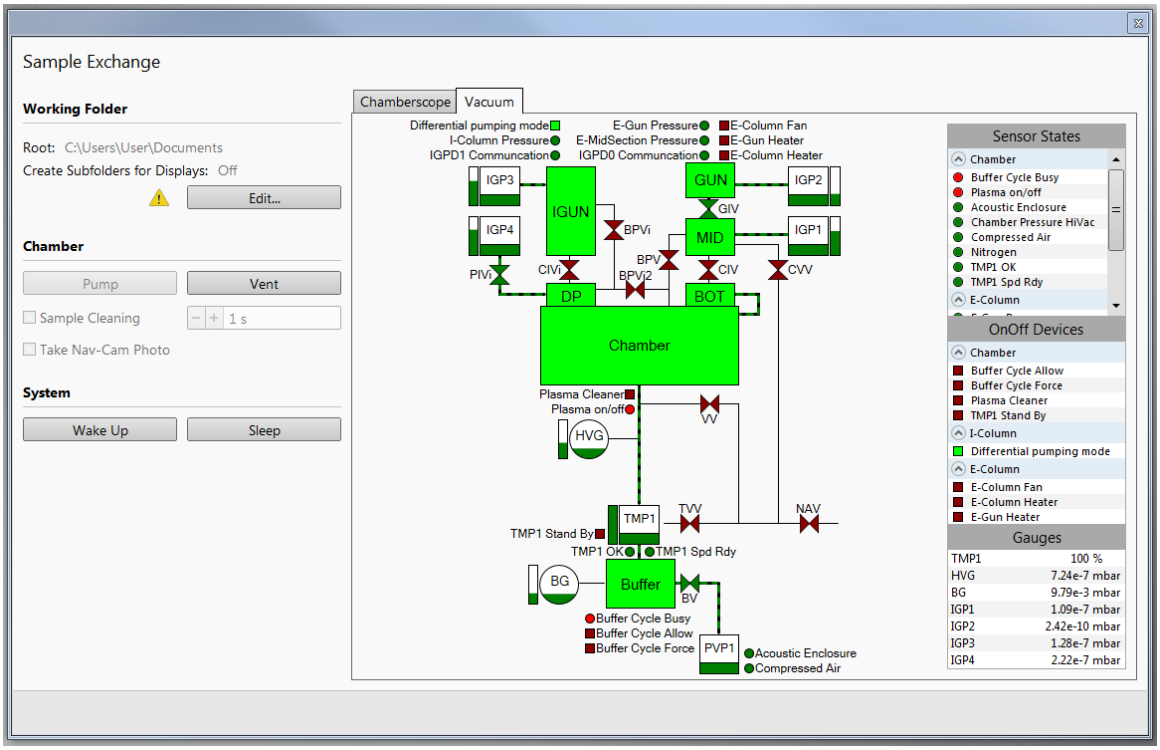


Loading samples

Take Nav-Cam Photo should be selected.
If not then click on it.

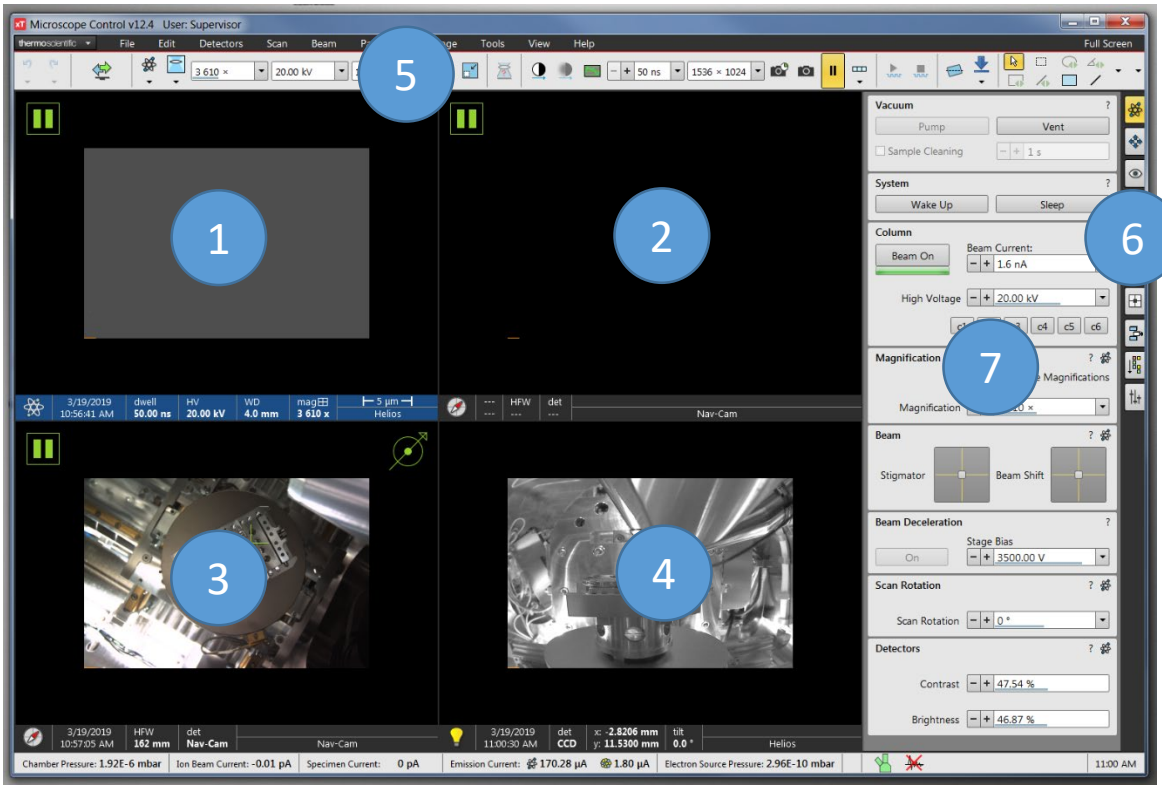
Click on Pump and GENTLY hold the front door shut

Do not begin until the chamber High Vacuum Gauge (HVG) reads less than $4x10^{-5}$ mbar. At this point all chambers should be green. If not then contact Mick.



Loading samples

The main screen consists of 7 areas:



1: Electron Image
2: Ion Image
3: Nav-cam image
4: TV image
5: Main taskbar
6: Operations Panel selector
7: Operations Panel

Imaging

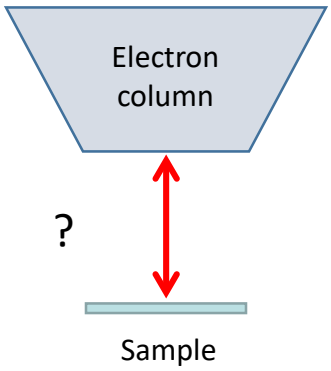
Using the Nav-Cam photo select an area on your sample to work and click on that location. Note the image in the Nav-Cam window will not move but your sample, as viewed in the TV image, will move.

Click on Beam Control and turn on the electron beam.

Un-pause the electron beam and focus on the area of interest.

The FIB does not know where the sample is in reference to the polepiece (vertical position). The Z value displayed in the Navigation window will be wrong. Therefore it is critical to tell the FIB where your sample is – a process called Linking.

