ZEISS NVision 40 FIB/SEM PROCEDURE

SPECIAL NOTES OR RESTRICTIONS:

- **You MUST be qualified to use the tool by NanoFab staff before using the tool**
- **You MUST have a Smart-SEM user account issued by a NanoFab staff member**
- **Always use the specimen exchange assembly to load and unload samples. If a sample breaks or becomes stuck in the specimen chamber, notify NanoFab staff immediately. DO NOT ATTEMPT UNASSISTED REMOVAL!**
- **If either the red OFF button light or the yellow STANDBY button light on the plinth is illuminated, contact NanoFab staff. DO NOT ATTEMPT TO START THE INSTRUMENT!**
- **ALWAYS TURN ON THE CHAMBER CAMERA BEFORE ATTEMPTING TO RAISE THE STAGE. Failure to observe the interior of the specimen chamber may result in damage to the FIB/SEM and the sample. Never move the stage without viewing the chamber camera image when the sample is in close proximity to the SEM column's objective lens cap.**
- **When screwing the sample exchange rod into the sample holder, DO NOT OVER-TIGHTEN.**

SAFETY PRECAUTIONS:

- **This instrument may generate radiation during operation. DO NOT remove any cover panels, particularly those on the electro-optic column and the specimen chamber.**
- **The maximum acceleration voltage is 30 kV.**
- **Keep the area in front of all ventilation openings clear to prevent fire hazard and overheating of electronics.**
- **Do not bump into the specimen exchange assembly or apply pressure that may bend the specimen exchange rod.**

PLEASE REPORT ANY PROBLEMS TO THE FOLLOWING NANOFAB STAFF

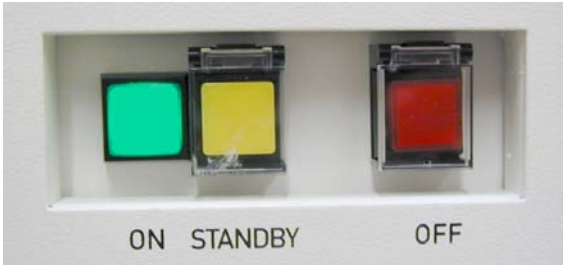
I. Mike Hernandez – EXT 4590 – mikehern@nist.gov
II. Eileen Sparks – EXT 8065 – esparks@nist.gov

I. Sample Prep

Always use powder-free gloves when handling SEM/FIB components!
Secure your sample to stub with carbon tape or adhesive carbon tabs or with bronze clips on the special holders
Use carbon paint to ground sample surface to stub to prevent charging (if sample is non-conductive)
Dry/heat carbon paint to remove solvent before putting into chamber

II. Startup

1. If computer is off, check to see if the STANDBY (yellow) light button on the column unit is on. If so, press the adjacent ON (green) button and wait for the system to re-start



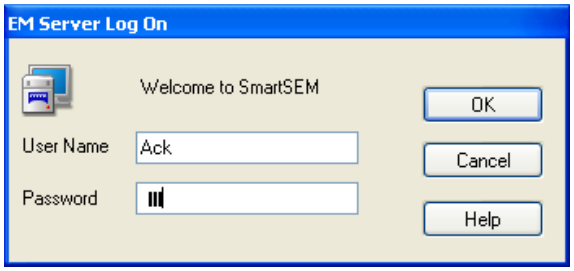
2. To access the instrument PC, log on to the Sun Ray client in the lab using your NIST HSPD-12 badge or an available Sun Ray card and enable the tool. If PC is already running,

   *Note: **DO NOT** forget to disable the tool in Coral when you are finished*

3. Log on to the PC using your NIST network credentials, and launch the EM Server by double-clicking on the SmartSEM icon 

4. Log into SmartSEM software with your NanoFab staff-issued username and password

   (Ignore subsequent y stage error popup)



