

FEI Helios G4 PFIB CXe standard operating procedure

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ANALYSIS OF RADIOACTIVE SPECIMENS IS STRICTLY PROHIBITED

What this document covers: an overview of basic operation of the instrument including loading/unloading of specimens, turning on/off the beams, setting proper specimen height, basic alignments, basic stage manipulations, basic imaging, executing and basic manipulation of milling patterns, Pt deposition, EasyLift manipulation, and proper idling of the system.

1. Preliminary sample preparation

- 1.1 Prior to I-beam exposure, samples may need to be coated with a conductive layer of sufficient thickness to prevent surface damage during the initial stages of I-beam imaging and protective I-beam Pt strap deposition. If surface preservation is not needed and the specimen is conductive, this is not necessary. However, this is necessary if the specimen is insulating, even if surface preservation is not required because it will be impossible to get stable images of an insulating specimen otherwise. In-situ electron-beam induced Pt or ex-situ conductive C and Cr work well for this application (again, if surface preservation is required, the coatings need to be sufficiently thick); Au-Pd is not recommended for a protective layer since it is known to become porous upon exposure to the I-beam.

2. Sample mounting

- 2.1. Samples should be mounted on standard SEM pin stub mounts (Ted Pella #16111, diameter ~13 mm or Ted Pella #16144, diameter ~25 mm) using carbon tape, conductive sticky tabs, or well-dried conductive paint (best option). The height to footprint ratio of the specimens should be kept as small as possible to ensure specimen stability. Ideally, specimen thickness should not exceed ~10 mm (prior to mounting on stubs). If you are unsure about the suitability of the geometry of your specimens for loading, please ask for staff assistance to before attempting to load your specimens.

3. The xT software environment

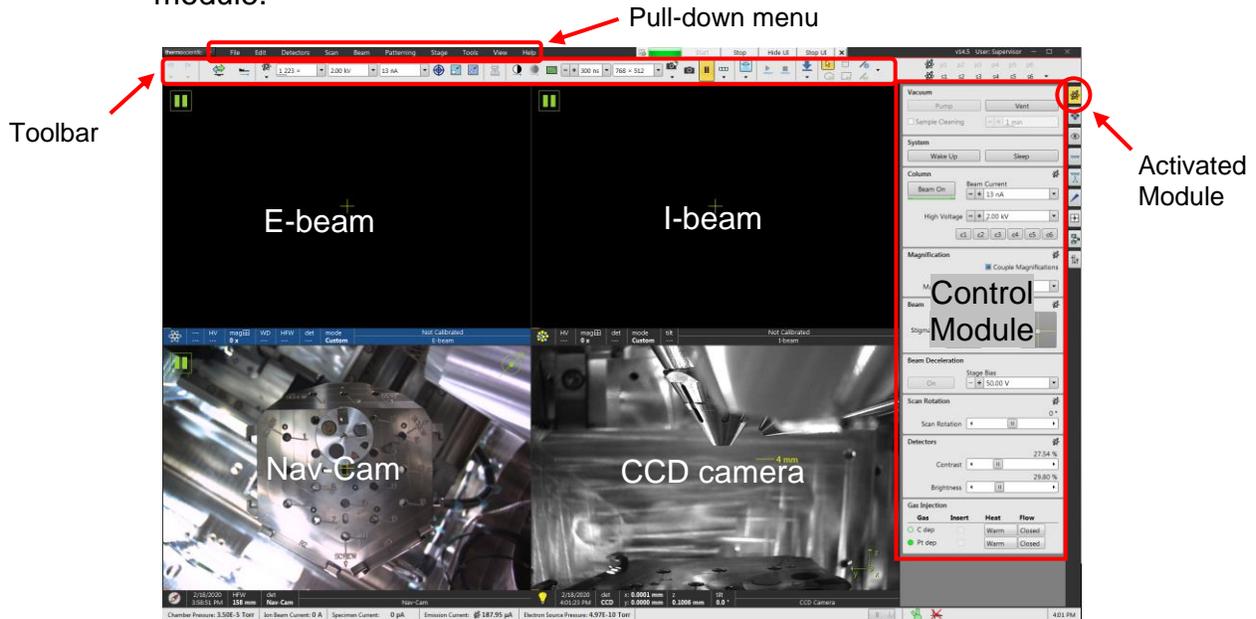
3.1. Log on to the TUMI system (you cannot proceed further until this is done).

3.2. xT should already be open and running; if there is a log on dialogue box present, use “Supervisor” (no quotations) for the password.

3.3. The xT software consists of four imaging quads labeled 1 – 4, a control module running vertically along the right, and a toolbar and pull-down menu.

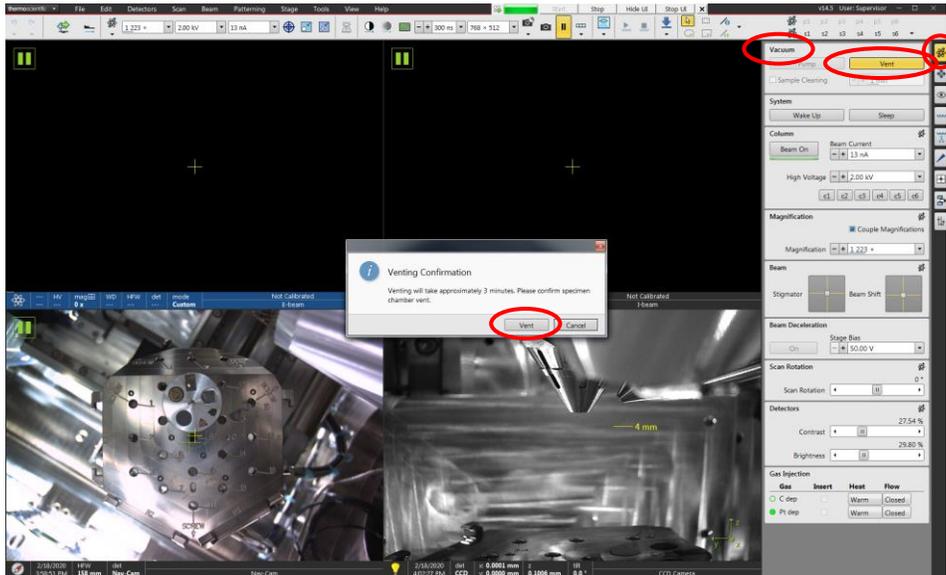
3.4. Quad 1 is used for E-beam imaging, quad 2 is used for I-beam imaging, quad 3 displays the Nav-Cam image of the stage, and quad 4 is a live image from the chamber-mounted CCD camera. Selecting a quad will activate the controls for that particular beam/image/camera (the databar will turn blue).

3.5. Selecting the different icons to the right of the control module will change the module.

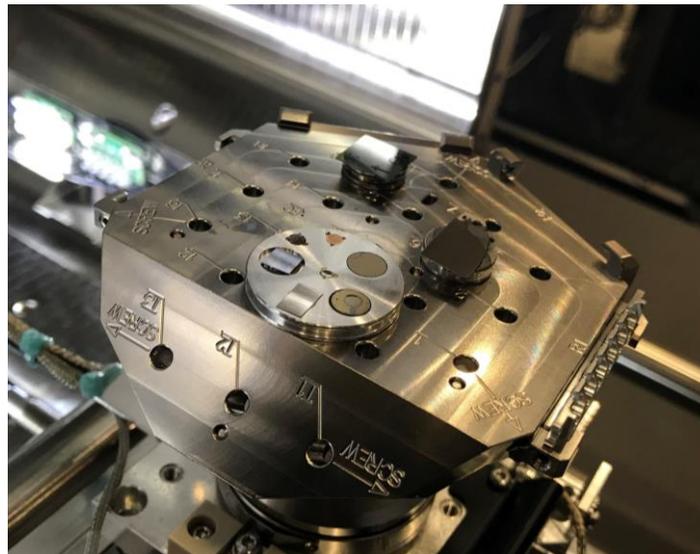


4. Sample loading

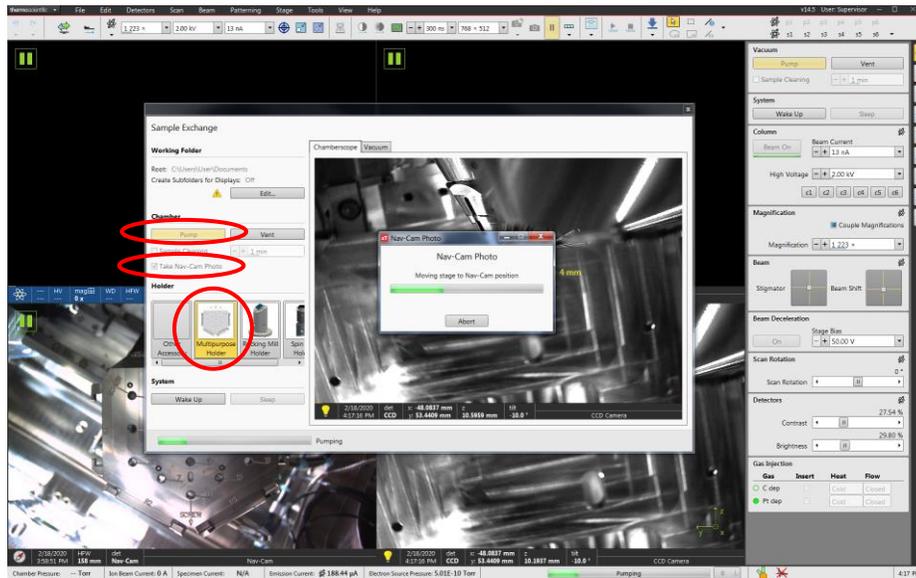
- 4.1. Enter the beam control module; in the “Vacuum” panel, select “Vent” to start venting the chamber (a dialogue box will pop up asking you to confirm venting); when venting is complete, gently open the chamber door until a firm stop is felt.



- 4.2. The instrument stage is equipped with a Multipurpose holder. You must wear gloves when loading/unloading the holder. The Multipurpose holder can accommodate several pin stubs and TEM row holders (with each row holder capable of holding up to 6 TEM grids). The stubs do not need to be secured in place unless loaded into one of the three positions labelled with “SCREW”; however, it is necessary to secure each TEM row holder using a screw.

