

## FEI Helios NanoLab 600 standard operating procedure

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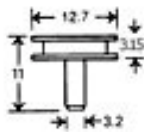
What this document covers: an overview of basic operation of the instrument including loading/unloading of specimens, turning on/off the beams, setting proper specimen height, basic alignments, basic stage manipulations, basic imaging, executing and basic manipulation of milling patterns, Pt deposition,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

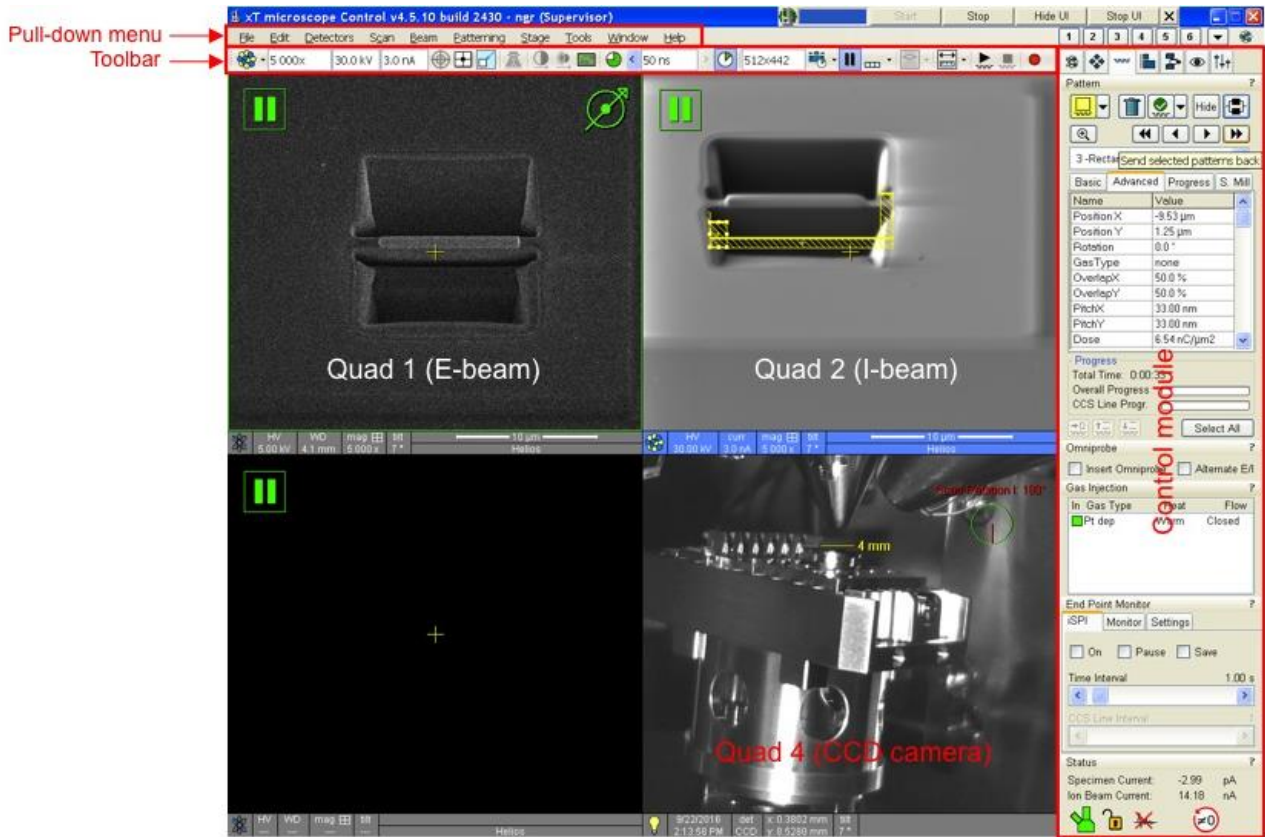
### 2. Sample mounting




- 2.1. Samples should be mounted on standard SEM pin stub mounts (Ted Pella product #16111) using carbon tape, sticky tabs, or well-dried conductive paint (best option). When mounted on one of these stubs, the total specimen thickness must not exceed 7 mm. If for some reason your specimens cannot fit within these mounting guidelines, please ask for staff assistance to help with determining alternative mounting methods 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 “user” (no quotations) for both username and 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.

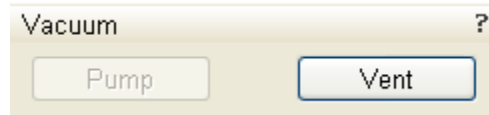


- 3.4. By default, quad 1 is used for E-beam imaging, quad 2 is used for I-beam imaging, quad 3 isn't used, and quad 4 is a live image from the chamber-mounted CCD camera. Selecting the E-beam or I-beam quad will activate control for that particular beam (the databar will turn blue).
- 3.5. Selecting the different tabs along the top of the control module will change the module. The most important modules are the first three: beam control , navigation , and patterning .