Do not adjust specimen height (Z) until you have Linked at low mag.



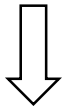
With the electron beam focused on the surface of your sample,

Link the stage by clicking on the Link button:

Imaging

In the Operations Panel selector choose the Navigation tab.

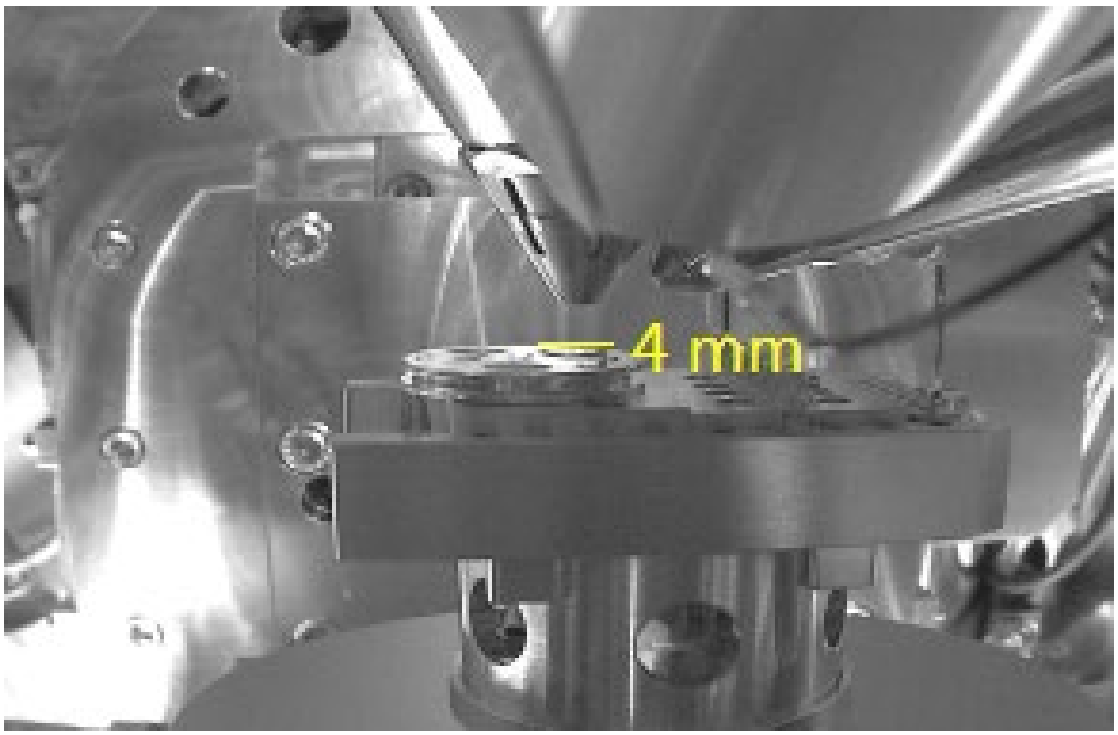
Important:
The Z control MUST have a hollow black arrow next to it pointing down:
If it does not then you have not linked and stage motion is dangerous!
Do not move the stage is the arrow is not a hollow black arrow!



Set the Z value to 6mm. Make sure the stage moves up.

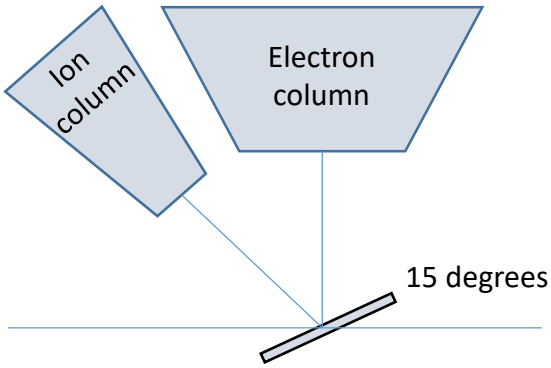
Refocus and re-Link the stage

Set the Z value to 4mm. Refocus and re-Link the stage. Set to 4mm again if needed.



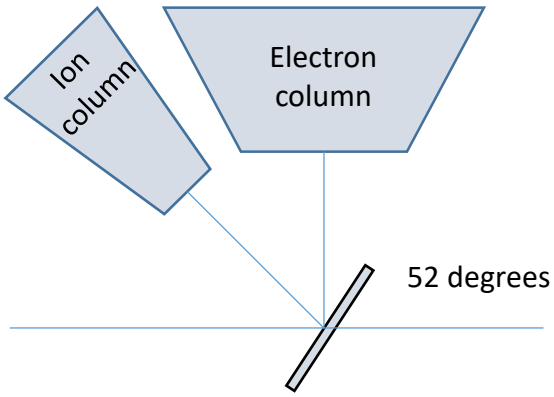
Imaging

Select an easily recognizable feature in the electron beam and tilt the stage to 15 degrees..



If the feature has moved up or down as seen on the e-beam image then use the Stage Z slider control in the Operation Panel to raise or lower the stage as needed.

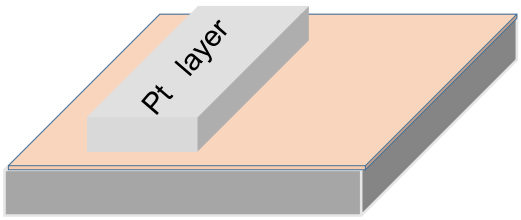
Continue the process of tilting and adjusting Z height until you reach 52 degrees



One-sided (SEM-type) samples

(For two-sided (TEM) samples skip to TEM sample Prep)

Add platinum, as shown below, to protect the sample during milling.



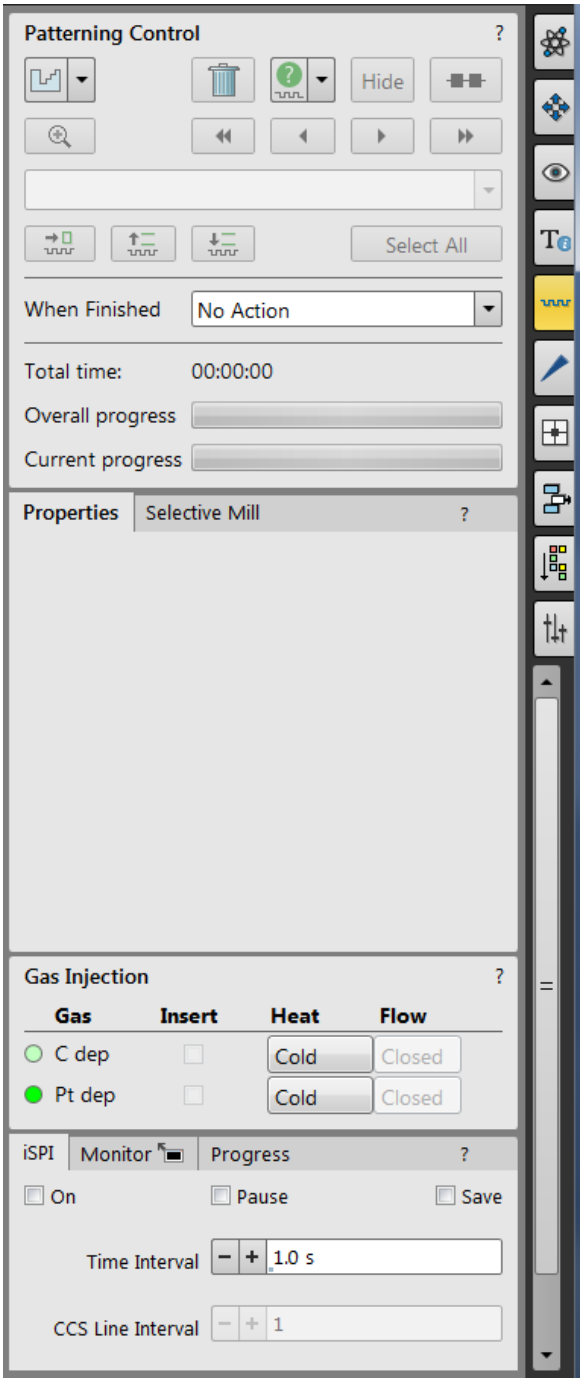
There is no specific restriction on the size of the pattern, but generally, for most cross section samples, it is around 3mm by 20mm. More than this will begin to take a large amount of time.