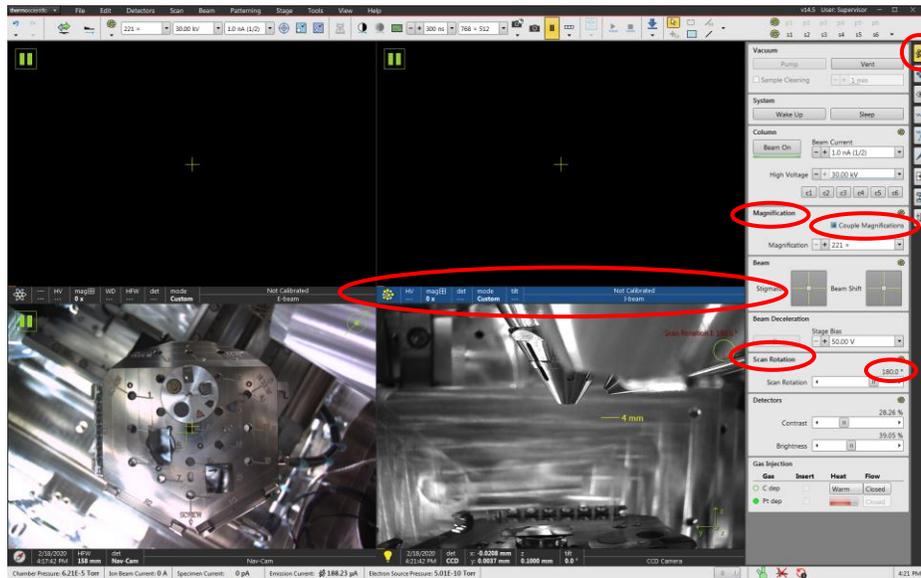


- 4.3. Once all stubs and/or TEM row holders are loaded, close the chamber door (make sure it is flush with the edge of the chamber); in the “Sample Exchange” window, verify “Multipurpose Holder” is selected and check the box next to “Take Nav-Cam Photo”, and then select “Pump”; apply pressure to the chamber door during the initial stage of evacuation to ensure a good seal; a Nav-Cam photo of the stage will be acquired automatically during evacuation.

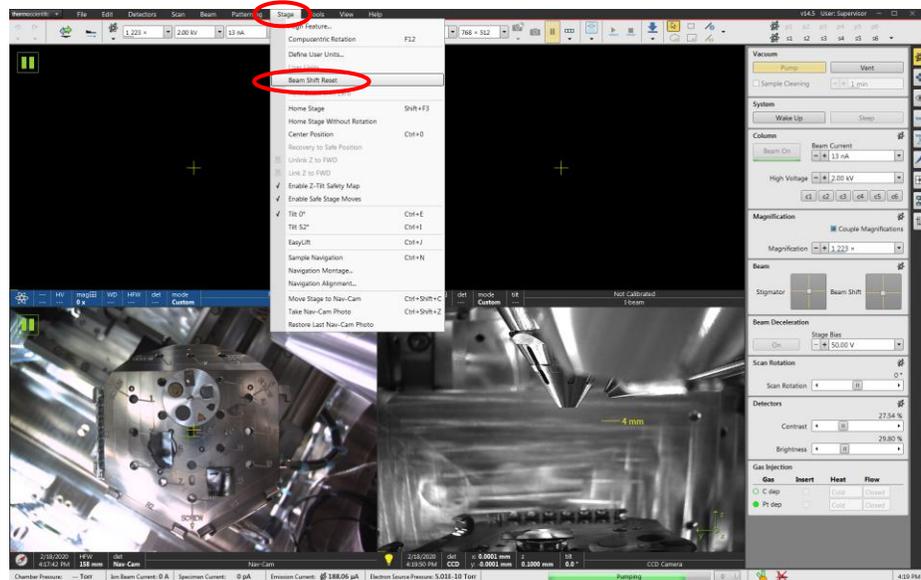


5. During evacuation

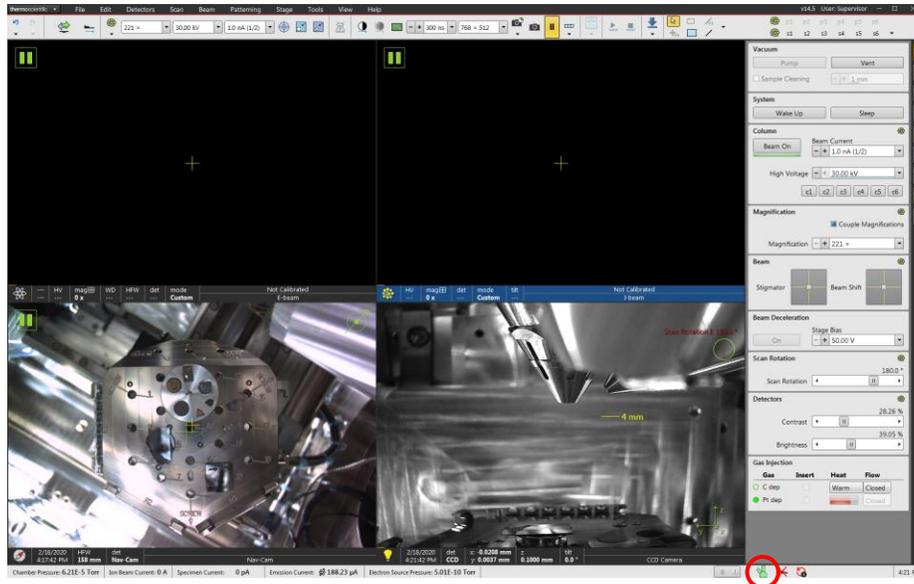
- 5.1. Activate the I-beam quad and then enter the beam control module; in the “Magnification” panel, verify “Couple Magnifications” is checked; in the “Scan Rotation” panel, set “Scan Rotation” = 180°; this rotates the I-beam image 180° relative to E-beam image and preserves the top-down orientation of the I-beam image when the stage is tilted.



- 5.2. Activate the E-beam quad. From the pull-down menu, select “Stage” and then “Beam Shift Reset” to reset the beam shift. Activate the I-beam quad and repeat to reset the beam shift for the I-beam.

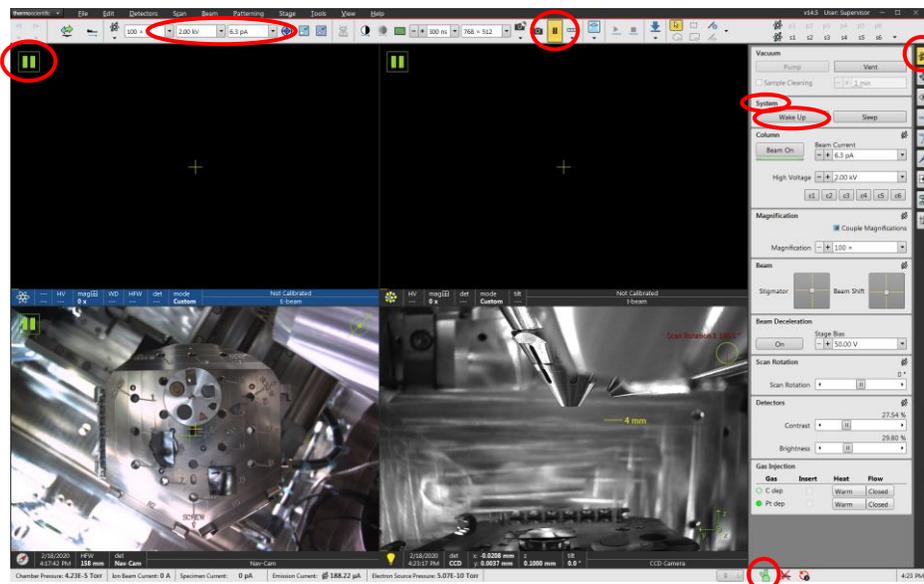


- 5.3. Check vacuum status indicator at the bottom of the window to determine if suitable vacuum has been reached to proceed. The chamber icon will be green when ready.



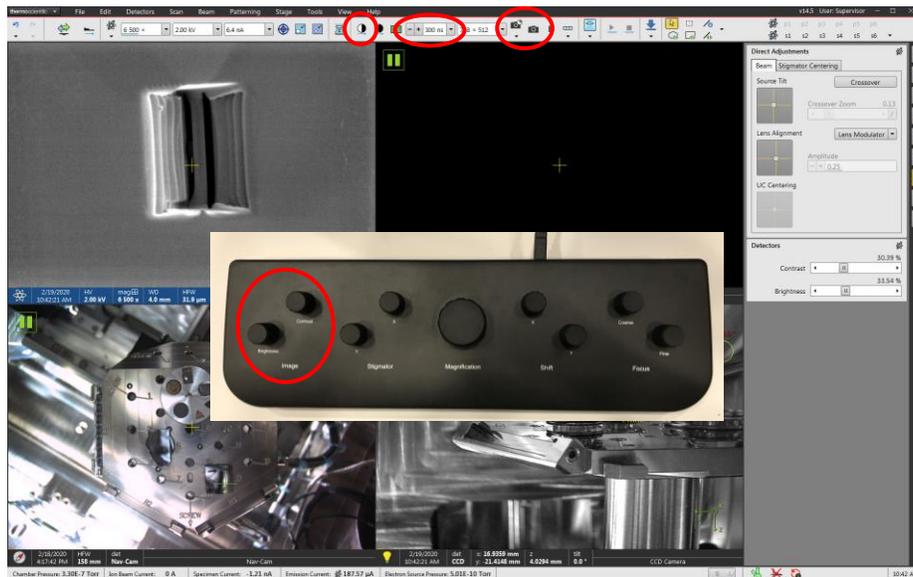
6. Turning on the beams, adjusting settings, live imaging

- 6.1. The beams can be turned on when the chamber icon at the bottom of the window is green. Activate either the E-beam or I-beam quad and enter the beam control module. In the “System” panel, select “Wake Up”.
- 6.2. Along the left side of the toolbar is the beam voltage and current *for the beam currently activated*. Clicking on either of these numbers pulls up a list of possible options for each parameter, which can be adjusted as needed.
 - 6.2.1. 2 kV is the recommended E-beam voltage for typical imaging applications.
 - 6.2.2. 30 kV is the recommended I-beam voltage for typical patterning or milling applications; however, this may be reduced during C or Pt strap deposition and/or the final stages of lamella prep (see separate lamella prep recipe).
- 6.3. If a live image is not actively being acquired for a particular quad, a pause icon will appear in the upper left corner of said quad. To start live imaging, activate the quad and select “Start Image Acquisition” (pause icon) from the toolbar; to stop the live image, select the same icon.