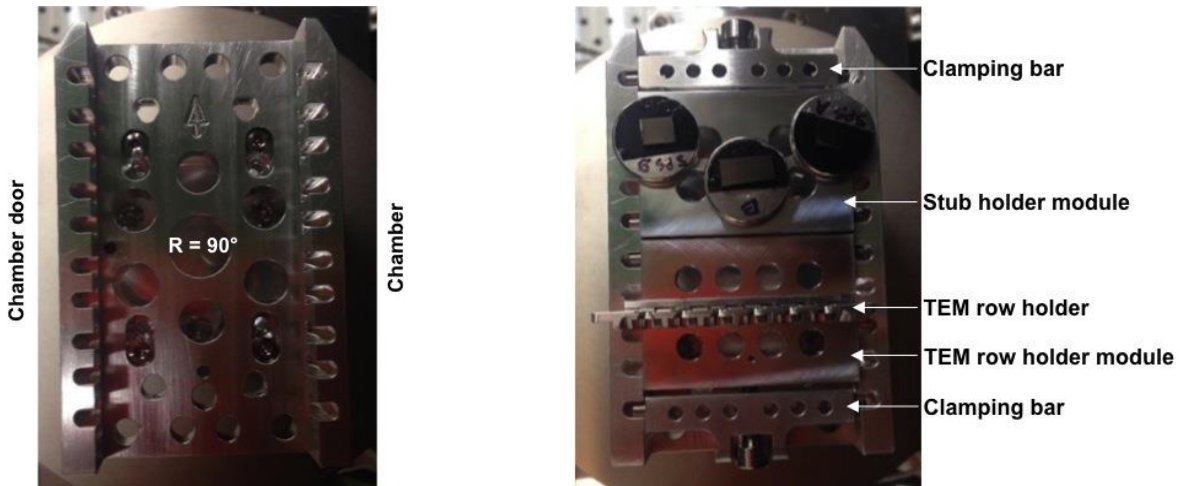


#### 4. Sample loading

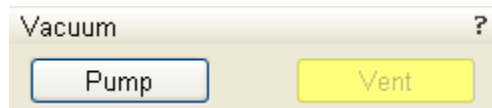
- 4.1. Enter the beam control module . Under “Vacuum”, select “Vent” to start venting the chamber (about 5 min to complete); when venting is complete, gently open the chamber door.



- 4.2. The instrument stage is equipped with a universal mounting base (UMB). You must wear gloves when handling the UMB or any of its components. The UMB can hold 1 or 2 modules, which are held in place using clamping bars. A stub module will accept 3 standard pin mount stubs, while a TEM row holder module will accept one TEM row holder (which itself can hold up to 6 TEM grids). Modules should be placed onto the UMB such that the pins on the modules fall into the fifth set of slots from either end of the UMB. Do not excessively tighten the clamping bars.

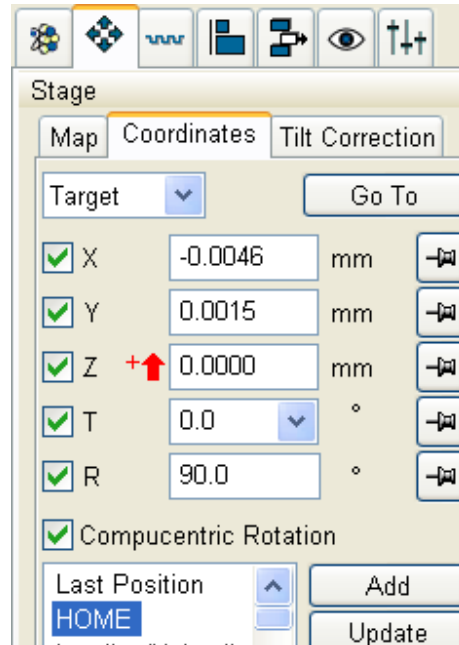



- 4.3. Once all modules are loaded onto the UMB and clamped into place, close the chamber door (make sure it is flush with the edge of the chamber) and select “Pump”; apply light pressure to the chamber door during the initial stage of evacuation to ensure a good seal.

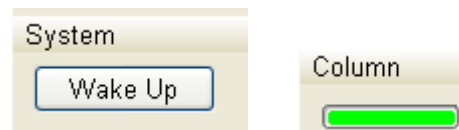



5. During evacuation

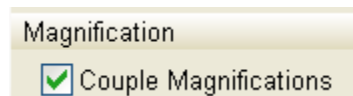
- 5.1. Enter the navigator module . Under “Stage”, select the “Coordinates” tab and double click on the stored “Home” stage position to drive the stage accordingly.




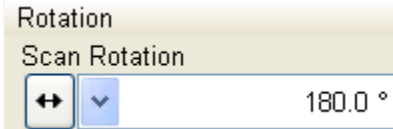
- 5.2. Activate the I-beam quad and enter the beam control module . Under “System”, select “Wake Up” to turn on the ion source. Under “Column”, the progress bar will turn green once the ion source is on (about 2 min to complete).



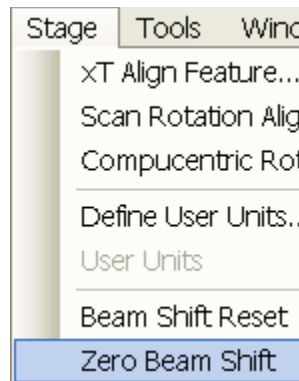
- 5.3. Enter the beam control module . Under “Magnification”, check “Couple Magnifications” to couple the E-beam and I-beam magnifications.



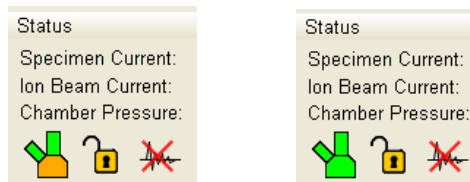
- 5.4. Activate the I-beam quad and then enter the beam control module . Under “Rotation”, set “Scan Rotation” to 180°; this rotates the I-beam image 180° relative to E-beam image and preserves the top-down orientation of the I-beam image when the stage is tilted.




- 5.5. Activate the E-beam quad. Select “Stage” from the pull-down menu and then “Zero Beam Shift” to reset the beam shift. Activate the I-beam quad and repeat to reset the beam shift for the I-beam.