Under Patterning Control, choose the Rectangle and draw it on you sample as seen in the electron or ion image.

Under Properties select Pt e-beam or Pt I-beam.

Under Gas Injection click on Insert.

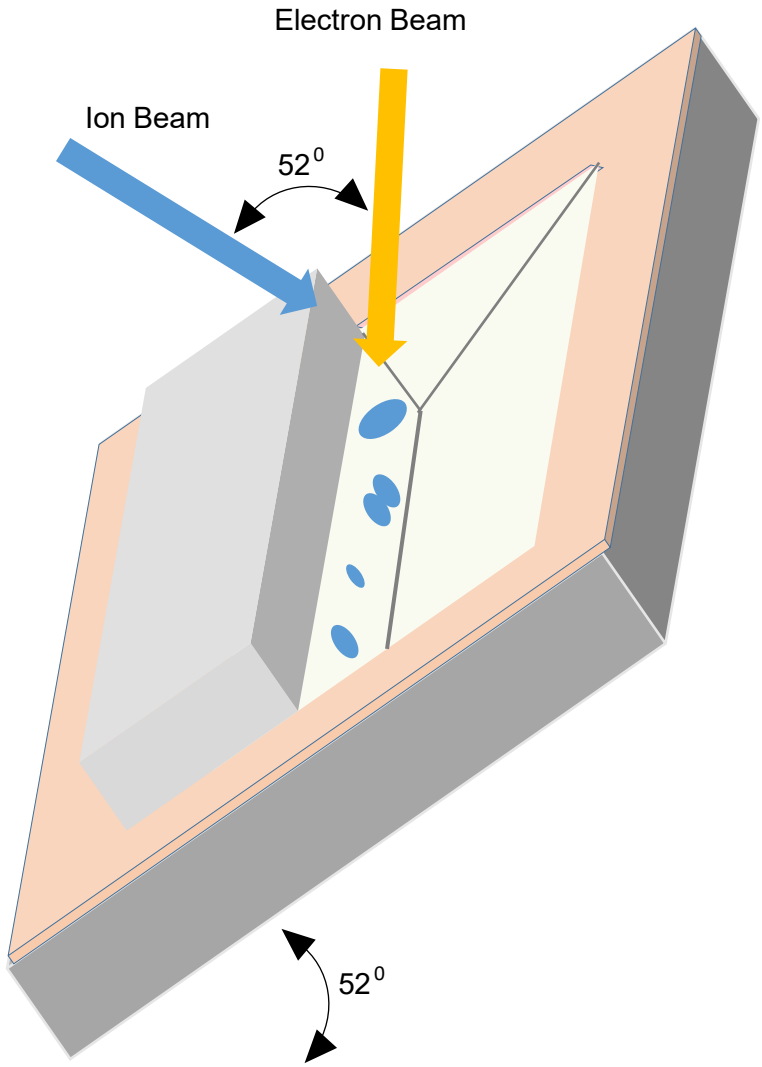
Click Play in top menu bar 



SEM (one sided) sample prep

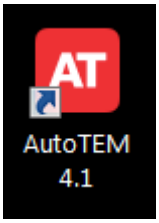
Under Patterning Control select the Regular Cross-section or the Cleaning Cross section. Both will create a triangular shaped volume which enables you to image the sub-surface feature of interest. For bulk milling usually 30keV is used to start, with the current dependent on the sample composition. The harder the material, the higher the beam current.

Final polishing is typically done with 5keV, with the current dependent of the hardness of the sample.

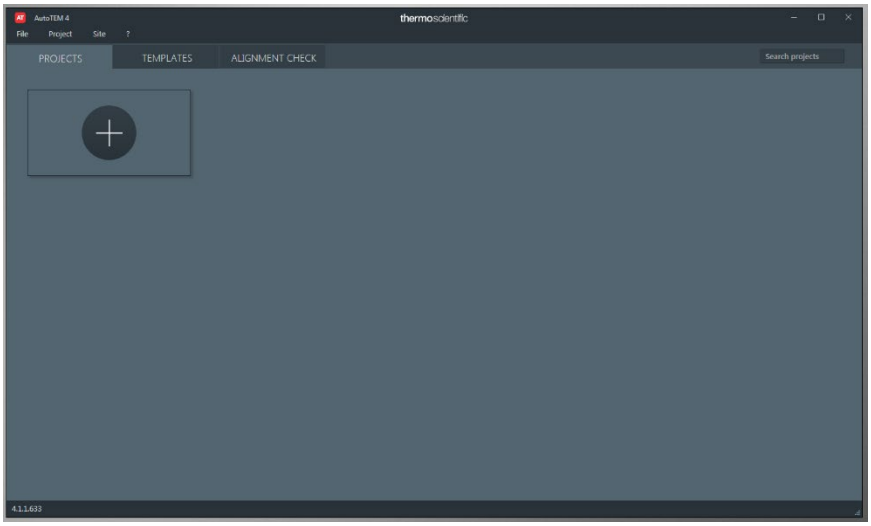


TEM (two sided) sample prep

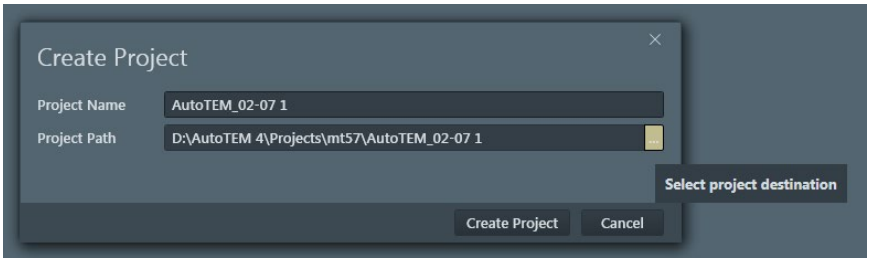
TEM sample prep can be done semi-automatically or completely manually. Here we will cover the semi-automatic procedure. For complete manual procedure contact Mick.