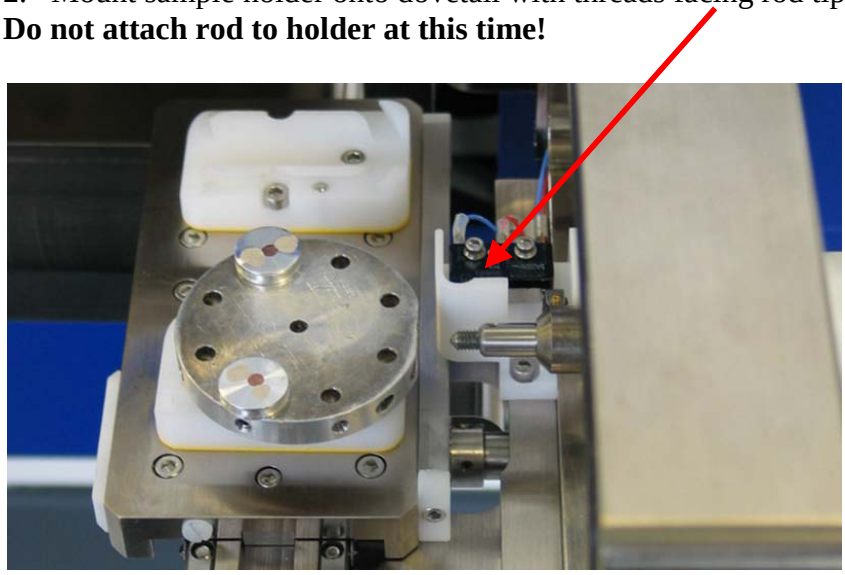
5. Watch for any errors reported in the EM Server window. Most errors are typically cleared during server boot up, and the server display will eventually indicate the error recovery. Should errors persist to the extent that tool operation is not possible, please write the event description in the logbook at the tool, and contact an available NOG staff member

I. Loading the Sample

1. Mount sample stub(s) to holder using small Allen key to fasten

2. Mount sample holder onto dovetail with threads facing rod tip

Do not attach rod to holder at this time!



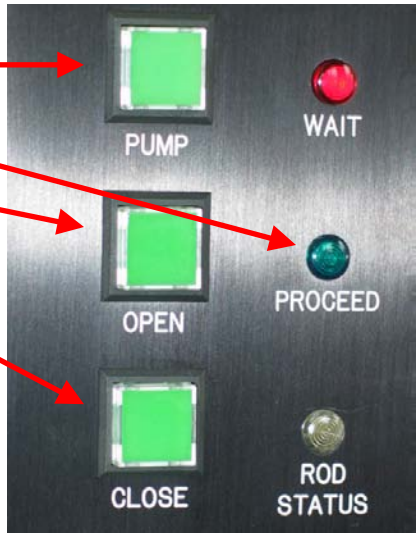
3. Click Exchange button (15) on the Hard Panel (image below). Airlock menu pops up

4. Reply YES to the popup asking if you want to move the stage to the Exchange position, and then click OK when the stage position at exchange confirmation window pops up.

On the airlock control panel:

5. Press Pump and then wait for the green Proceed light to illuminate
   Press the Open button
   Press the Close button

6. Switch to camera view
   by pressing Camera button (17)
   Popup Warning says
   'Soft Vacuum Interlock…' Ignore…

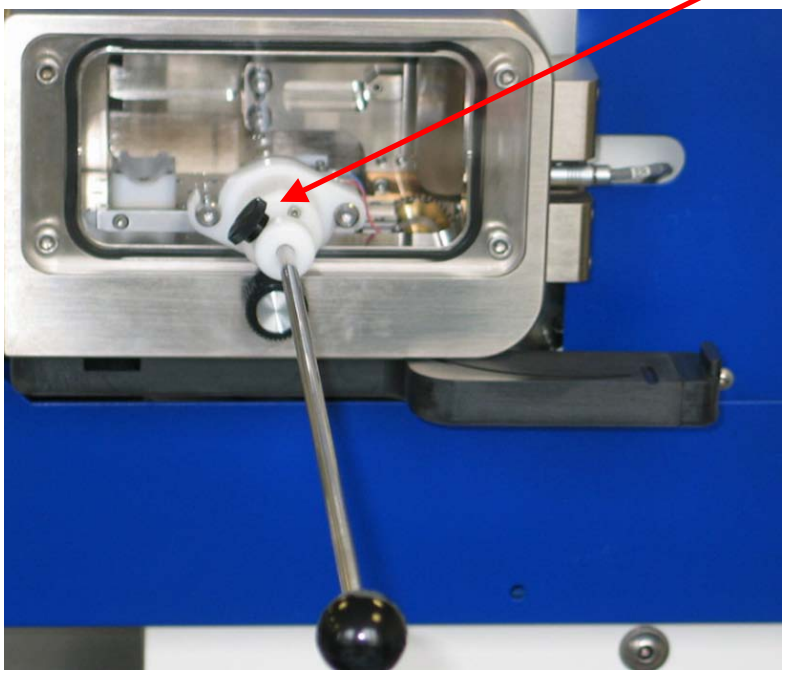


When airlock gate valve opens, the chamber camera view will get brighter

7. Attach rod (screw-on) to sample holder and insert into chamber until it slides onto the dovetail on the stage

8. Carefully unscrew rod and retract it, making sure that the sample holder stays fully attached to the stage dovetail

9. Retract rod all the way and fasten secure it in place with setscrew knob



10. Unclick Open

11. Click Close

12. Wait for Gate to fully close

13. Unclick Pump

14. Click Purge

15. When purged (vacuum seal releases) unclick Purge to stop flow of nitrogen

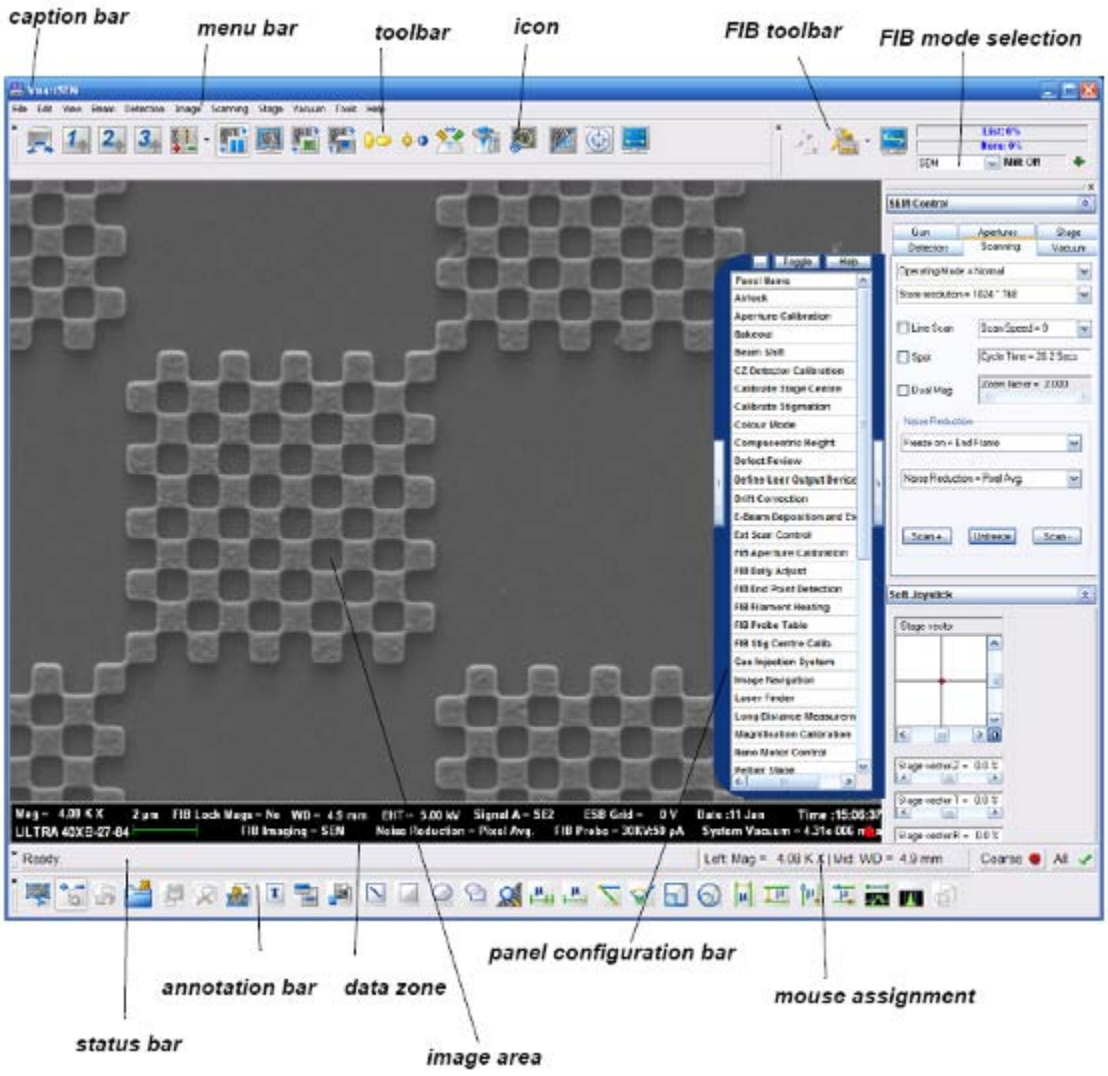
16. Remove exchange rod arm from the airlock (if desired) and replace it with Teflon plug

17. On the airlock menu, click Resume Exchange (or press Resume button [15]) and reply Yes to the popup: “Move stage to the center of Chamber?”