- 5.6. Check “Status” at the bottom of the currently activated control module to determine if suitable vacuum has been reached to proceed. The chamber icon will change from orange to green when ready.

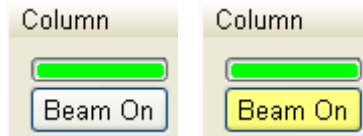


- 5.7. If Pt deposition will be needed during the session, the Pt GIS needs to be warmed. Enter the patterning module . Under “Gas Injection” double click “Cold” to start heating the Pt. When the Pt heating is complete (about 2 min), the heat status will read “Warm”. Warming cannot take place until suitable chamber vacuum has been reached.

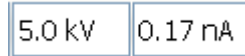


## 6. Turning on the beams




- 6.1. The beams must be turned on individually. Activate the E-beam quad and then enter the beam control module . Under “Column”, select “Beam On” (the color will change from white to yellow). Activate the I-beam quad and repeat to turn on the I-beam.




- 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.



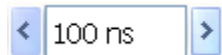
## 7. Live imaging, snapshots, and photos

- 7.1. If a live image is not actively being acquired for a particular imaging quad,  will appear in the upper left corner of the quad. To start live imaging, activate the quad and select  from the toolbar; to stop the live image, select  from the toolbar.



- 7.2. 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 this is satisfactory, or this process can be done automatically by selecting auto contrast/brightness  from the toolbar. *It will usually be necessary to adjust contrast and brightness every time a beam setting (voltage and/or current) is changed. Also note that auto contrast/brightness can only be used during live imaging.*

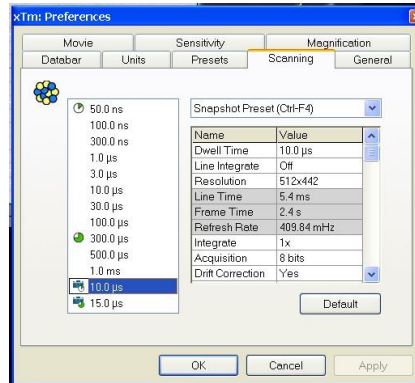


- 7.3. The dwell time used to acquire a live image can be adjusted using

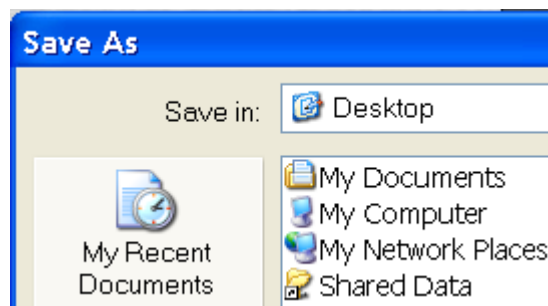


from the toolbar; the response of the image will be faster using low dwell times, but at the expense of a poorer signal to noise ratio.

- 7.4. “Snapshot” and “photo” images of the area being imaged can be acquired by selecting  or  from the toolbar, respectively. Once a snapshot or photo is complete, the live image will stop. To configure the settings for each, select “Scan” from the pull-down menu, then “Preferences”, and then enter the “Scanning” tab. A typical snapshot (photo) setting is a resolution of 512x448 (1024x896) with dwell time of 10 (15)  $\mu\text{s}$ .





- 7.5. 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 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. To move the stage, either double click on a position in the image to move that position to the middle of the image or use the joystick.

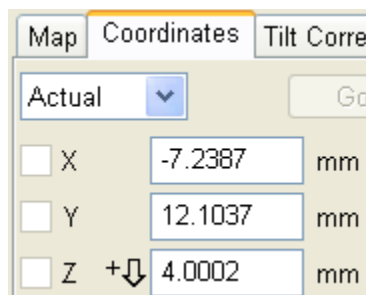


- 8.2. Once the general region of interest has been located, mag in and focus the live E-beam image using the “Focus” knobs on the MUI. When finished, select  from the toolbar to link the Z stage position with the FWD; the icon will change to .



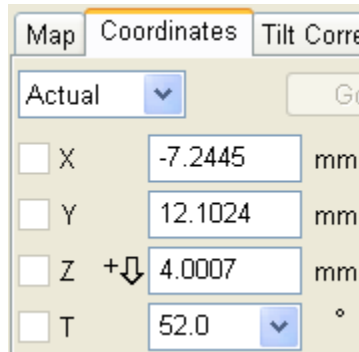
9. Setting eucentric height

- 9.1. Verify that the live E-beam image is well focused and Z is linked to the FWD . Failure to do so before proceeding may lead to a collision between the specimen and internal instrument components.
- 9.2. Enter the navigation module . Under the “Coordinates” tab, enter 4 mm for Z; the stage will move to a FWD of 4 mm and the image should remain focused.

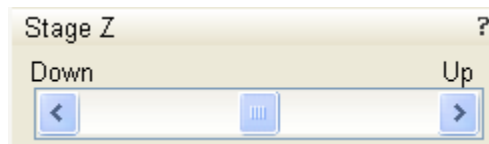





- 9.3. Set the image magnification to 1000× and center a recognizable feature in the image by moving the stage (double clicking on the image or using the joystick).
- 9.4. Go back to the “Coordinates” tab and set T = 52° to tilt the stage.




- 9.5. Go to the “Stage Z” control and use the sliding bar to shift the image up or down to bring the feature of interest back to the center of the image.