7. Snapshots, and photos

- 7.1. If the live image appears faint or otherwise difficult to see, contrast and brightness on the manual user interface (MUI) can be manually adjusted until satisfactory, or automatically by selecting “Auto contrast/brightness” (white/black circle icon) from the toolbar. *It will usually be necessary to adjust contrast and brightness every time a beam setting (voltage and/or current) is changed or the stage tilt is changed. Also note that auto contrast/brightness can only be used during live imaging.*
- 7.2. The dwell time used to acquire a live image can be adjusted from the toolbar; the response of the image will be faster using low dwell times, but at the expense of a poorer signal to noise ratio; *300 ns is a recommended default dwell time for live imaging.*
- 7.3. “Snapshot” (low quality) and “Photo” (high quality) images of the area being imaged can be acquired by selecting the respective icons (camera icons) from the toolbar. Once a snapshot or photo is complete, the live image will stop. The default settings for snapshots and photos are usually sufficient for most applications; if you wish to temporarily change the settings for your session, please consult staff for assistance.



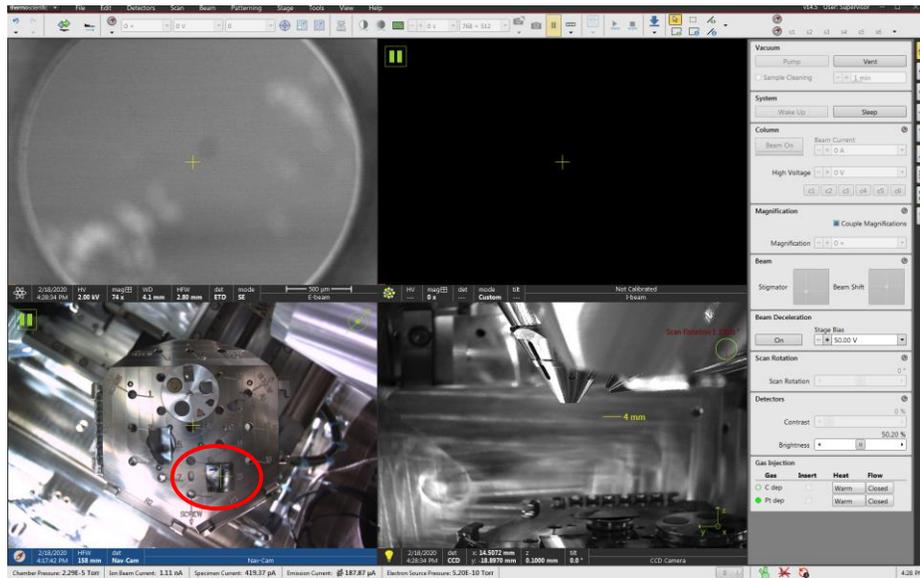
- 7.4. Any saving of snapshots or photos must be done on the *Support PC*. *Do not save any images on the Microscope PC*. To save an image, activate the image quad, then select “File” from the pull-down menu and then “Save As”. Navigate to the desktop and select “Shared Data” (you may create a folder in the directory for your images).

8. Finding a region of interest, linking Z to FWD

- 8.1. Activate the E-beam quad and start live E-beam imaging. Use the “Magnification” knob on the MUI to reduce the magnification to low as possible, which will make it easier to navigate around initially. You have three options for moving the stage (subsequently described):



- 8.1.1. Double left click on a position in the Nav-Cam photo to move that position directly under the E-beam (course movement); the green cross now corresponds to the stage position.

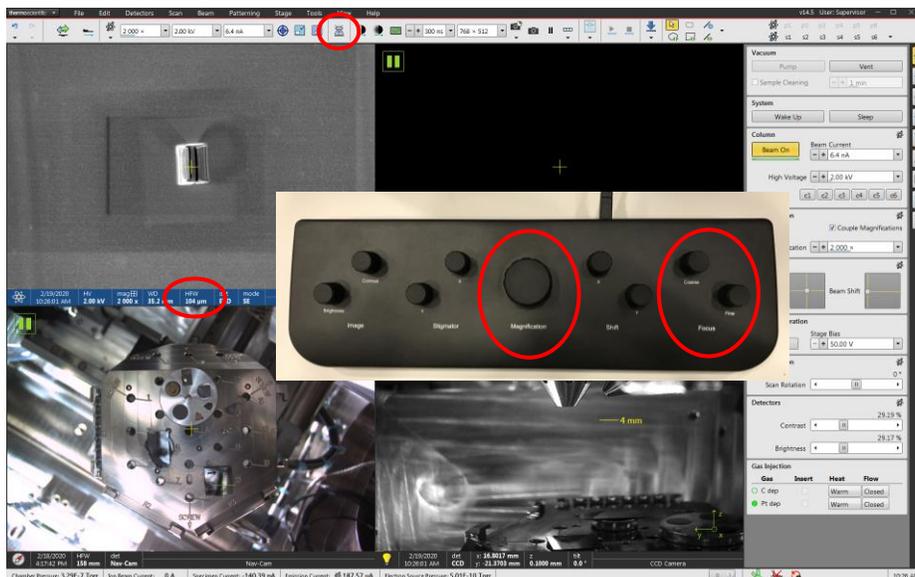


8.1.2. Click, hold, and drag in the live E-beam image using the mouse scroll wheel (a circle and arrow will appear in the image); release the scroll wheel to stop moving (medium movement)



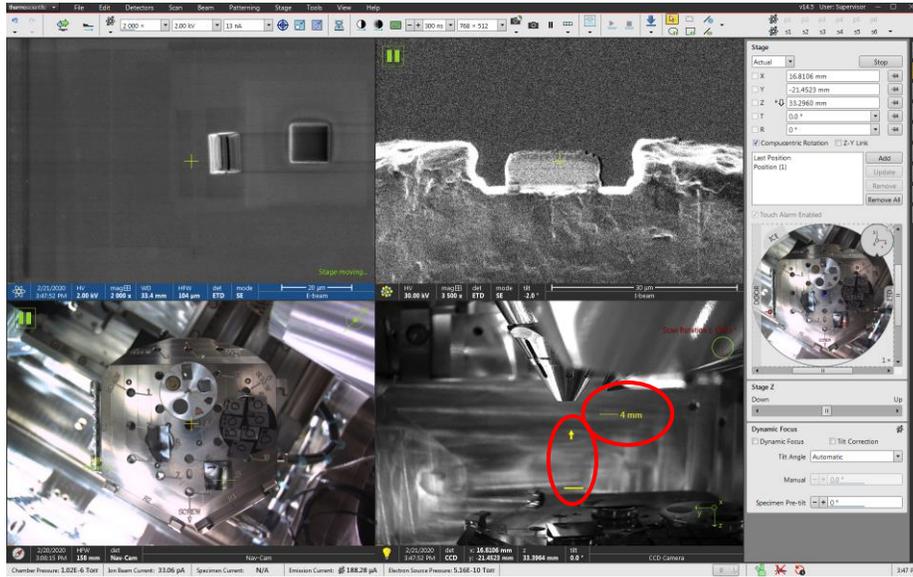
8.1.3. Double left click on a position in the live E-beam image to move that position to the middle of the E-beam image (fine movement);

8.2. Once the general region of interest has been located, use the “Magnification” knob on the MUI to set the horizontal field width (HFW) to ~100 µm and finely focus the live E-beam image using the “Focus” knobs on the MUI. When finished, select “Link Z to FWD” (UFO with question mark icon) from the toolbar to link the Z stage position with the working distance (WD).

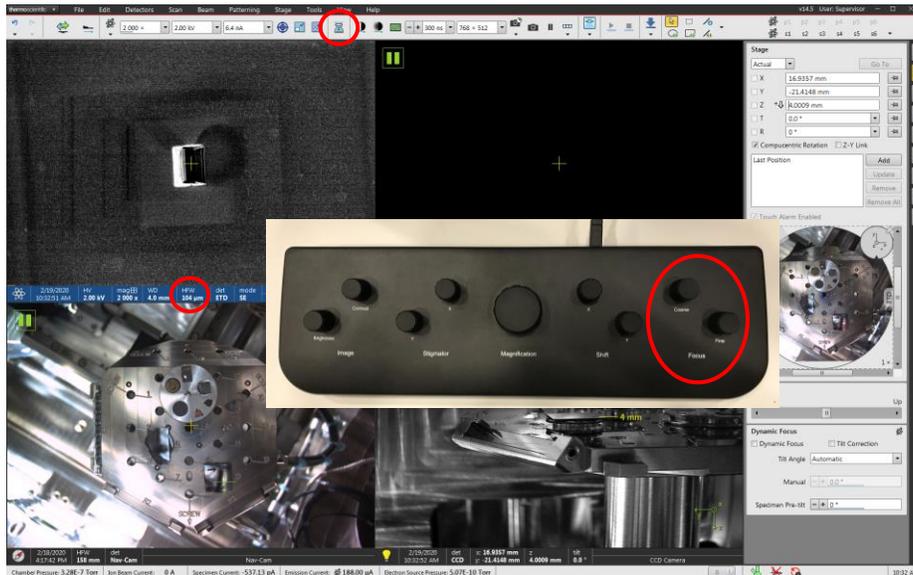


9. Setting eucentric height

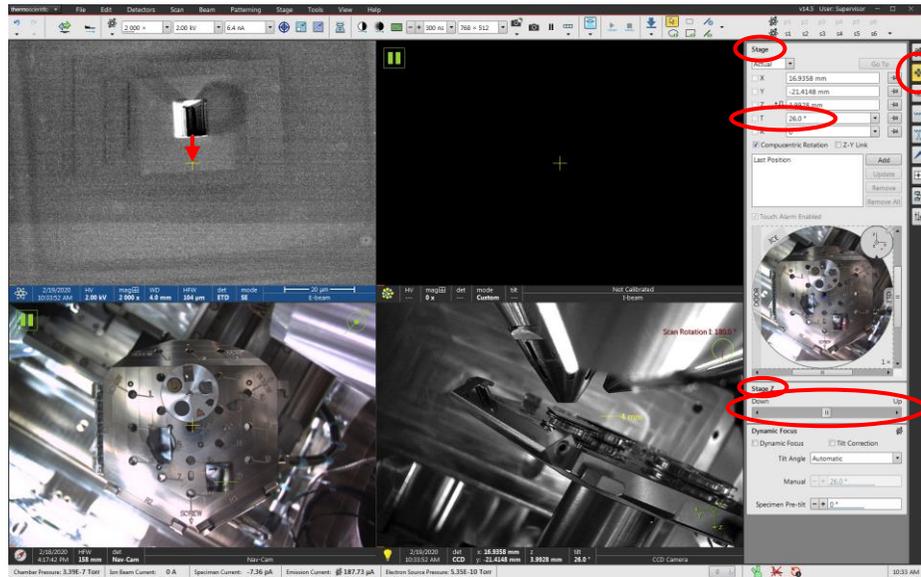
- 9.1. In the CCD camera quad, click, hold, and drag upward with the scroll wheel (an arrow and horizontal line will appear) and move the region of interest up up to approximately the same position as the 4 mm marker.