This will move the stage to the center of the main chamber, and open the column valve to allow the electron beam to scan the sample

18. Click OK on the Resume Completed popup

The picture below shows the layout and description of the components of the SmartSEM GUI:

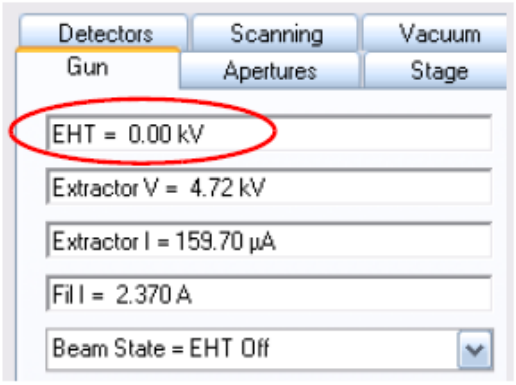


II. Preparing the System

1. Turn EHT on by clicking on the EHT "button" just below the SEM Control panel area

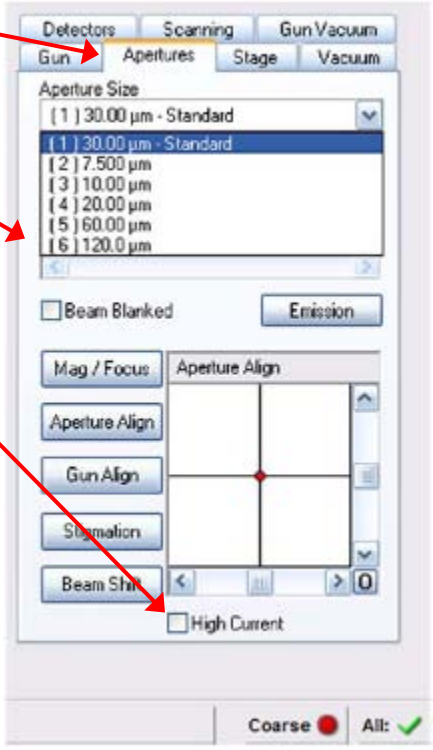
If EHT value is not set, EHT display will read 0, and no SEM image will be visible

2. On the SEM Control panel, click the Gun tab, and double click the EHT value window and enter 5 kV



Note: For most applications, an EHT value of 5 kV works best for SEM imaging during FIB work

3. Click on the Aperture tab in the SEM Control panel, and select the 120µm aperture [6] and check the High Current box

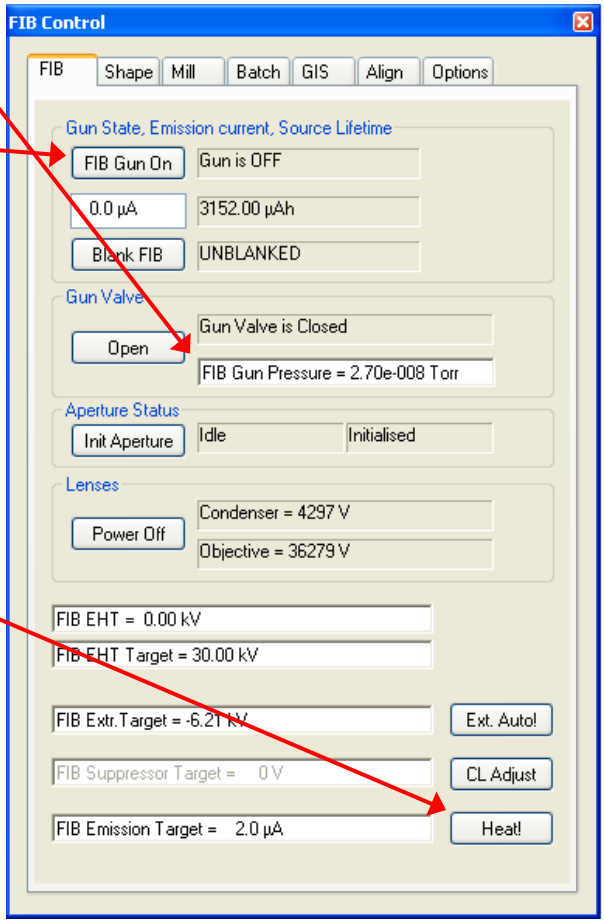


Note: The combination of EHT= 5kV, aperture [6] and High Current provides the best possible alignment between the SEM and FIB images, and also generates enough SEM signal to overcome the electrostatic discharge noise generated by the FIB lenses