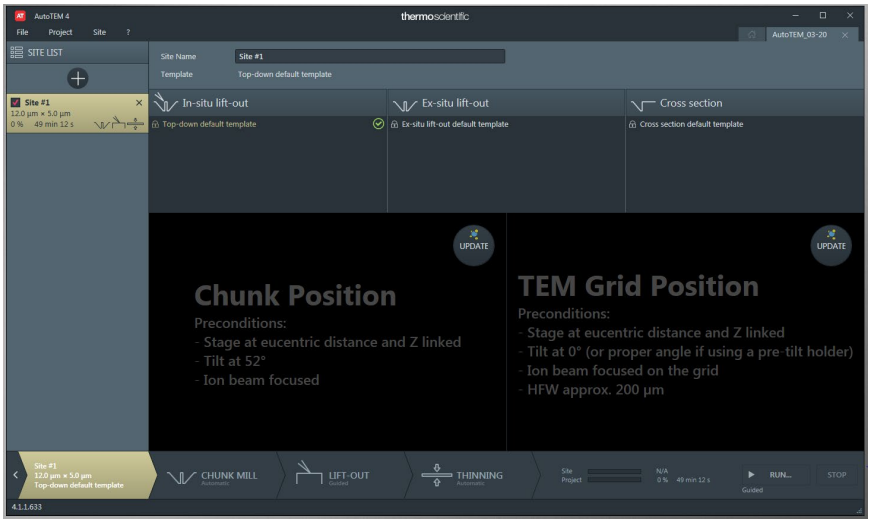
Click on the AutoTEM 4.1 Icon



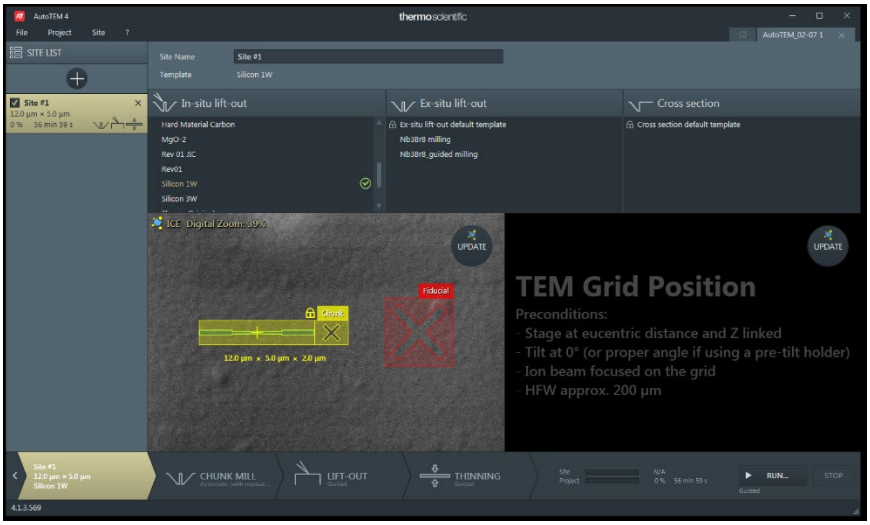
Click on + sign



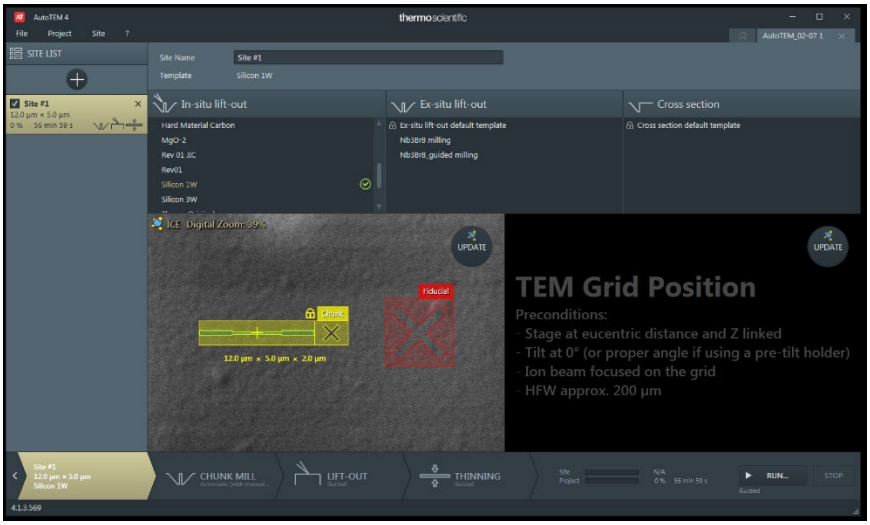
Create a project name and select its location



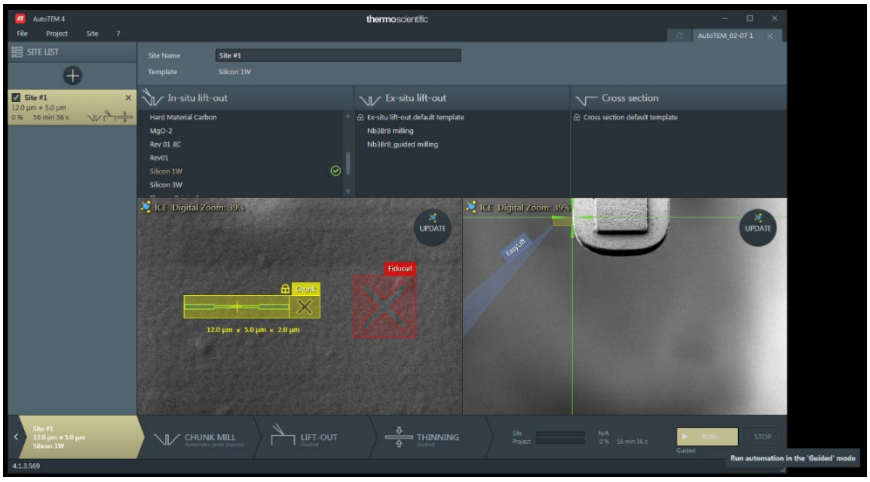
Choose an In-situ lift-out recipe. Follow instructions for "Chunk Position" using a 30keV and 0.44nA ion current. Click on Update within the Chunk window.



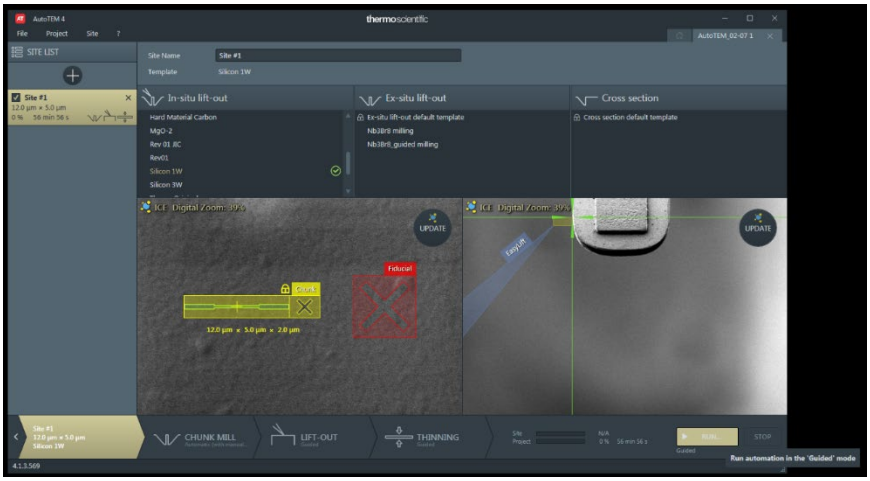
The image will look as at left.