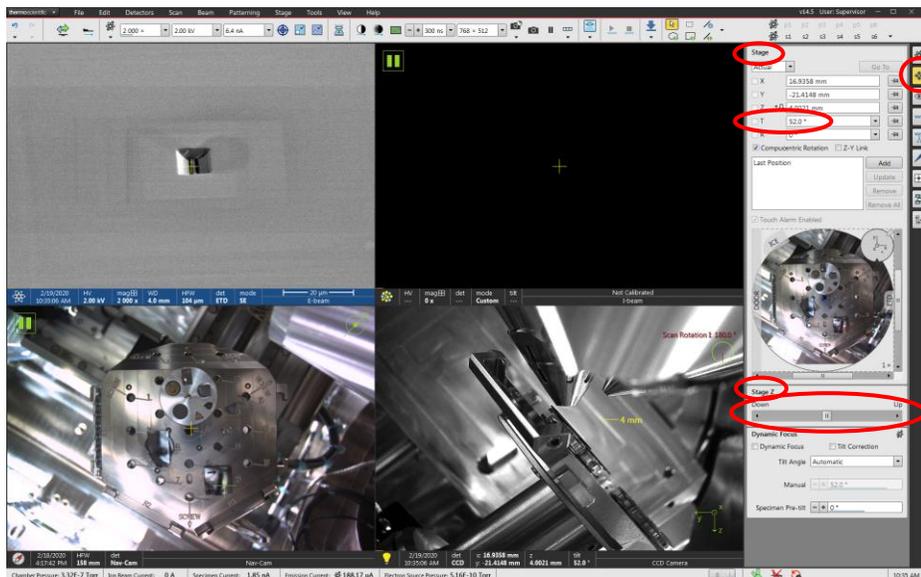
- 9.2. In the live E-beam image, center a recognizable feature by double left clicking in the image, set HFW ~100 μm , and finely refocus the image; select “Link Z to FWD” from the toolbar again.



- 9.3. Enter the navigation module; in the “Stage” panel, set T = 26°; use the “Stage Z” control to return the feature to the center of the live E-beam image.



- 9.4. Remain in the navigation module; in the “Stage” panel, set T = 52°; use the “Stage Z” control to return the feature to the center of the live E-beam image.



9.5. Go back to the “Stage” panel and return the stage tilt to $T = 0^\circ$; center the feature of interest in the image using a mouse double click; then refocus the image and select “Link Z to FWD” from the toolbar again.

9.5.1. After tilting back to $T = 0^\circ$, DO NOT use the “Stage Z” control to re-center the feature of interest.

9.6. Repeat the previous four steps for an additional iteration; if you did this process correctly, the feature of interest should barely shift (if at all) during the second iteration and the WD should be close to 4 mm.

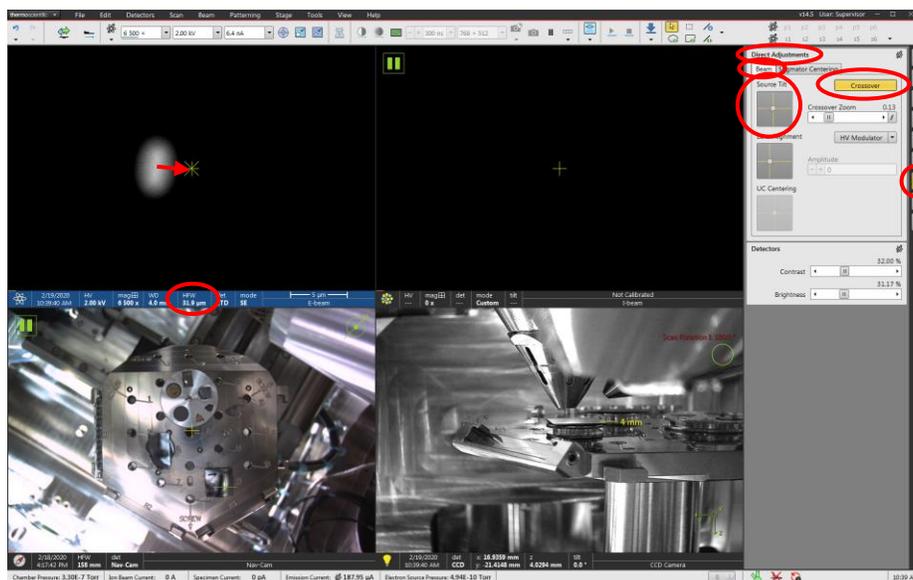
9.7. Eucentric height is now set for the current location; however, if any large stage moves are performed (moving between specimens, for example), you should reset eucentric height at the new location.

10. E-beam alignments

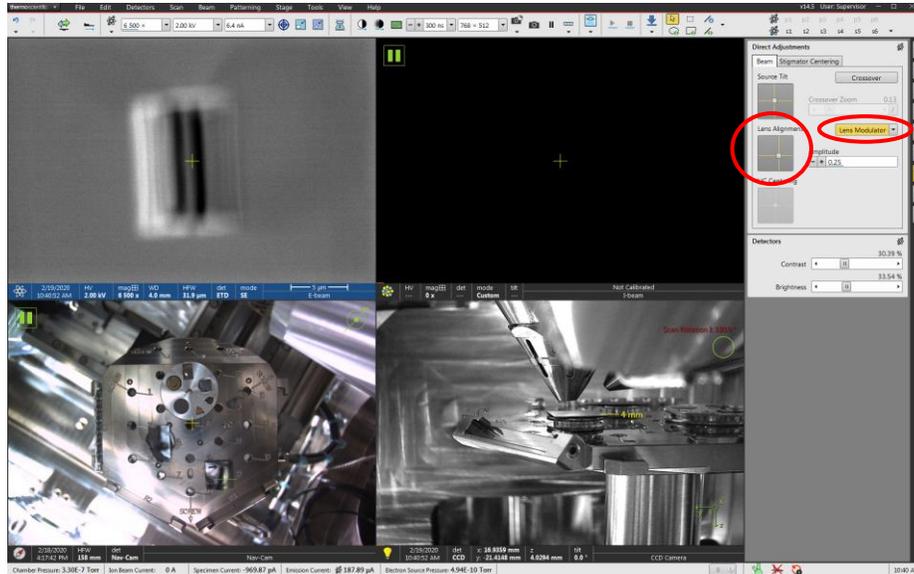
10.1. Make sure the E-beam quad is activated and you are acquiring a live image; you should be at eucentric height with stage tilt $T = 0^\circ$ before attempting any alignments.

10.2. Find a feature of interest; on the MUI, use the “Magnification” knob to set $\text{HFW} \sim 50 \mu\text{m}$ (or less), then use the “Focus” knobs to focus the image as sharply as possible.

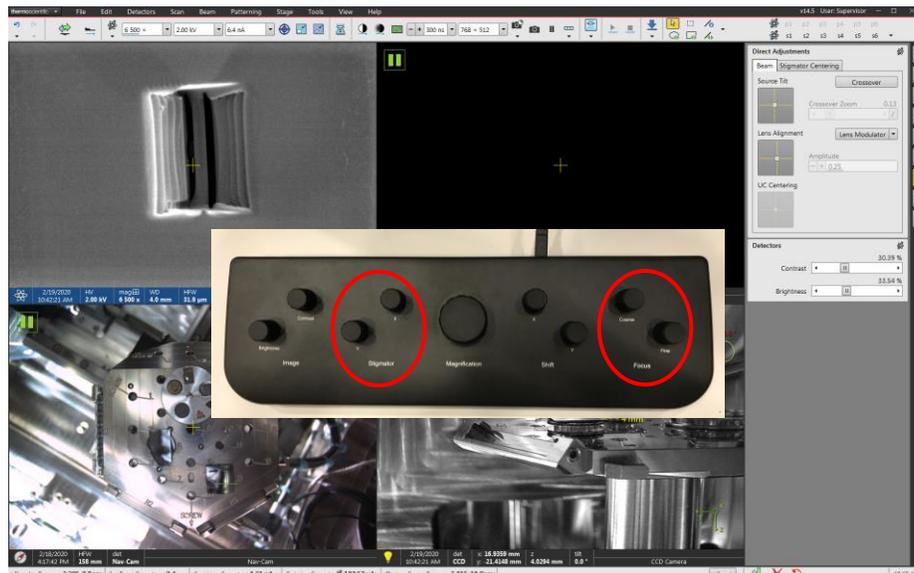
10.3. Enter the “Direct Adjustments” module; in the “Direct Adjustments” dialogue box; select the “Beam” tab and then “Crossover”; use the “Source Tilt” crosshairs to move the bright circle so it is centered on the center cross; select “Crossover” when finished to return to image mode



- Select “Lens Modulator” to start modulating the objective lens; the image should start going in and out of focus and/or shifting. Use the “Lens Alignment” crosshairs to minimize any shifting of the image; select “Lens Modulator” when finished and close the dialogue box.



- On the MUI, use the “Focus” knobs to focus the image as best as possible; then adjust the “Stigmator” knobs to obtain the sharpest image possible (you may then need to refocus and readjust the stigmators a few times to get the sharpest E-beam image).



- Note that alignment of the E-beam is sensitive to the chosen beam voltage and beam current settings; if one (or both) of these settings is changed the alignment will likely need to be adjusted again.

11. I-beam alignment

11.1. When operating at > 8 kV

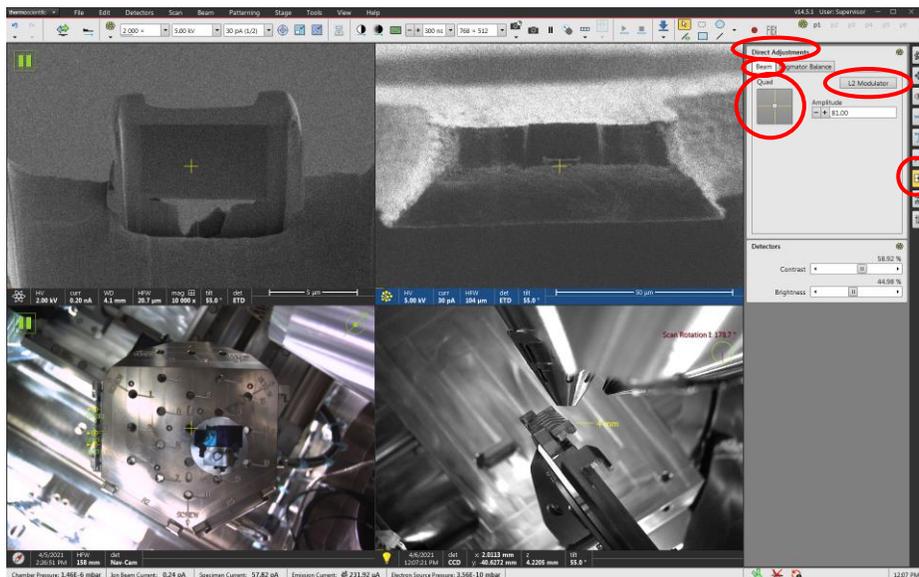
11.1.1. If the I-beam image appears clearly out of focus or astigmatic, you can start live I-beam imaging and use the MUI “Focus” and “Stigmator” knobs to obtain the sharpest image possible (at high beam currents, you want to do this as quickly as possible to minimize damage to the specimen); these adjustments should be minor.