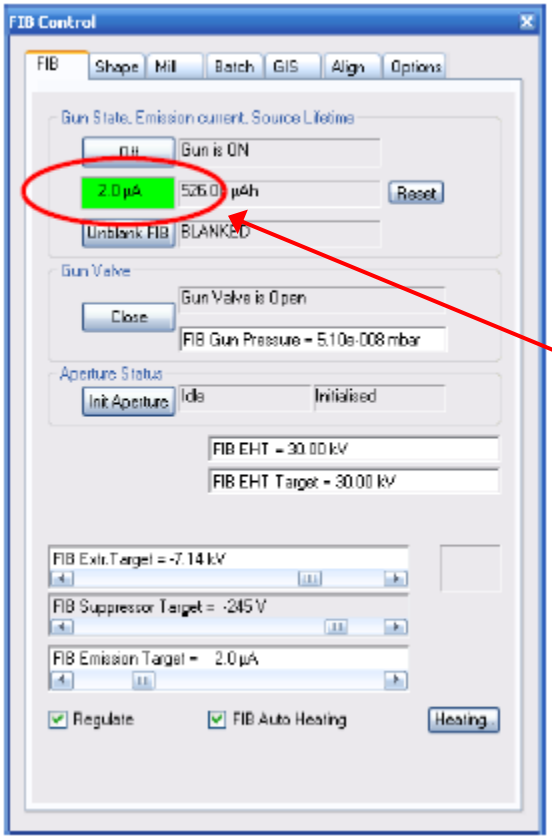
4. Referring to the SmartSEM GUI display, click on the FIB toolbar button to launch the FIB Control panel

5. On the FIB control panel, confirm that the FIB gun pressure is $\leq 7.5 \times 10^{-8}$ Torr

6. If pressure is adequate, click the "FIB Gun On" button to turn on the FIB

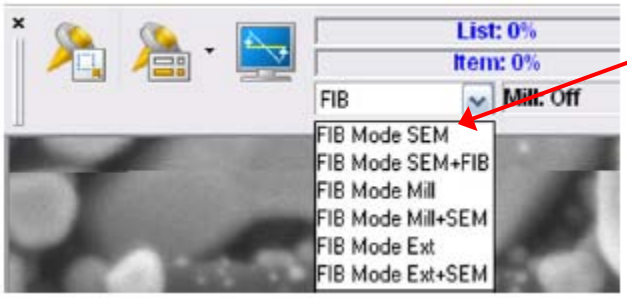


Notes: The FIB EHT will ramp up to 30 kV and the Emission current should stabilize at 2 .0 µA
If successful, the FIB will achieve the following conditions and display a green background in the emission current display window as shown below.
If steady 2 .0 µA emission is ***not*** *achieved, through the above procedure click "FIB Gun Off" to ramp the EHT down, and the click "Heat!" to re-establish LMIS flow*



7. After heating, click "FIB Gun On" again and verify that 2.0µA emission is achieved

Note: As the LMIS ages, it may require more frequent heating cycles before successful stable emission is achieved. If three heating cycles do not restore the FIB, contact an available NOG support staff member and report the issue



8. Referring to the SmartSEM GUI, select FIB mode SEM in the FIB Mode selection window

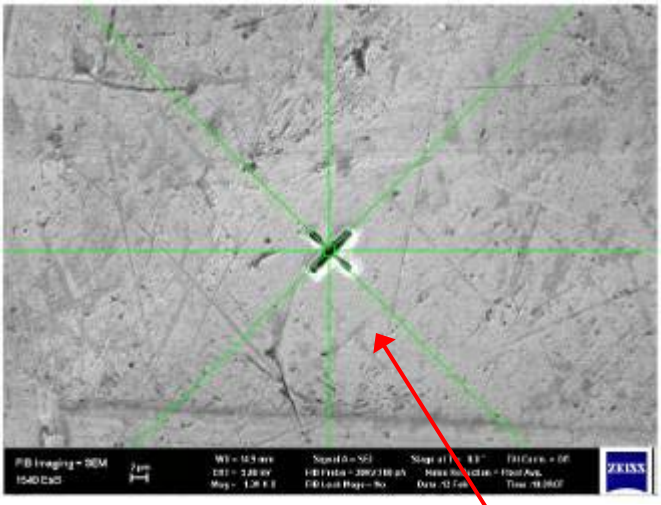
Note: the F8 function key toggles between the SEM and FIB images

9. Click on the Detectors tab of the SEM Control panel, click on the pull-down selector for Signal A, and select SE2

10. Set brightness level to about 50% and change contrast until an SEM signal is visible in the image area

11. Adjust magnification, using knob (1) on the Hard Panel until image is more recognizable. You may need to double-click FIB Lock Mags = Yes to toggle value to No, in order to lower SEM magnification. Follow up by focusing the image, using knob (11) on the Hard Panel to image and orient the sample

12. At this point, note the working distance (WD) display in the image data zone
The WD should be 10mm or further away from the objective lens. If not, lower the stage using the Z-control joystick and refocus to insure the sample moved away from the objective lens (larger WD value)
You can switch to camera view by pressing the Camera button (17) anytime, in order to verify the sample position.