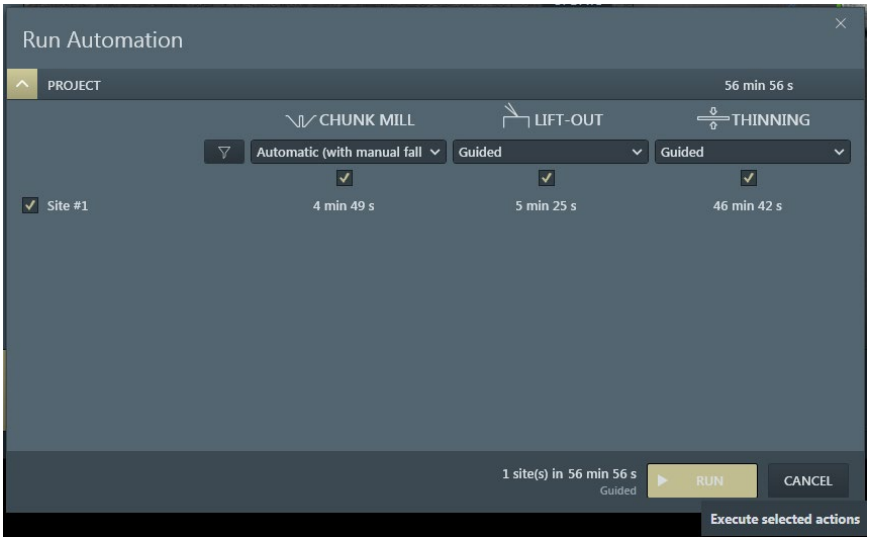
Follow the instructions in the “TEM Grid Position” window.



A cartoon diagram will show up with a sample on a needle. Drag the sample/needle combination to the spot as shown.



Click on RUN

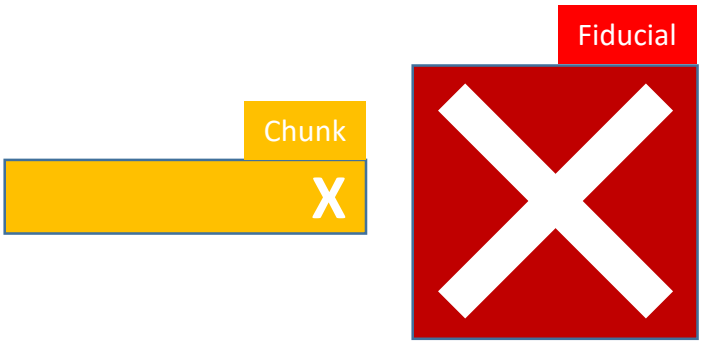


Generally the Chunk Mill is Automatic

The LIFT-OUT is Guided

The THINNING is Guided.

Click on RUN.



An X is milled in the ion window.

Then a Rectangle of Platinum is deposited.

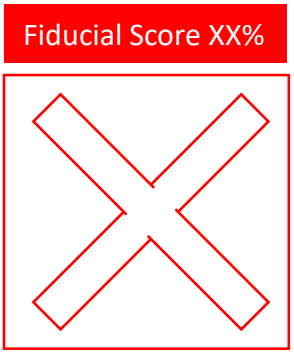
Generally the Chunk Mill is Automatic

The LIFT-OUT is Guided

The THINNING is Guided.

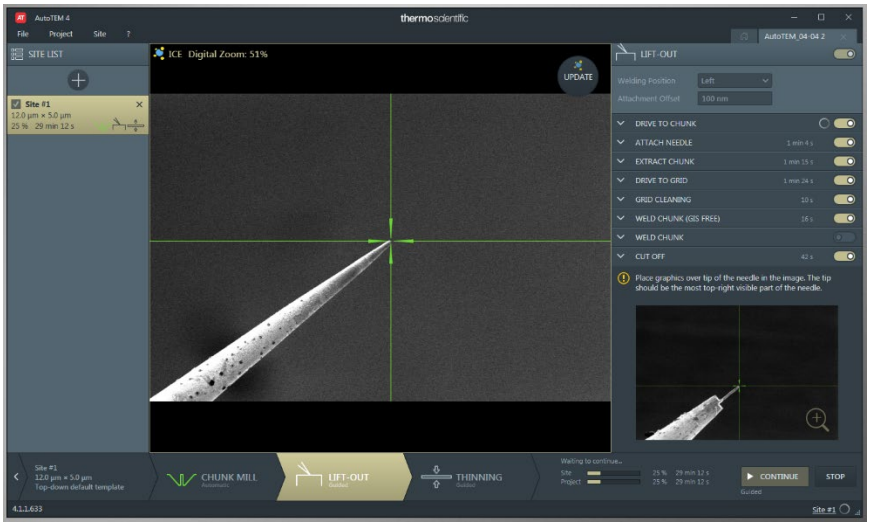
Click on RUN.

Software begins milling trenches on both sides of sample.

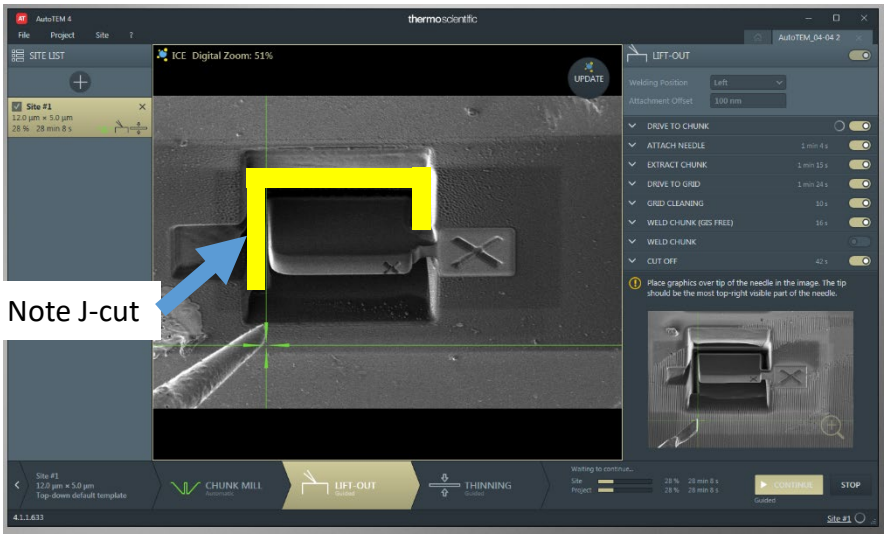


You may have to re-locate the fiducial mark several times.

First line up the needle in the electron beam.