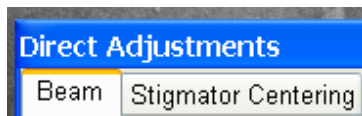


- 9.6. Go back to the “Coordinates” tab and return the stage tilt to T = 0° and center the feature of interest in the image using a mouse double click or the joystick; then refocus the image and select  to update the FWD (it should still be close to 4 mm).
- 9.7. Repeat the previous four steps for an additional iteration; if you did this process correctly, the feature of interest should barely shift (if at all) during the second iteration and the FWD should be close to 4 mm.
- 9.8. Eucentric height is now set for this particular location; however, if any large stage moves are performed (moving between specimens, for example), you will need to reset eucentric height at the new location.

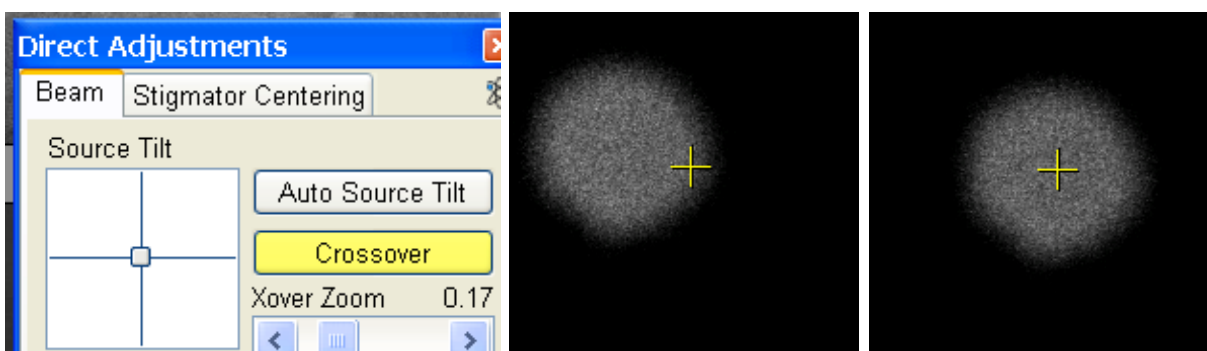
## 10. E-beam alignments

- 10.1. Make sure the E-beam quad is activated and you are acquiring a live image; you must be at eucentric height before attempting any alignments.
- 10.2. Find a feature of interest; on the MUI, use the “Magnification” knob to set the magnification to ~10000×, then use the “Focus” knobs to focus the image as sharply as possible.

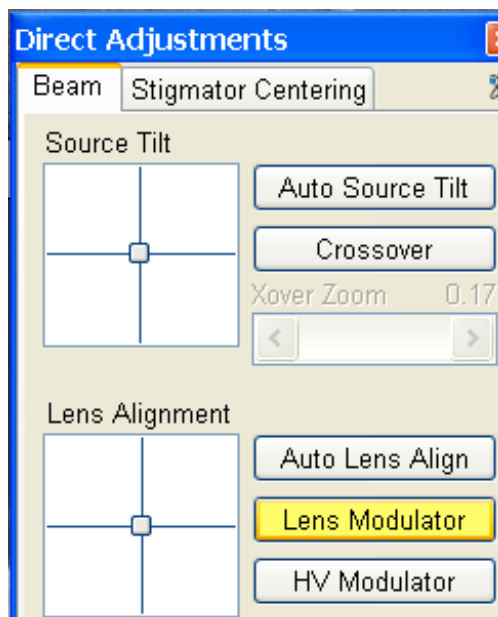
- 10.3. From the toolbar, select “Direct Adjustments”  to open up the “Direct Adjustments” dialogue box; then select the “Beam” tab in this dialogue box.



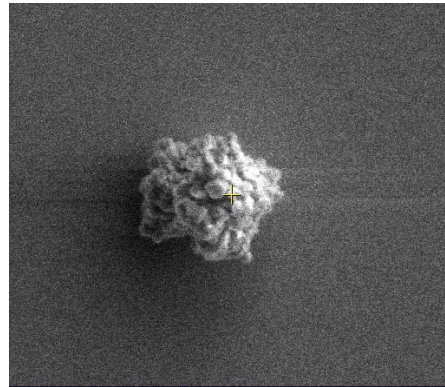
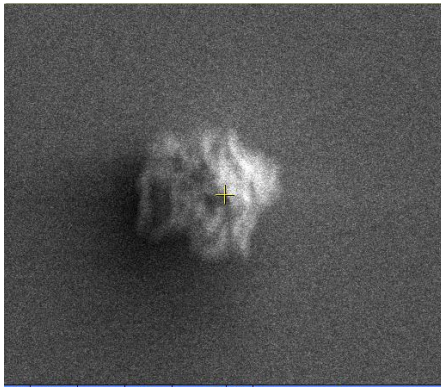
- 10.4. Select “Crossover” to enter crossover mode; use the “Source Tilt” crosshairs to move the bright circle so it is centered on the center cross; select “Crossover” when finished to return to image mode




- 10.5. 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.

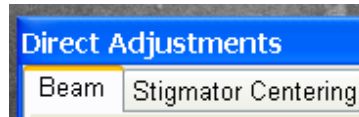


- 10.6. On the MUI, use the “Focus” knobs to focus the image as best as possible; then adjust the “Stigmator” knobs to obtain the sharpest image possible (you may then need to refocus and readjust the stigmators a few times to get the sharpest E-beam image).