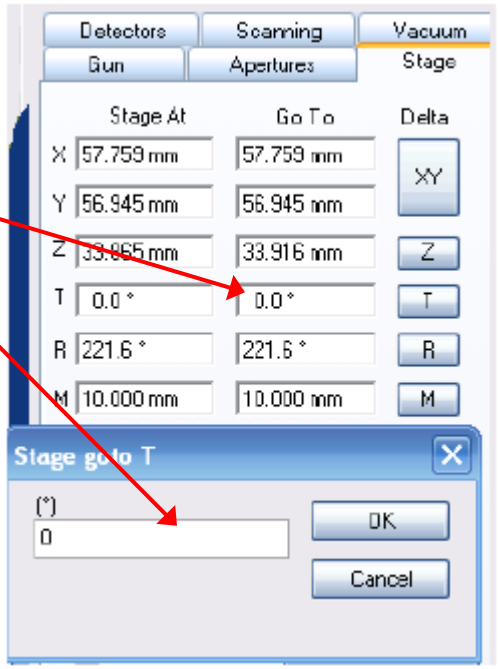


13. Click view on the menu bar, and select “Crosshairs” from the list. This will display a fixed set of graphics used as reference to adjust tilt-eucentricity and coincidence between the SEM and FIB

14. Find a feature on the SEM image that will be easy to re-find if moved away from the current field of view, and center it using the X-Y joystick, or by pressing Ctrl+Tab, and clicking on the feature with the ensuing small cross cursor

15. If the "Protected Z" checkbox is checked on the stage tab, click box to un-check. With feature positioned at the center of the crosshair graphics, tilt the stage to 10° by double-clicking in the Go To display for T.

16. Enter 10 for the Stage go to value in the ensuing popup and click OK



Note: As the stage tilts away from 0°, it is likely that the image will shift in the Y-direction, sometimes by a large distance

17. Using the M up/down arrow buttons on the stage navigation joystick panel, return the feature to the center of the crosshair graphics by pressing and holding the appropriate button

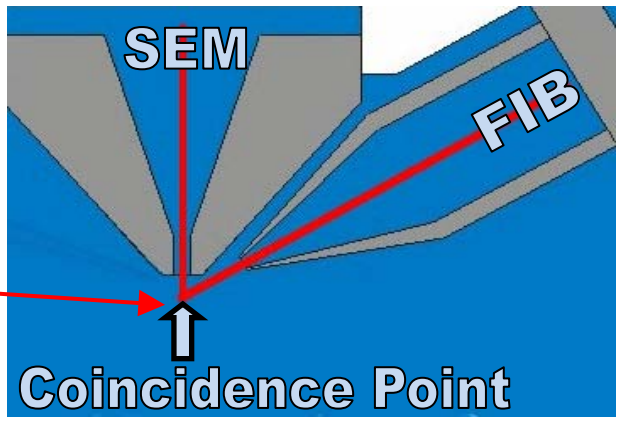
Note: Press the M button w/arrow pointing in the direction that the feature moved (in reference to the original center position)

18. Refocus the SEM image and repeat the above procedure three more times, increasing tilt values to 20°, 40°, and finally 54°

19. To test the accuracy of the current eucentric position setting, tilt the stage back to a lower tilt value and make sure that the feature of interest stays at (or very close to) the center of the stage. If the feature moves substantially away from center, return to 0° tilt and repeat procedure.

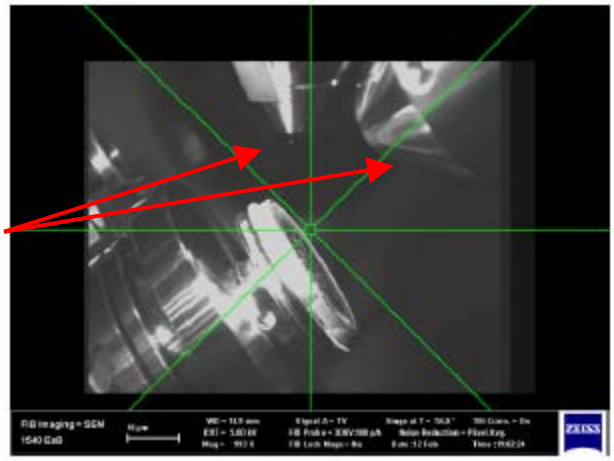
Note: The established eucentric position should be valid for any position along the X-axis of stage travel (post adjustment). Any substantial travel in the Y-direction will modify the eucentric position, and will require re-adjustment

Once the sample is tilted 54° it must be raised in the chamber in order to achieve coincidence between the SEM and the FIB optics. The coincident point is defined as the position in the chamber where the SEM and FIB are both focused on, and scanning the same area of the specimen.