11.2. When operating at ≤ 8 kV

11.2.1. Activate the I-beam quad, start live I-beam imaging, and enter the “Direct Adjustments” module; in the “Direct Adjustments” dialogue box; select the “Beam” tab and then “L2 Modulator”; the image will start going in and out of focus; use the “Quad” crosshairs minimize any shifting of the image; select “L2 Modulator” when finished

11.2.2. On the MUI, use the “Focus” and “Stigmator” knobs to obtain the sharpest image possible.



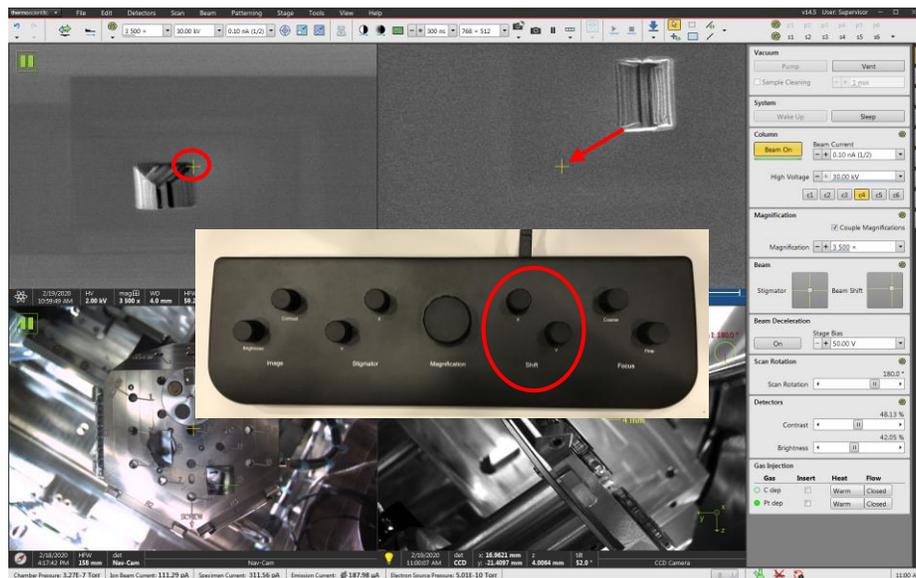
11.3. I-beam alignment is sensitive to the chosen beam voltage and beam current settings; if one or both settings is/are changed, quad alignment (if ≤ 8 kV), focusing, and/or stigmation may need to be adjusted again.

12. Linking the beams (I-beam shifting)

12.1. The area of interest must be at eucentric height before attempting to link the beams. Use the “Magnification” knob to set HFW $\sim 50 \mu\text{m}$, and center a feature of interest in the E-beam image by moving the stage (this should be in the near vicinity of the point where eucentric height was previously established); set stage tilt as needed (feature should remain basically centered).

12.2. Activate the I-beam quad and start live imaging; on the MUI, use the “Shift” knobs to center the same feature of interest in the I-beam image; remember that the I-beam image will be rotated 180° relative to the E-beam image.

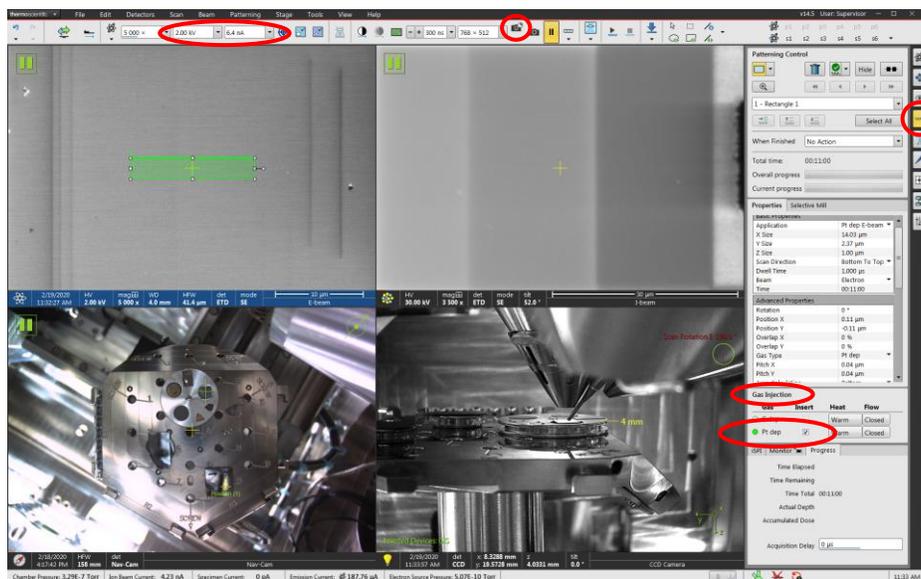
12.2.1. DO NOT move the stage to center the feature of interest in the I-beam image.



12.3. The link state of the beams is slightly sensitive to the stage tilt. In other words, if you change the stage tilt from $T = 52$ to $T = 0^\circ$, the I-beam shift will need to be (slightly) adjusted again. Likewise, the I-beam image may also slightly shift if the beam voltage/current are adjusted (though again, the adjustment to correct this shouldn't be extremely large).

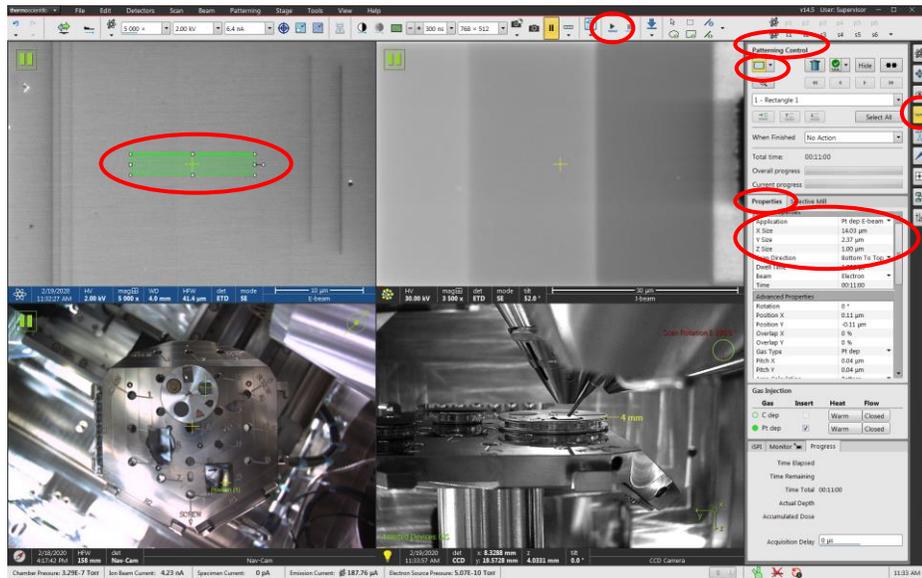
13. E-beam Pt deposition

- 13.1. NOTE: E-beam material deposition is limited to Pt only; it not practically possible to perform E-beam C deposition.
- 13.2. The region of interest where Pt deposition is to be performed should be at eucentric height; Pt deposition with the E-beam is typically performed for protective strap deposition, which should be done with stage tilt set to $T = 0^\circ$
- 13.3. E-beam Pt deposition is most effectively using low voltages and high currents (remember, if you change the E-beam settings the E-beam will need to be realigned). Enter the Patterning module; in the “Gas Injection” panel, check the box next to “Pt dep”; Activate the E-beam quad and acquire a snapshot of the region.
 - 13.3.1. Note: a beam voltage of 2 kV with a few nA of current is recommending for E-beam Pt deposition.

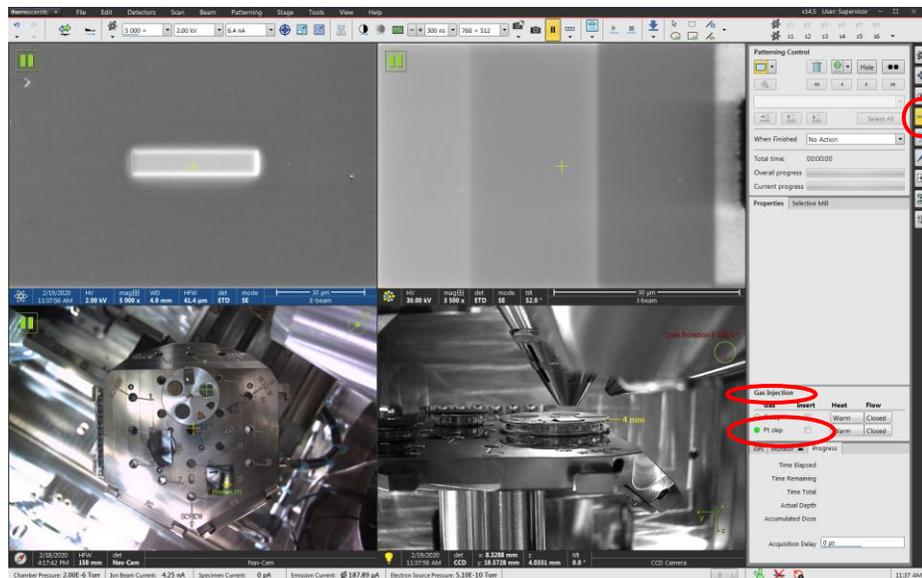


13.4. Remain in the patterning module. In the “Patterning Control” panel, select the rectangle pattern; you can then click and drag on the E-beam quad to draw the pattern. In the “Properties” tab, select the “Pt dep E-beam” application file and manually adjust the pattern dimensions as needed.