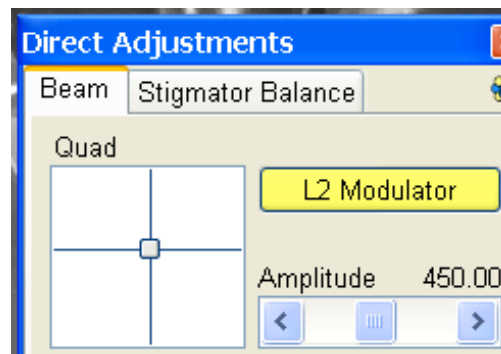


- 10.7. Note that alignment of the E-beam is dependent on the chosen beam voltage and beam current settings; if one or both of these settings is/are changed the alignment will likely need to be adjusted again.
11. I-beam alignments (if needed)
- 11.1. As long as you have properly set eucentric height, the I-beam alignment should not need much (if any) adjustment, particularly at voltages of 16 or 30 kV. That being said, 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).
- 11.2. When executing patterns at I-beam voltages of 8 kV or less at low beam currents, having a well-aligned I-beam becomes more critical for accurate milling. If the low kV image does not appear as sharp as possible (granted, it will not be as sharp as is possible at 16 or 30 kV), you can attempt to manually align the I-beam.

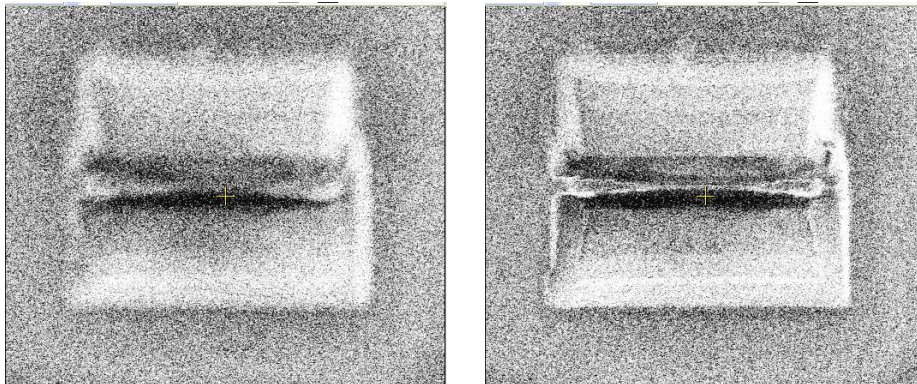
- 11.3. Start acquiring a live I-beam image; on the MUI, use the “Magnification” knob to set the magnification to ~5000×; from the toolbar, select “Direct Adjustments”  to open the “Direct Adjustments” dialogue box; then select the “Beam” tab in this dialogue box.



- 11.4. Select “L2 Modulator”; the image should start going in and out of focus and/or shifting. Use the “Quad” crosshairs to minimize any shifting of the image; select “L2 Modulator” when finished and close the dialogue box.



- 11.5. 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 I-beam image).



- 11.6. Note that alignment of the I-beam is dependent on the chosen beam voltage and beam current settings; if one or both of these settings is/are changed the alignment will likely need to be adjusted again.


## 12. Linking the beams

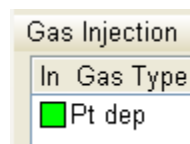
- 12.1. The area of interest must be at eucentric height before attempting to link the beams.
- 12.2. Set the magnification to 1000 $\times$  and center a feature of interest in the E-beam image by moving the stage (again, this should be in the near vicinity of the point where eucentric height was established).
- 12.3. Activate the I-beam quad and set the beam voltage and current to   using the toolbar and start live imaging.
- 12.4. On the MUI, use the “Shift” knobs (do not move the stage) 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.






- 12.5. The link state of the beams is dependent on the stage tilt. In other words, if you change the stage tilt from  $T = 52$  to  $T = 0^\circ$ , the beams will need to be relinked for the new stage tilt (though the adjustment shouldn't be extremely large). Likewise, the link state of the beams may also change if the beam settings are adjusted, particularly as the I-beam voltage is reduced (though again, the adjustment to correct this shouldn't be extremely large).

## 13. E-beam Pt deposition

- 13.1. The region of interest where Pt deposition is to be performed should be at eucentric height; Pt deposition with the E-beam is usually only performed for purposes of protective strap deposition, which should be done with stage tilt set to  $T = 0^\circ$
- 13.2. To check if the system is ready, enter the patterning module . Under “Gas Injection”, the square next to “Pt dep” will be **green** if the system is ready to deposit Pt; if not, it will be **gray**.




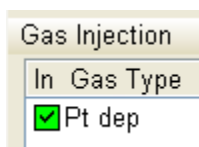
13.3. A   beam should be used for E-beam Pt deposition; keep in mind that if you changed the E-beam settings the E-beam will need to be realigned. Activate the E-beam quad and acquire a snapshot image  of the region.


13.4. Enter the patterning module . Under “Pattern”, select the rectangle pattern . You can then click and drag on the E-beam quad to produce a pattern. In the “Basic” tab next to “Application”, select “Pt dep E-beam” (the pattern should turn **green**). The specific dimensions needed for the pattern can be entered in the “Basic” tab.



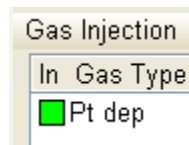
| Basic       |                     | Advanced | Progress | S. T |
|-------------|---------------------|----------|----------|------|
| Name        | Value               |          |          |      |
| Application | Pt dep E-beam       |          |          |      |
| X size      | 10.00 $\mu\text{m}$ |          |          |      |
| Y size      | 1.00 $\mu\text{m}$  |          |          |      |
| Z size      | 2.00 $\mu\text{m}$  |          |          |      |

13.5. Under “Gas Injection”, check the green box under “In” to insert the Pt GIS; a dialogue box will pop up asking you to confirm this. Then take one more snapshot image ; if any shifting of the image occurred, move the pattern as needed.