For the NVision40 the coincident WD is just slightly less than 5mm from the SEM objective lens's bottom

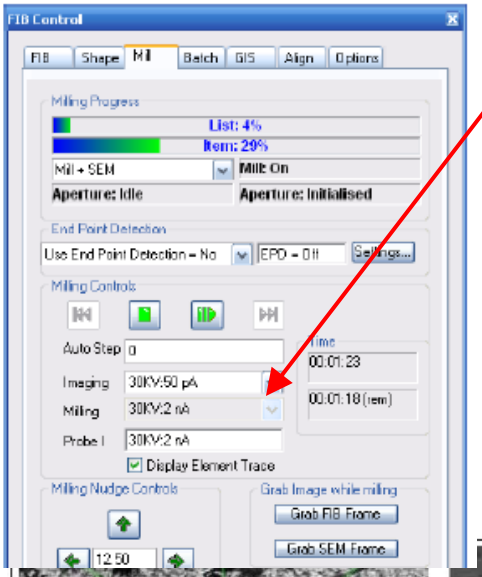
20. In order to arrive at coincidence, double-click the WD display in the data zone and enter 5 for the WD value in the popup. The SEM image will go out of focus.



21. Press the Camera button (17) on the Hard Panel to switch image to chamber view, and confirm that the stage is not in very close proximity to the SEM and FIB lenses

22. Switch back to the SEM image by pressing the Camera button again, and begin moving the stage closer (up) to the lenses. The stage Z-travel motor speed is linked to magnification. The lower the magnification, the faster the motion. As the image begins to come back into focus, increase magnification and continue moving the stage up until the sample is in focus at 5 mm WD

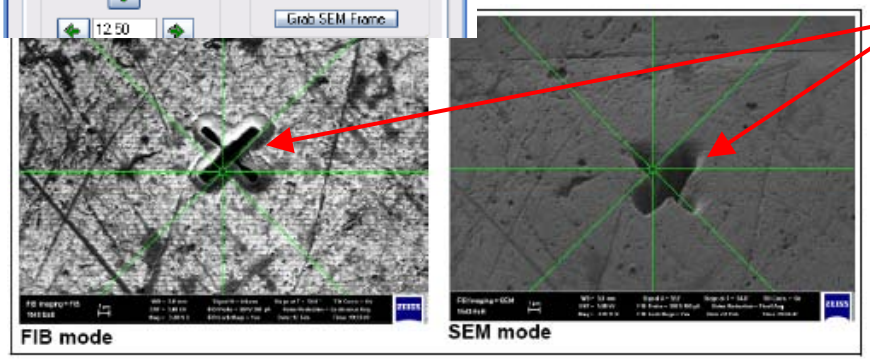
23. On the data zone, double-click FIB Lock Mags = No to toggle value to Yes (if necessary)



24. In order to reduce the amount of material removed during FIB imaging, select a low FIB imaging probe current from the Mill tab of the FIB Control panel. Use a probe current ≤ 80 pA

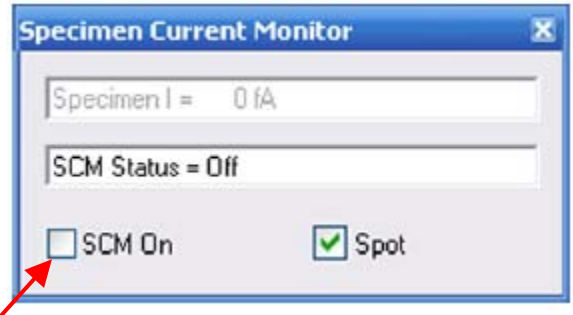
25. Toggle between SEM and FIB images using F8. The position of a reference feature of interest may be shifted in the Y-direction by a substantial amount between the two images. In SEM mode, move the reference feature back to the center of the crosshairs (if necessary)