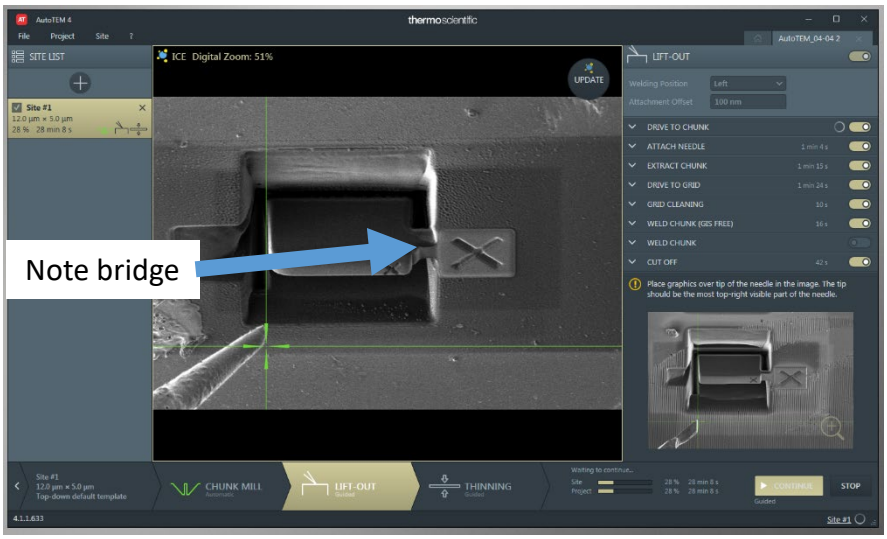


Then line up the needle with the ion beam.

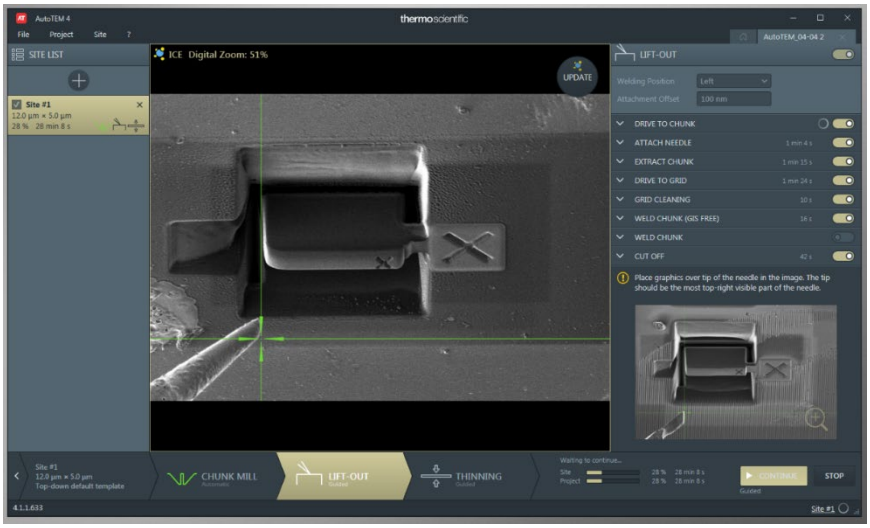


Automatic J-cutout of underside

Note that after it tilts to do the underside the sample may go out of the field of view.

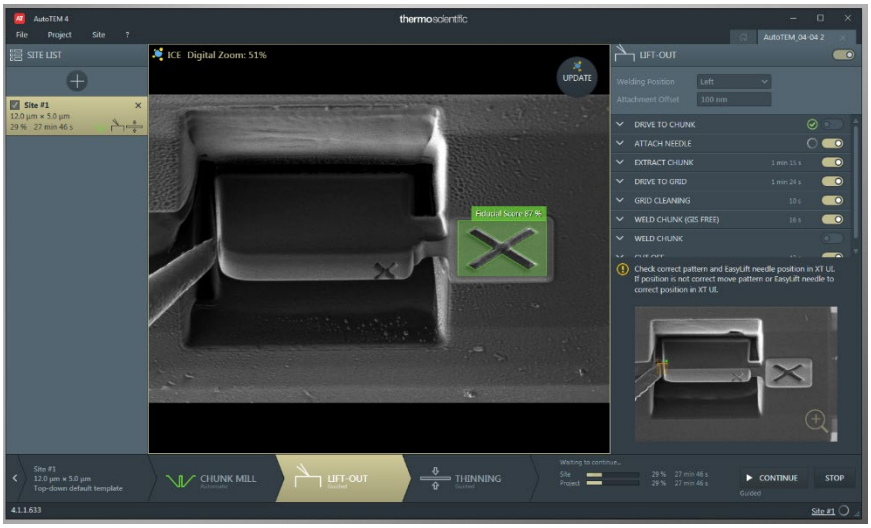


Do not move the stage just lower the mag until you can see it, click update, and move the red x and reset the X position

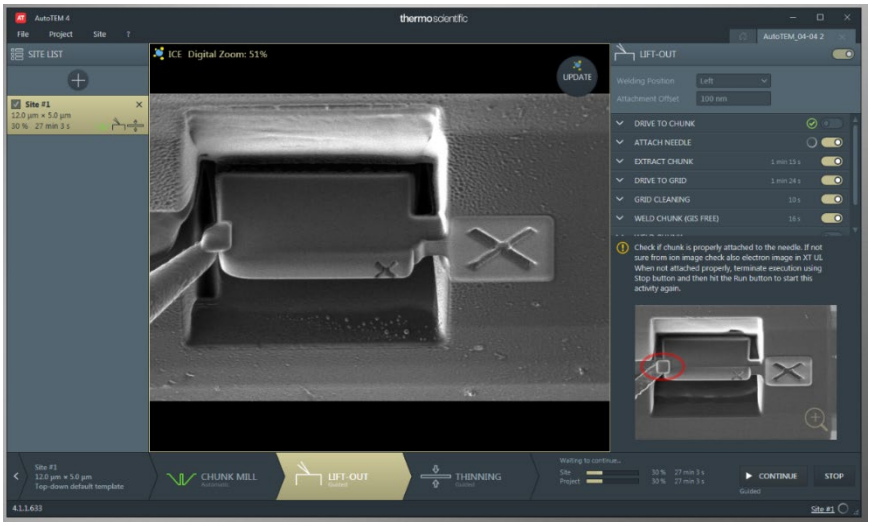


The needle will move into position, guided by the user

Needle approaches the sample, guided by the user.



The needle reaches its final position



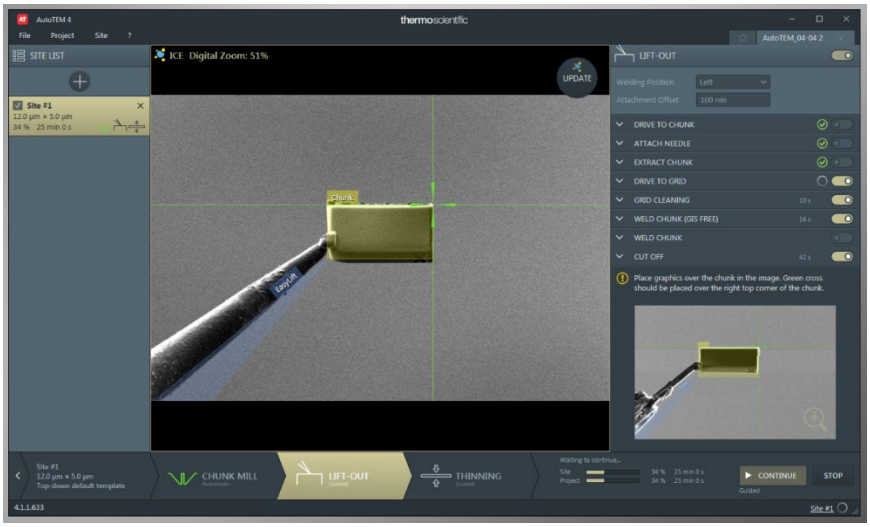
The needle is bonded to the sample, guided by the user.