13.6. Select  from the toolbar to start the pattern.

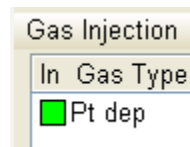
13.7. Once the pattern is complete, retract the Pt GIS by unchecking the green box under “In”.




## 14. I-beam Pt deposition



14.1. The region of interest where 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  $T = 0^\circ$ .

14.2. To check if the system is ready, enter the patterning module . Under “Gas Injection”, the square next to “Pt dep” will be **green** if the system is ready to deposit Pt; if not, it will be **gray**.




14.3. If Pt deposition is intended for producing a protective strap, use the   setting; if Pt deposition is intended for welding two

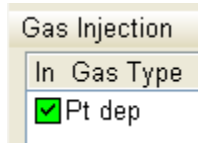
surfaces together, use the   setting instead. Once a beam setting has been selected, activate the I-beam quad and acquire a snapshot image  of the region.


14.4. Enter the patterning module . Under “Pattern”, select the rectangle pattern . You can then click and drag on the I-beam quad to produce a pattern. In the “Basic” tab next to “Application”, select “Pt dep I-beam” (the pattern should turn **green**). If specific dimensions are needed for the pattern, those can also be entered under the “Basic” tab.

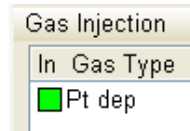


| Basic       |                     | Advanced | Progress | S. t |
|-------------|---------------------|----------|----------|------|
| Name        | Value               |          |          |      |
| Application | Pt dep I-beam       |          |          |      |
| X size      | 10.01 $\mu\text{m}$ |          |          |      |
| Y size      | 1.00 $\mu\text{m}$  |          |          |      |
| Z size      | 1.50 $\mu\text{m}$  |          |          |      |

- 14.5. Under “Gas Injection”, check the green box under “In” to insert the Pt GIS; a dialogue box will pop up asking you to confirm this. Then take one more snapshot image ; if any shifting of the image occurred, move the pattern as needed.







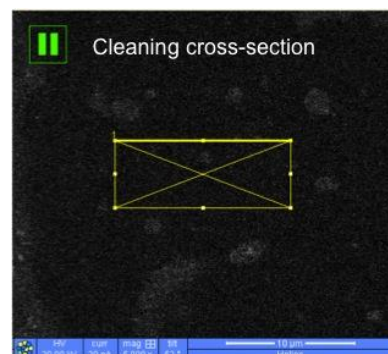
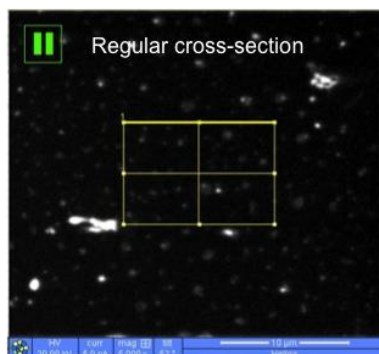
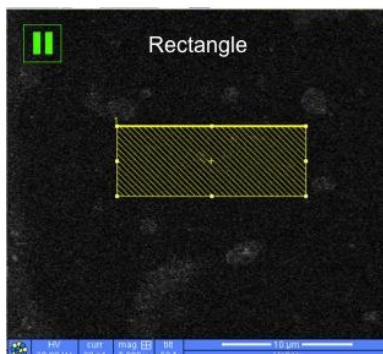
- 14.6. Select  from the toolbar to start the pattern.
- 14.7. Once the pattern is complete, retract the Pt GIS by unchecking the green box under “In”. NOTE: if the Omniprobe is inserted at the same time as the Pt GIS, the Omniprobe should be moved clear (and freed, if necessary) of any nearby surfaces/objects before attempting to retract the Pt GIS. Otherwise, this could result in rupturing of a Pt weld between a specimen and grid or between a specimen and the Omniprobe.



## 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 as expected.

- 15.2. Enter the patterning module . Under “Pattern”, there are several different options available. The most commonly used 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.










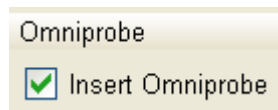



- 15.3. In the “Basic” tab next to “Application”, select “Si” (the pattern should turn yellow). If specific dimensions are needed for the pattern, those can also be entered under the “Basic” tab. If the rotation of the pattern needs to be adjusted (relevant for regular and cleaning cross-sections), this can be done in the “Advanced” tab under “Rotation”.

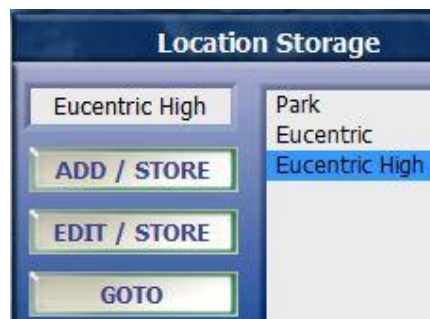
| Basic       |                     | Advanced | Progress |
|-------------|---------------------|----------|----------|
| Name        | Value               |          |          |
| Application | Si                  |          |          |
| X size      | 10.55 $\mu\text{m}$ |          |          |
| Y size      | 2.04 $\mu\text{m}$  |          |          |
| Z size      | 1.00 $\mu\text{m}$  |          |          |

| Basic      |                     | Advanced | Progress |
|------------|---------------------|----------|----------|
| Name       | Value               |          |          |
| Position X | -203.98 nm          |          |          |
| Position Y | -5.22 $\mu\text{m}$ |          |          |
| Rotation   | 0.0 °               |          |          |