13.4.1. Select “Start Patterning” (play icon) from the toolbar to start the deposition

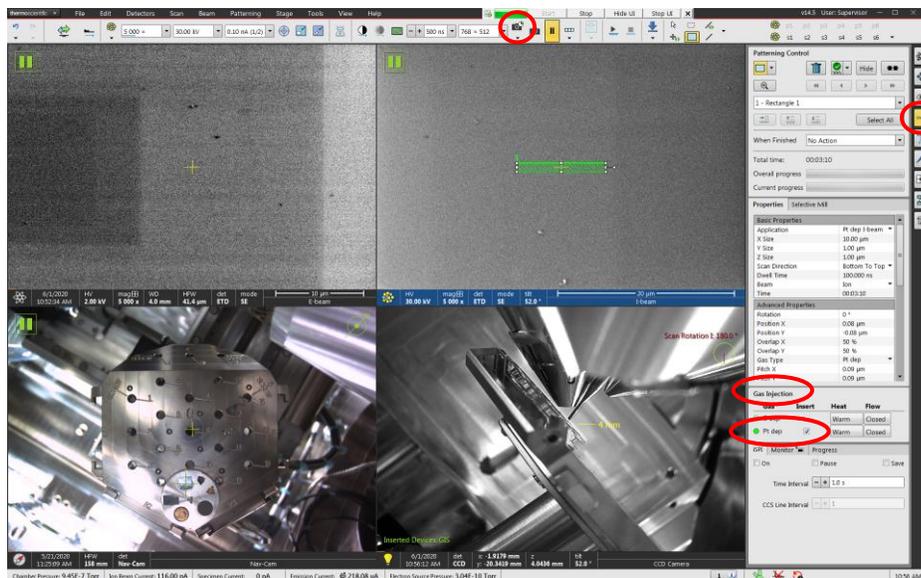


13.5. Remain in the patterning module; once the pattern is complete, click on the pattern in the quad and delete it; in the “Gas Injection” panel, uncheck the box next to “Pt dep”.



14. I-beam Pt or C deposition

- 14.1. When depositing Pt with the I-beam, a beam voltage of 12 kV is recommended for protective strap deposition; a beam voltage of 30 kV is recommended for all other Pt deposition scenarios (welding two objects together, etc.).
- 14.2. When depositing C with the I-beam, a beam voltage of 16 kV is recommended for protective strap deposition; it is otherwise not recommended to use I-beam deposited C for any other application.
- 14.3. The region of interest where C or Pt deposition is to be performed must be at eucentric height; if performing protective strap deposition, the stage tilt will usually be set to $T = 52^\circ$; if welding 2 surfaces together, the stage tilt will usually be set to (or close to) $T = 0^\circ$.
 - 14.3.1. Regardless of the material to be deposited or the beam voltage, an I-beam current density of $10 \text{ pA}/\mu\text{m}^2$ is recommended for optimal deposition; e.g., for a protective strap with area $10 \mu\text{m}^2$, use current = 0.1 nA. If the necessary current to obtain a current density of $10 \text{ pA}/\mu\text{m}^2$ is between two available values, opt for the lower value.
- 14.4. After selecting a current and focusing/stigmating as needed, activate the I-beam quad and acquire a snapshot of the region; enter the Patterning module; in the "Gas Injection" panel, check the box next to the chosen material ("Pt dep" shown here as an example); Activate the I-beam quad and acquire a snapshot of the region.

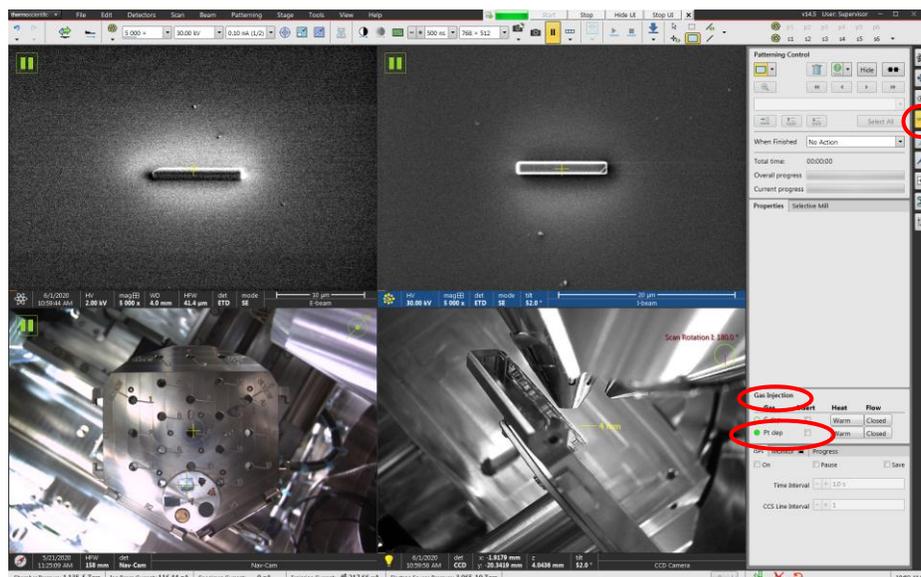


- 14.5. Remain in the patterning module. In the “Patterning Control” panel, select the rectangle pattern; you can then click and drag on the I-beam quad to draw the pattern. In the “Properties” tab, select the appropriate application file and manually adjust the pattern dimensions as needed (remember, the optimal current density for I-beam Pt deposition is $10 \text{ pA}/\mu\text{m}^2$); If depositing a protective strap, set “Scan Direction” to “Left to Right”.

14.5.1. Select “Start Patterning” from the toolbar to start the deposition.

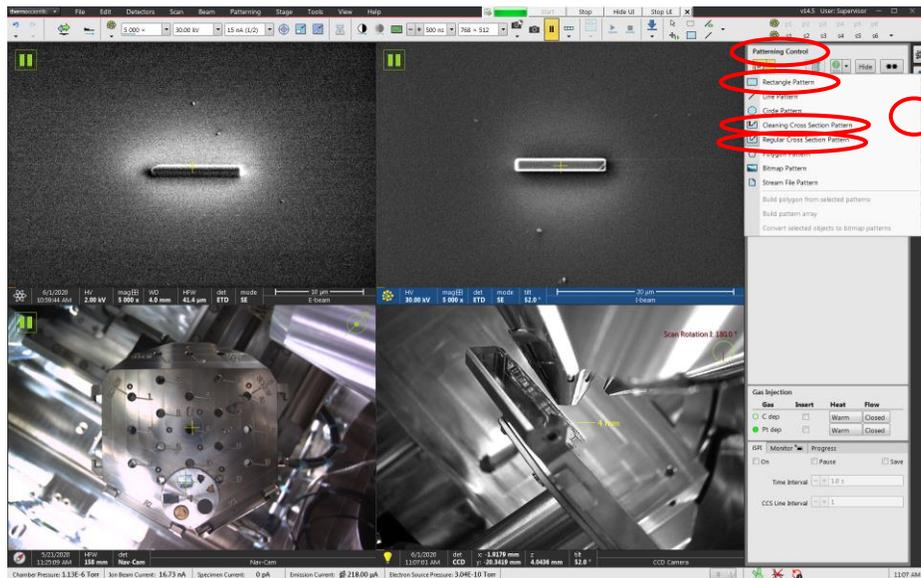


- 14.6. Remain in the patterning module once the pattern is complete; , click on the pattern in the quad and delete it; in the “Gas Injection” panel, uncheck the box next to the appropriate material; select the pattern in the I-beam quad and delete it.

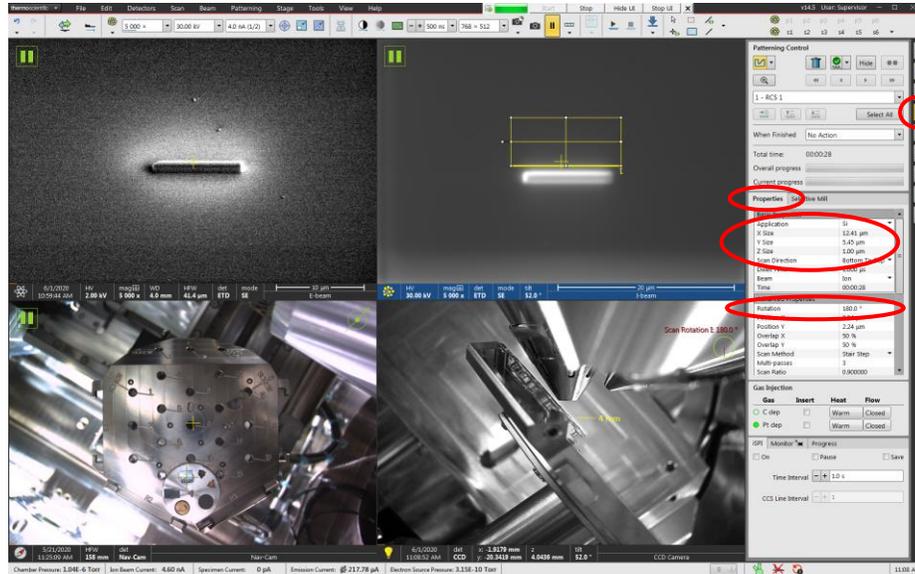


15. Milling

- 15.1. The region of interest where milling is to be performed must be at eucentric height with the beams linked before attempting milling or milling may not work correctly.
- 15.2. Enter the patterning module. In the “Patterning Control” module, under “Pattern”, there are several different options available. The most commonly used patterns during specimen preparation are the “Rectangle”, “Regular cross-section”, and “Cleaning cross-section” patterns. Once a pattern type is selected, click and drag on the I-beam quad to produce a pattern.

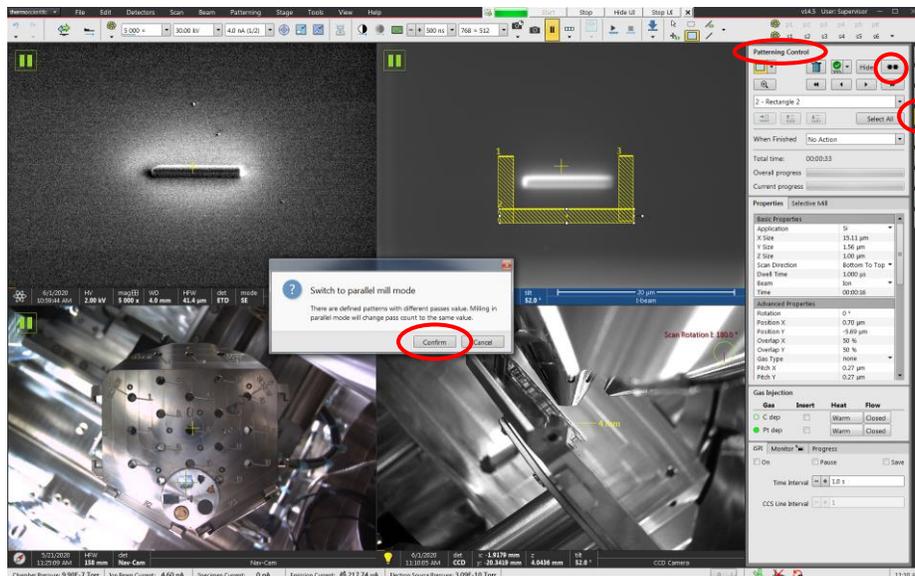


- 15.3. In the “Properties” tab, select the “Si, SiC, or Al₂O₃” application files (as appropriate) and manually adjust the pattern dimensions as needed. If the rotation of the pattern needs to be adjusted (relevant for regular and cleaning cross-sections), this can also be done in the “Properties” tab.