Helios FIB

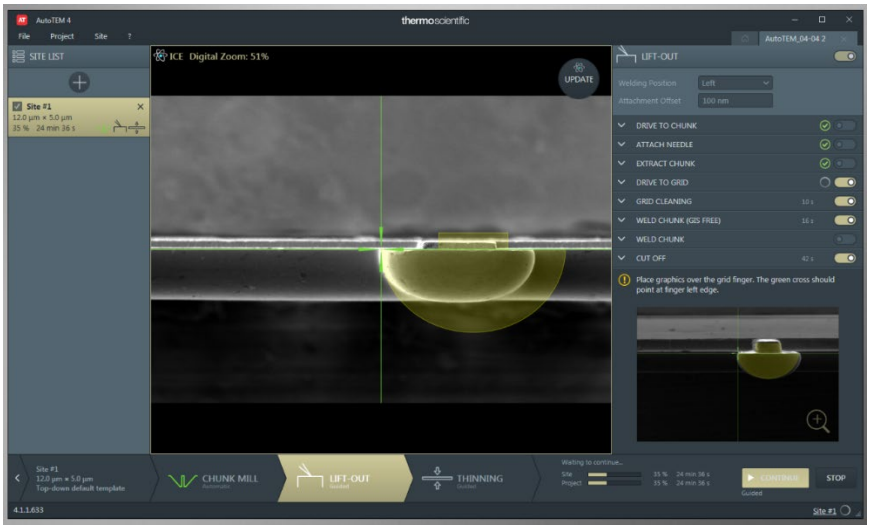
part of the CCMR EM Facility

The bridge holding the sample on is cut off.

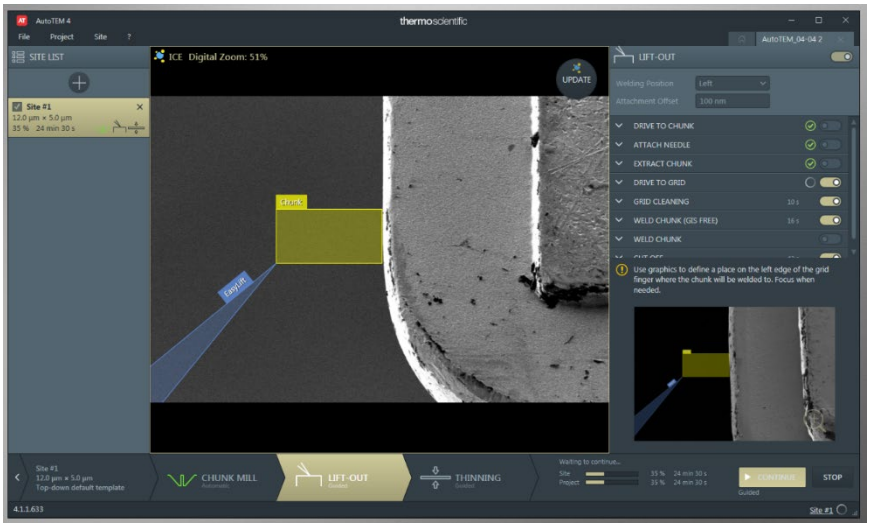
The sample is lifted out by the needle automatically.



User marks the position of the lamella.

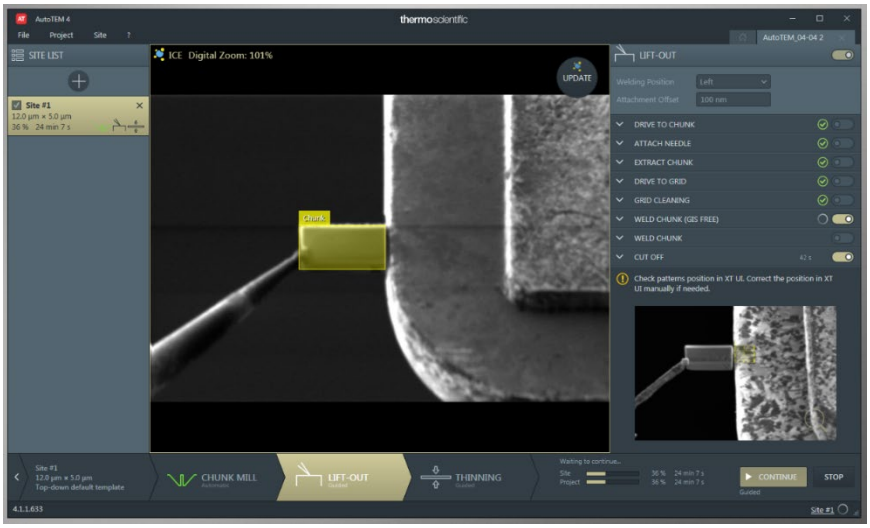


The top of the grid is identified by the user.



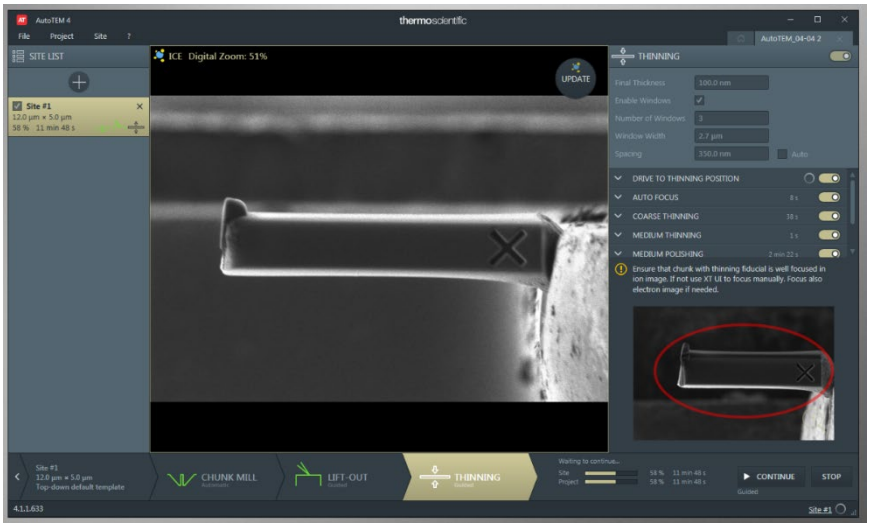
The user indicates where the lamella is to be bonded.

The user positons the needle with respect to the copper grid.



The user again defines the position of the sample.