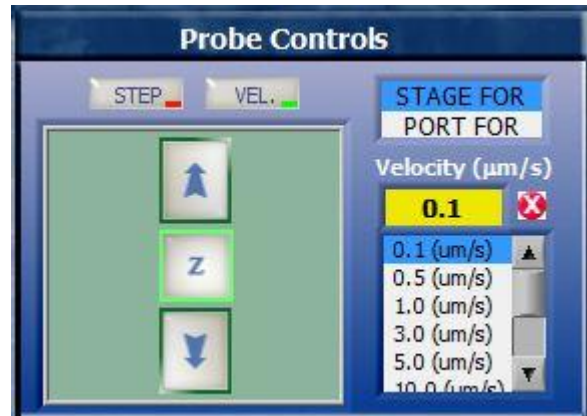
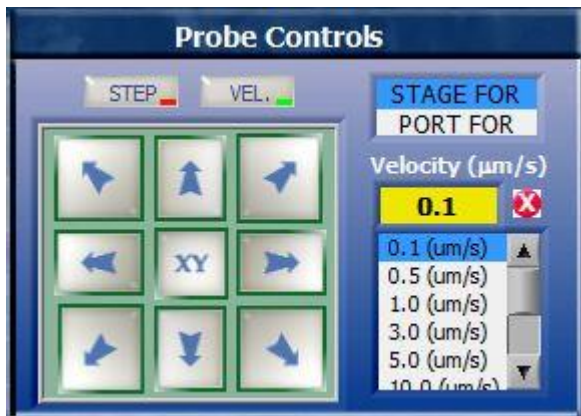
- 15.4. If more than one pattern was drawn, the order of execution can be changed under “Pattern” by selecting  for parallel execution or  for serial execution. If serial execution is selected, the patterns will be executed in the order of being drawn.
- 15.5. Once all patterns have been drawn and positioned as needed, take a snapshot I-beam image ; if any shifting of the image occurred, move the pattern(s) as needed. When ready, select  from the toolbar to start milling; if the pattern needs to be stopped prior to completion, select  from the toolbar.
- 15.6. To erase a pattern from the image, simply select it in the I-beam quad and then select .
16. Using the Omniprobe
- 16.1. The region of interest must be at eucentric height with stage tilt  $T = 0^\circ$  before attempting to insert the Omniprobe; otherwise, the Omniprobe will likely crash when inserted.
- 16.2. Enter the patterning module . Under “Omniprobe”, check “Insert Omniprobe”.



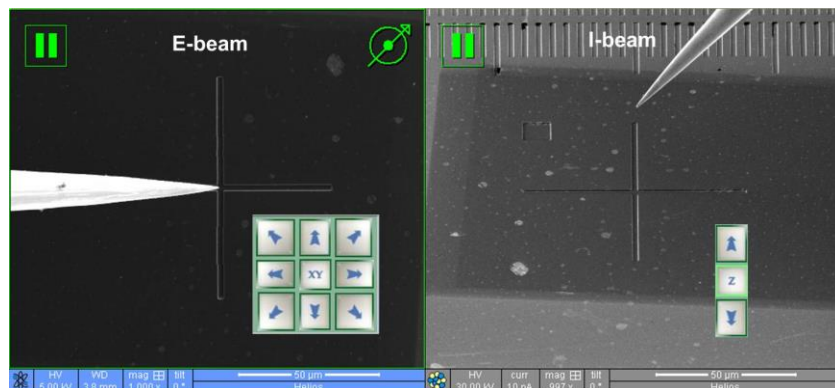
- 16.3. The Omniprobe inserts into the “Park” position, which is well above and away from the region of interest. In Autoprobe (left monitor) under “Location Storage”, select “Eucentric High” and then “GOTO”; this automatically moves the Omniprobe to the middle of the E-beam image and ~300  $\mu\text{m}$  above the specimen surface. The “GOTO” button will change to  during movement and then return back to “GOTO” when finished.




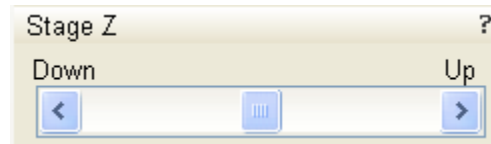
- 16.4. Use “Probe Controls” to bring the Omniprobe down to the specimen surface; clicking on the button in the middle of the arrows will change the control from **XY** to **Z** and vice versa. The Omniprobe speed can be adjusted under “Velocity”; generally speaking, the speed should be reduced as the Omniprobe gets closer to the specimen surface to allow for more precise positioning.



- 16.5. In the E-beam (I-beam) image with scan rotation = 0° (180°), the Omniprobe movement directions in the **XY** (**Z**) panel will correspond to same directions in the image (see below). As a general rule, it is best to use the live E-beam (I-beam) image when adjusting the **XY** (**Z**) Omniprobe position.




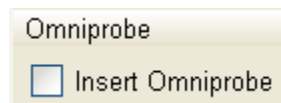
- 16.6. When you are finished using the Omniprobe, enter the navigation module ; use the “Stage Z” control to lower the stage so the Omniprobe is at least several  $\mu\text{m}$  clear of anything (grid, specimen surface, etc.).





- 16.7. In “Autoprobe”, go to “Location Storage”, select “Park”, and then “GOTO” to move the Omniprobe to the park position. Under “Current Positions”, the X, Y, and Z values will be similar to what is shown below once park position has been reached. the “Stage Z” control and use the sliding bar to shift the image up or down to bring the feature of interest back to the center of the image.