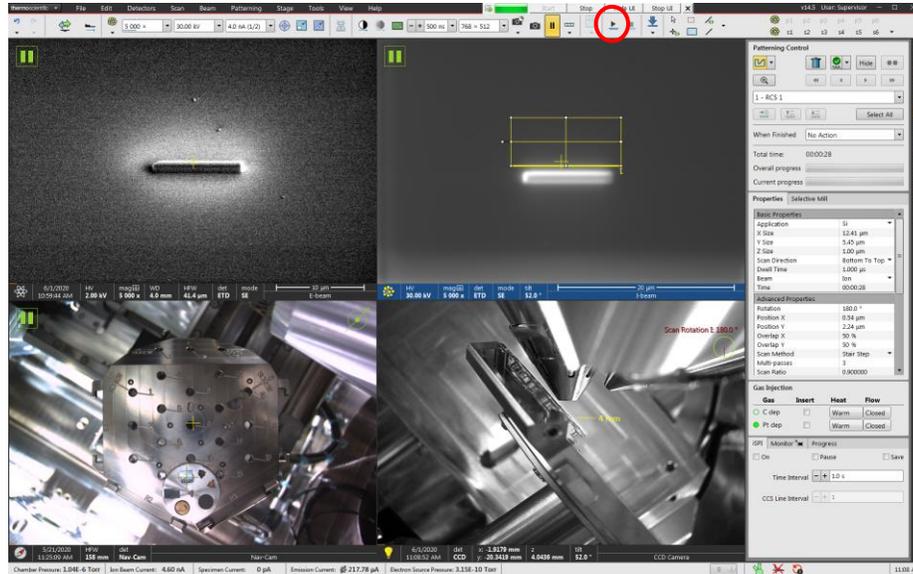


- 15.4. If more than one pattern was drawn, the order of execution can be changed in the “Pattern Control” panel (confirm changing of patterning order in the dialogue box that pops up) from serial to parallel.

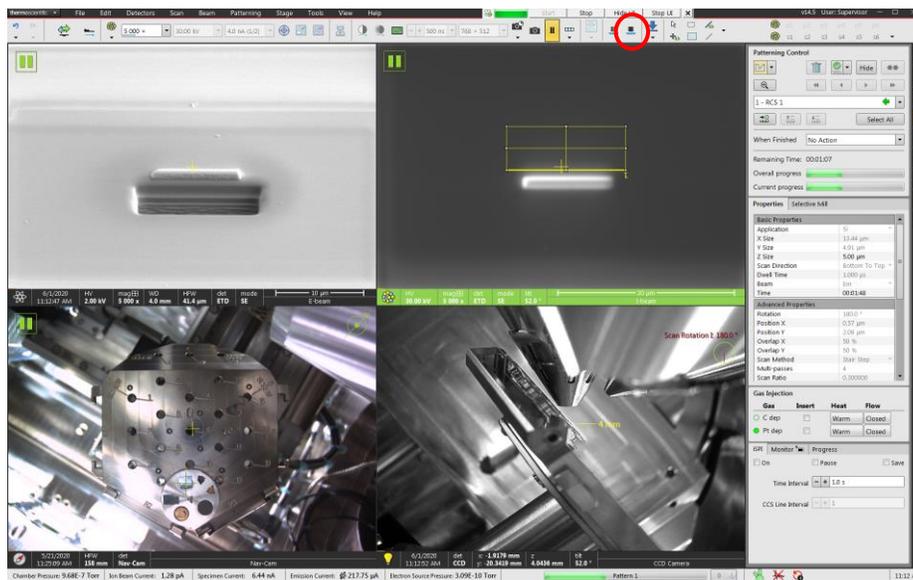
- 15.4.1. Note: if serial execution is selected, the patterns will be executed in the order of being drawn.



- 15.5. Once all patterns have been drawn and positioned/rotated as needed, take an I-beam snapshot; if any shifting of the image occurred, move the pattern(s) as needed. When ready, select “Start Patterning” from the toolbar to start milling.



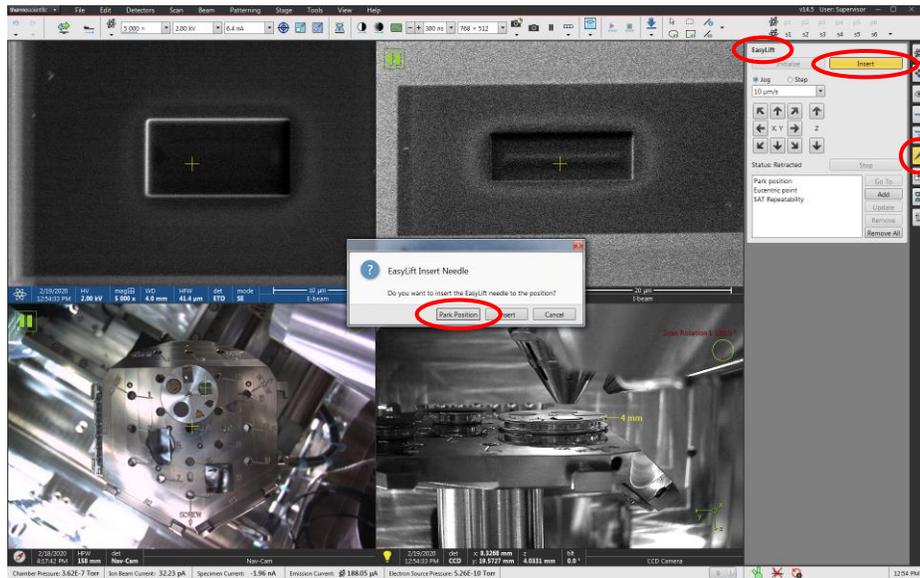
- 15.6. While milling is active, the “Start Patterning” icon on the toolbar will change from “play” to “stop”; if the pattern needs to be stopped prior to completion, select “Stop Patterning” (stop icon) from the toolbar.



16. Using the EasyLift

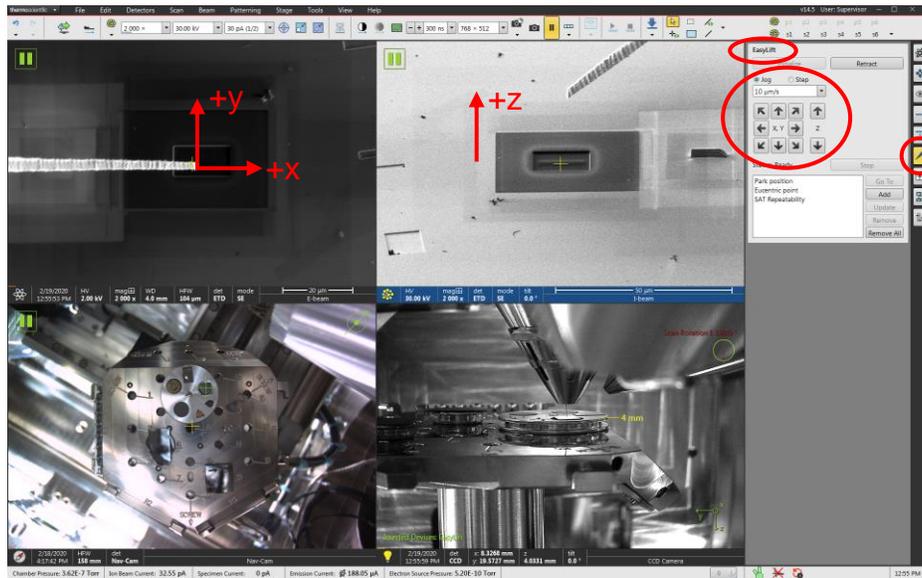
16.1. The region of interest must be at eucentric height with stage tilt $T \sim 0^\circ$ before attempting to insert the EasyLift;

16.2. Enter the EasyLift module; in the “EasyLift” panel, select “Insert”; in the dialogue box that pops up, select “Park Position”.



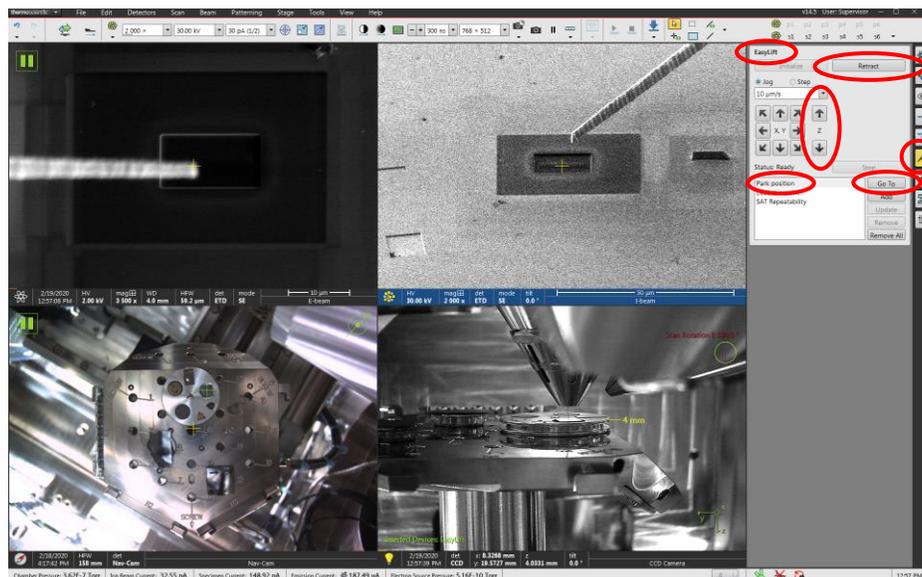
16.3. In the “EasyLift” panel, use the controls to adjust the speed and movement direction of the EasyLift. In the E-beam (I-beam) quad with scan rotation = 0° (180°), the EasyLift movement directions in the x, y, and z directions will correspond to same directions in the quads shown below.