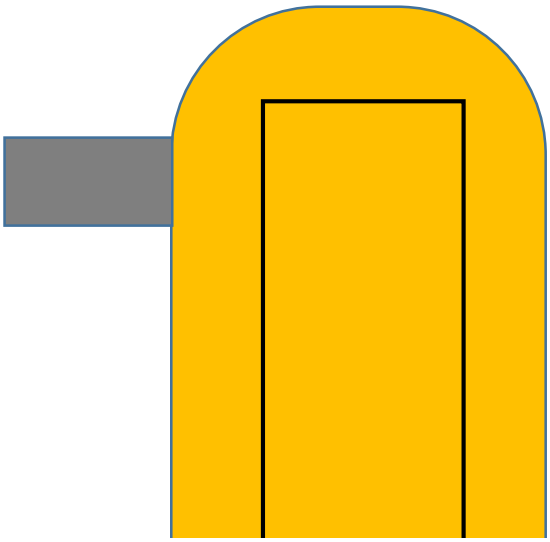
The lamella is automatically bonded to the copper grid without platinum.



The sample is now mounted on the grid.

The sample is thinned manually.

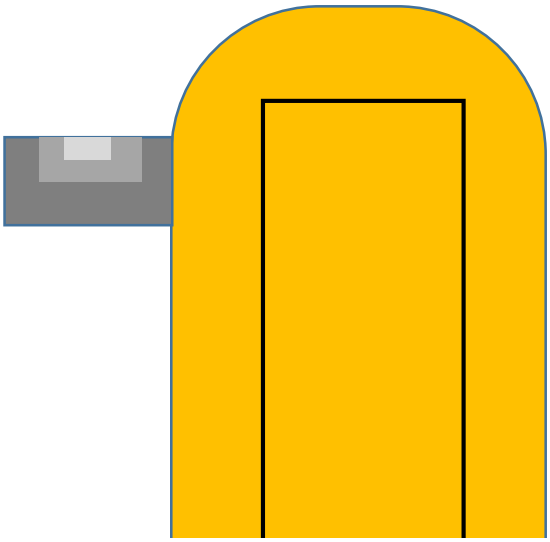
Once mounted, the sample will look like this when viewed sideways.

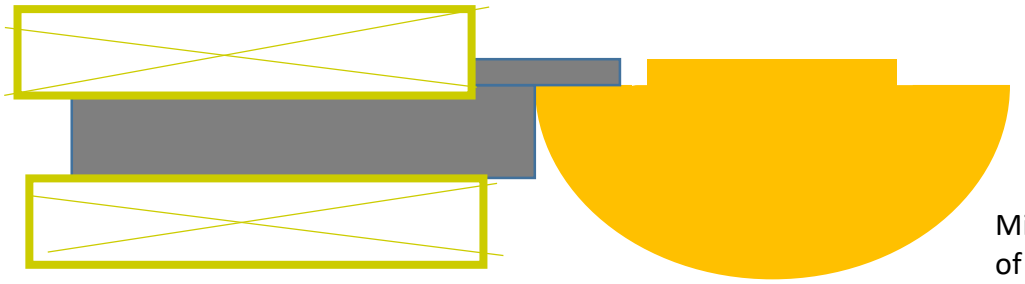


When finished, the sample will look something like this. There is a lot of variation from sample to sample.

Instructions to reach this point are on the following pages.

Note that this is a process that requires a lot of decisions as you mill. It is very sample-dependent so it is best learned while training. As a result, the instructions that follow are very general.

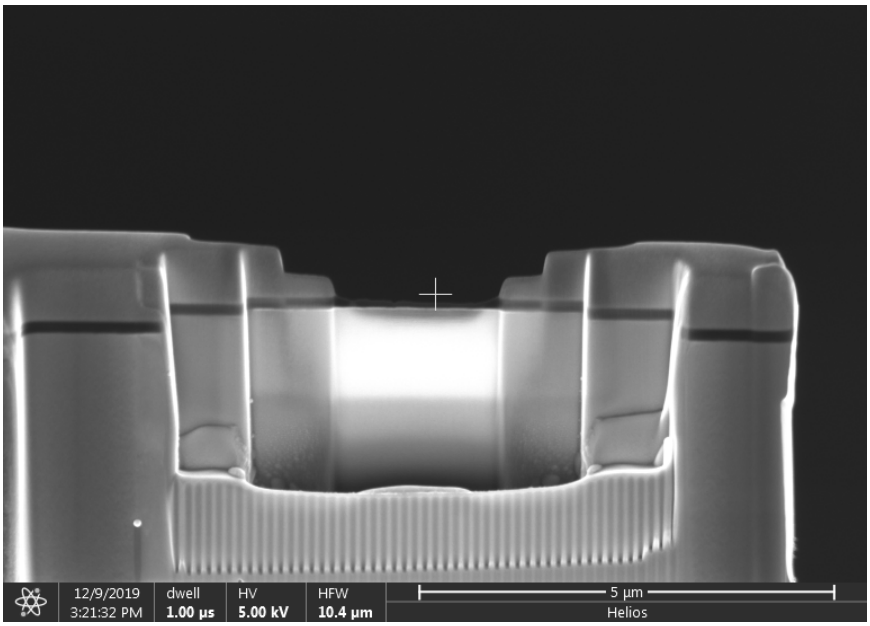




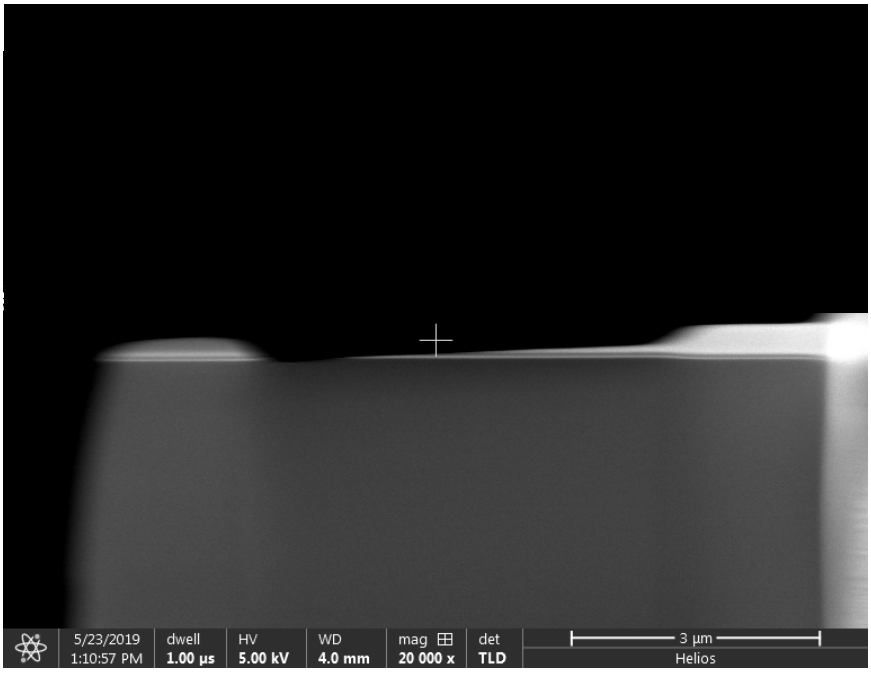
Mill both sides of the sample with a 30keV 1.2mA ion beam. You should just barely clean off both sides.

Increase the angle to about 54 degrees (sample-dependent) Lower voltage to 5keV 0.11 mA and gradually thin down the center portion. You do not need to go all the way to the bottom of the sample

Using 5keV 0.11 mA and gradually thin down the center portion.



Final sample thickness may look like this



Or this