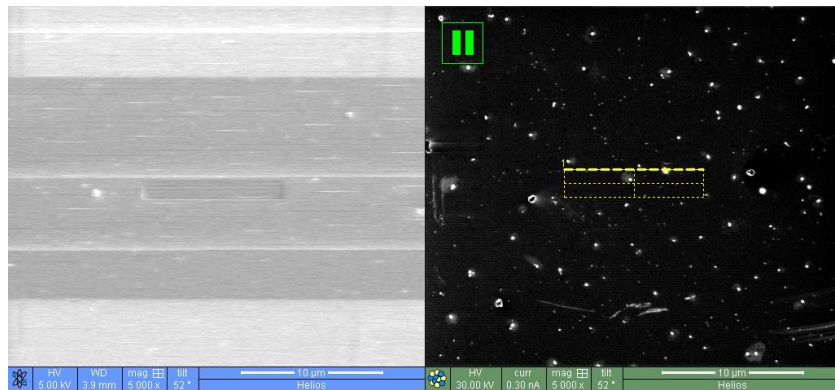




- 16.8. When the Omniprobe is finally in “Park” position, enter the patterning module  and under “Omniprobe”, uncheck “Insert Omniprobe” to retract the Omniprobe.

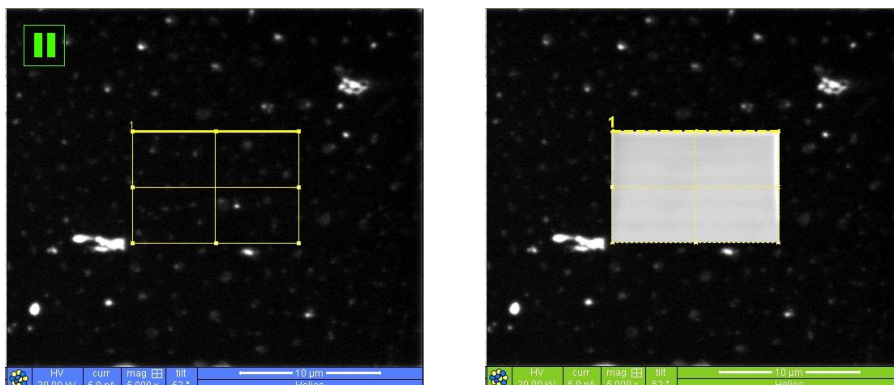


## 17. Live imaging while FIB milling


- 17.1. The E-beam can be used to live image patterning being performed by the I-beam. After starting a pattern , simply activate the E-beam quad and select  from the toolbar to start acquiring a (probably noisy) live E-beam image during patterning. You will need to manually adjust contrast and brightness on the MUI to make the E-beam image more easily viewable. Also keep in mind that the E-beam image will be more easily observable the higher the ratio of E-beam current to I-beam current.




- 17.2. It is also possible to observe a live I-beam signal from the regions that are being patterned (but not the whole image). After starting a pattern , activate the I-beam quad and select ; a live signal from the regions being actively patterned will appear. 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.

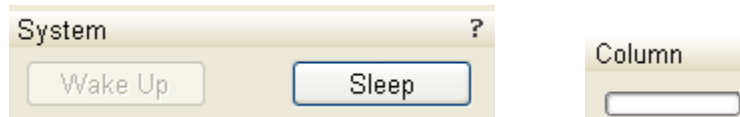



## 18. Finishing the session

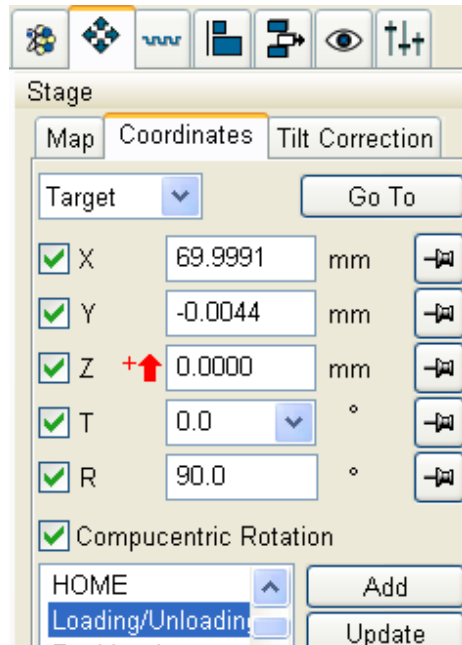
- 18.1. Activate the E-beam quad and enter the beam control module . Under “Column”, select “Beam On” to turn off the beam (the color will change from yellow back to white). Activate the I-beam quad and repeat to turn off the I-beam.




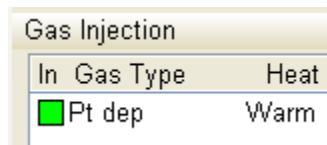
- 18.2. Leave the I-beam quad activated and remain in the beam control module . Under “System”, select “Sleep” to turn off the ion source. Under “Column”, the progress bar should turn white (a few seconds to complete).




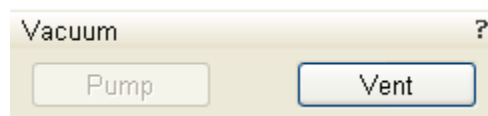
- 18.3. Enter the navigator module . Under “Stage”, select the “Coordinates” tab and double click on the stored “Loading/Unloading” stage position to drive the stage accordingly.



- 18.4. Enter the patterning module . Under “Gas Injection” double click “Warm” to turn off heating of the Pt GIS.



- 18.5. Once stage has finished driving to the loading/unloading position (verify using the live CCD camera image), the instrument can be vented. Enter the beam control module . Under “Vacuum”, select “Vent” to start venting.



- 18.6. Once the chamber is vented, remove the modules from the UMB, close the chamber door, and under “Vacuum”, select “Pump”.