16.3.1. It is best to use the live E-beam (I-beam) image when adjusting the x-y (z) EasyLift positions.



16.4. To retract the EasyLift, first use the z control to raise the EasyLift several µm above any nearby objects (grid, specimen surface, etc.); then select “Park Position” and “Go To”; once in park position, select “Retract”.

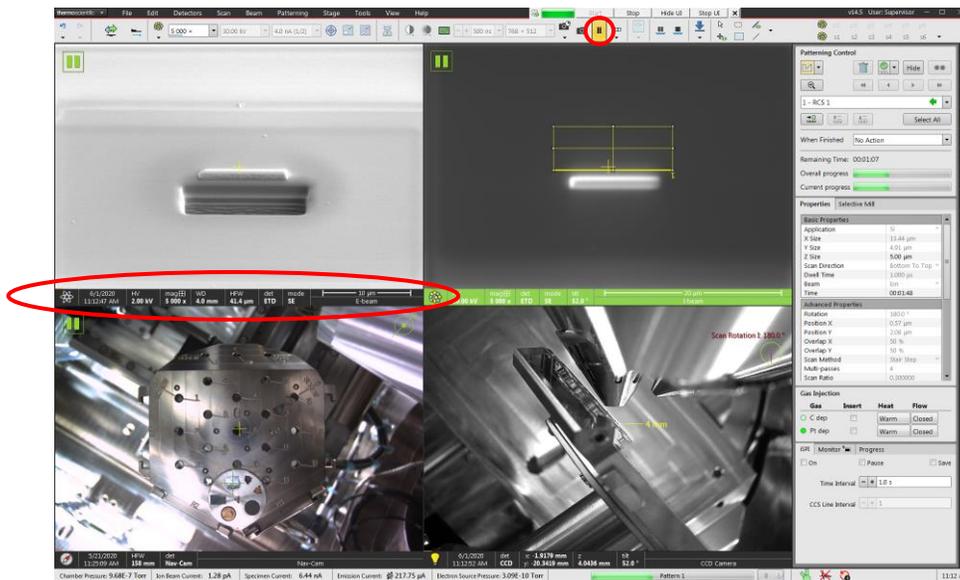
16.4.1. Do not retract the EasyLift unless is in the park position.



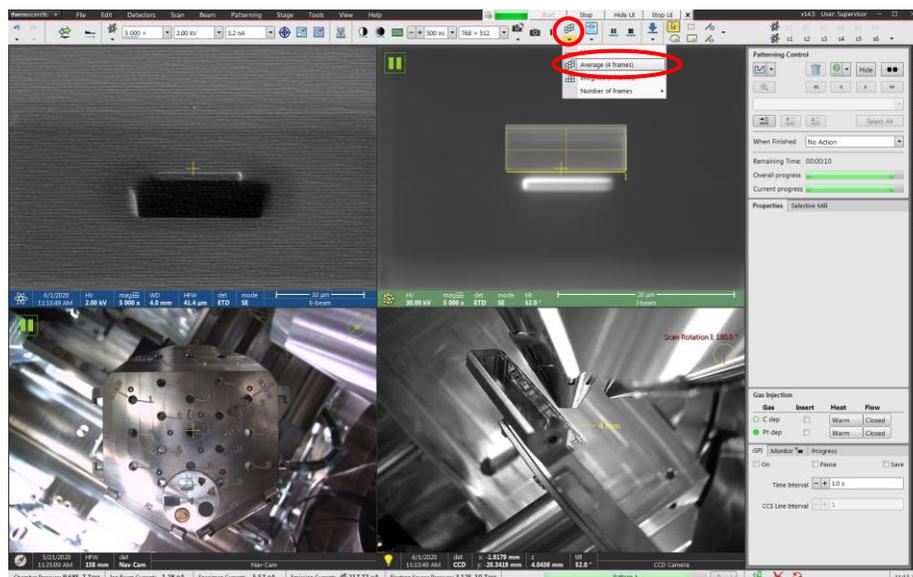
Live imaging while FIB patterning

16.5. To live E-beam image while patterning with the I-beam, simply activate the E-beam quad and select “Start Image Acquisition” from the toolbar to start acquiring a (probably noisy) live E-beam image during patterning. You may need to manually adjust contrast and brightness on the MUI to make the E-beam image more easily viewable.

16.5.1. The live E-beam image will have higher signal to noise the larger the ratio of E-beam current to I-beam current.

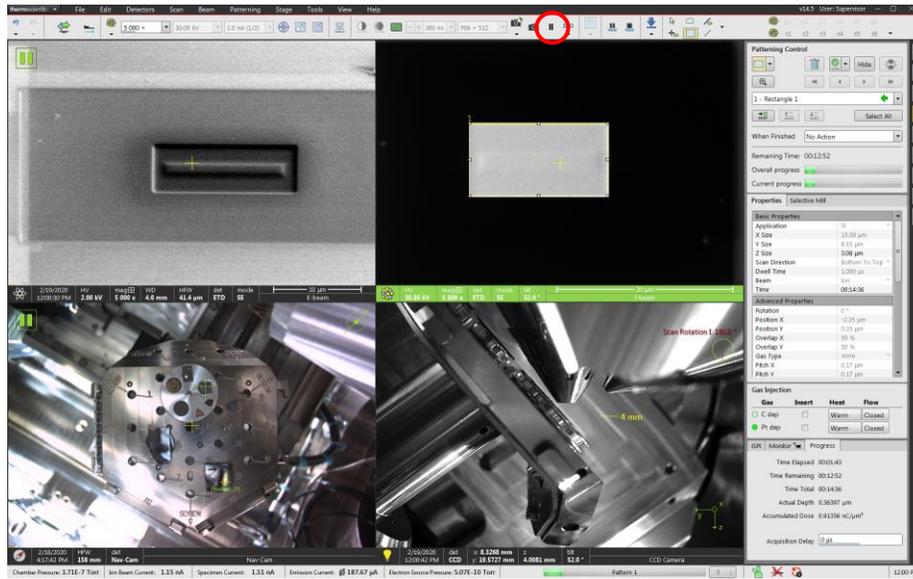


16.5.2. Turning on the “averaging” filter of the live E-beam image while FIB patterning is occurring will also make it easier to observe.



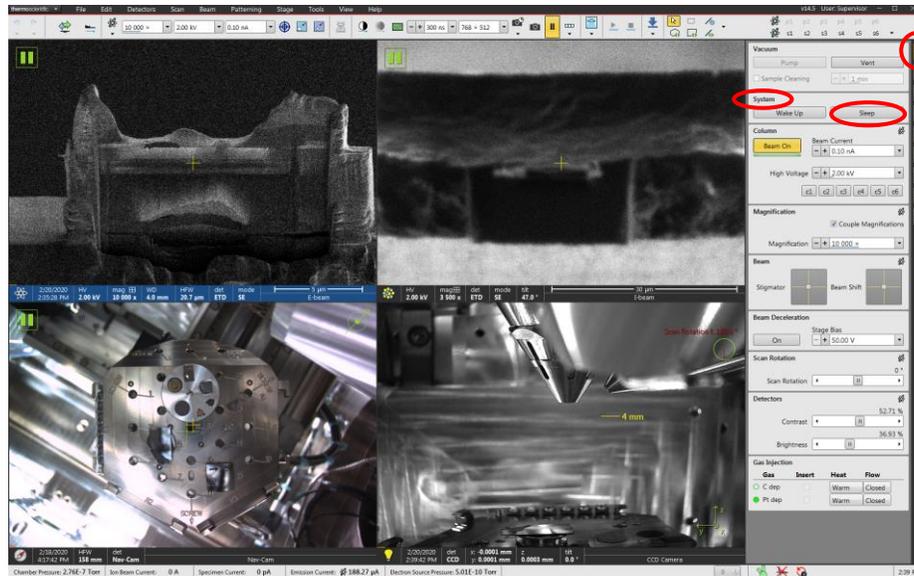
16.6. To live I-beam image while patterning with the I-beam, activate the I-beam quad and select “Start Image Acquisition”; a live signal from the region(s) being actively patterned will appear (not the whole image). If the patterned area is large enough, it may be possible to observe an actual image of the region. Again, contrast and brightness on the MUI may need to be adjusted so this can be observed.

16.6.1. Live I-beam imaging while patterning with the I-beam is generally only effective when executing large rectangle or regular cross-section patterns.

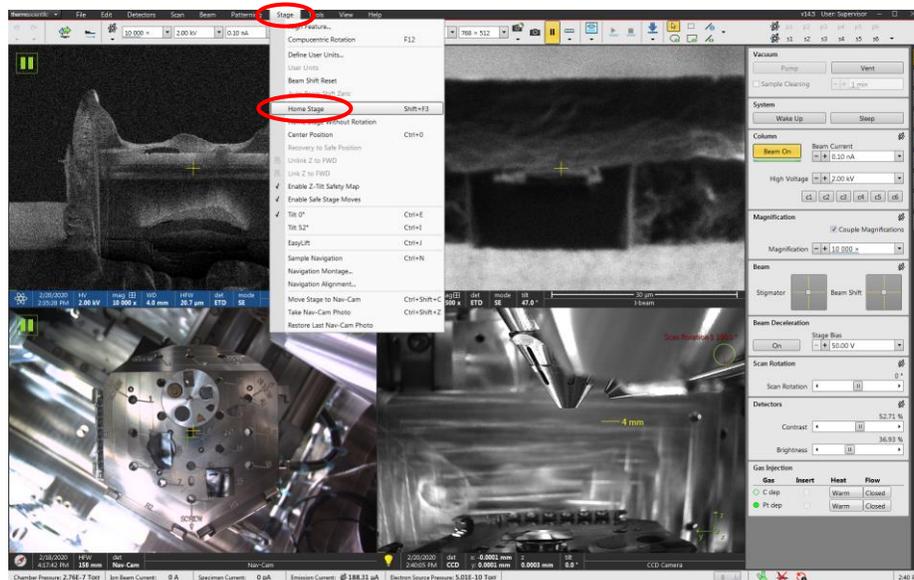


17. Finishing the session

17.1. Activate either the E-beam or I-beam quad and enter the beam control module. In the “System” panel, select “Sleep” to turn off both beams.



17.2. Select the “Stage” pull-down menu, and then “Home Stage”; wait until homing is complete before proceeding.



17.3. Enter the beam control module and vent the chamber (described previously); remove all pin stubs/TEM grid holders from the Multipurpose holder; return the chamber back to vacuum (described previously).