26. Toggle to the FIB image, and using the Z control joystick move the reference feature to the center as needed



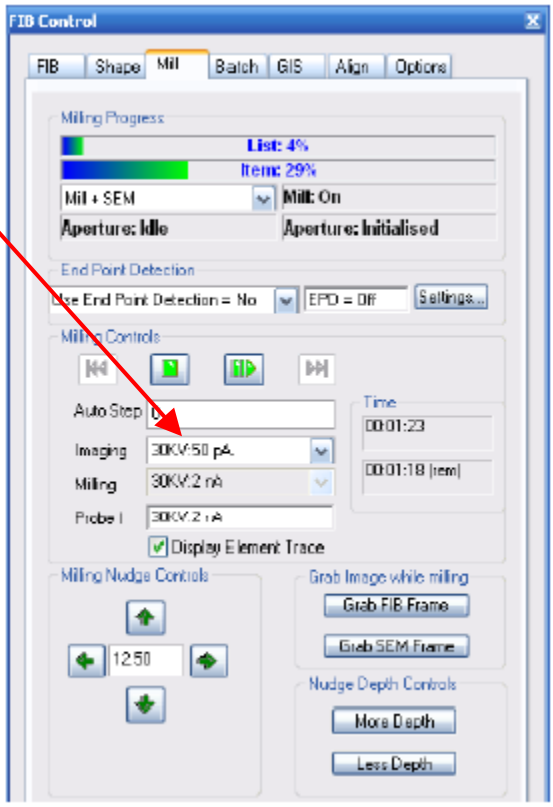
Notes: Any shift in the X-direction cannot be corrected via mechanical stage motion. Use the Shift X and Shift Y knobs (7 & 8) on the Hard Panel to adjust the SEM image's X-direction position relative to the FIB image.

***Be very careful moving the stage in the Z-direction when at 5 mm WD**. Moving the stage up in close proximity to the lens may bring the sample in contact with the lens. If this occurs, you will hear an audible touch alarm and see a warning popup. If this happens, open the panel configuration bar, and click on item Specimen Current Monitor. When the popup appears click on the SCM On check box to silence the alarm*



At this point turn on the Camera and carefully lower the stage away (Z joystick downward motion) from the lens until the highest point on the tilted sample is clearly below the level of SEM lens. Next click stage on the menu bar, and select Stage Initialize. Reply yes to the popup and wait for the procedure to execute and finish. If stage initialization fails, discontinue using the tool, and contact a support staff person.

27. The FIB is capable of milling or depositing material on a specimen. The FIB Control panel allows the user to select separate FIB probe currents for imaging and milling



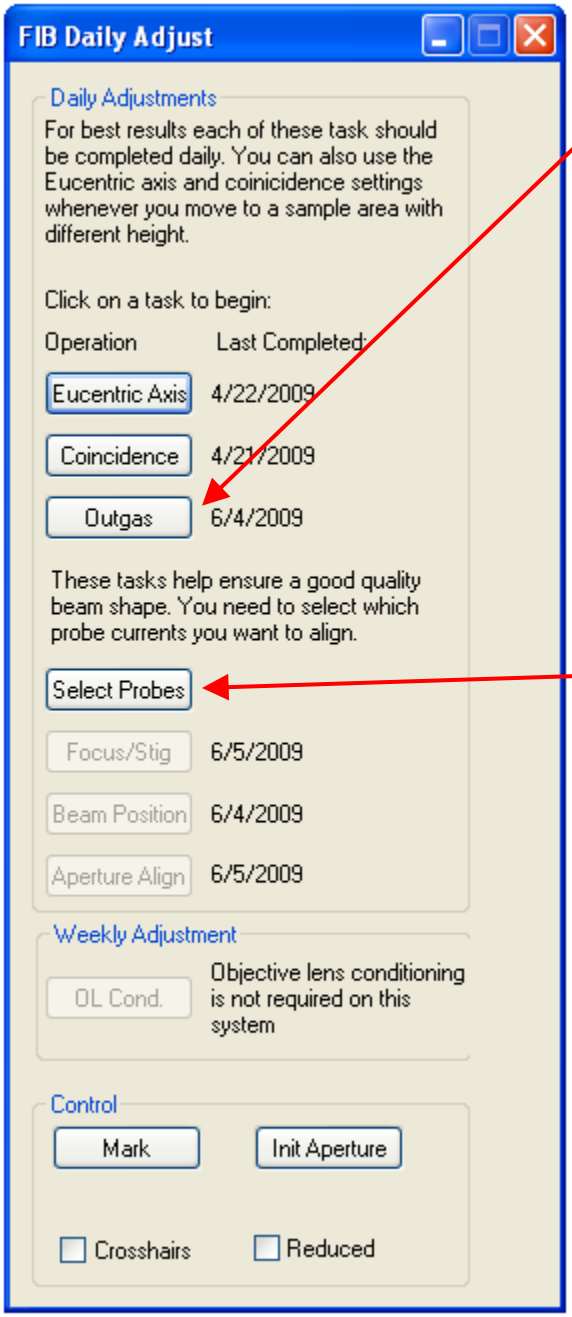
The user must define and plan the procedure in order to use the FIB efficiently. In order to deposit material, the gas precursors must first be out-gassed. The out-gassing procedure is part of the FIB Daily Adjust procedure

Key parameters for successful material deposition are:

- Setting the milling object's resolution pixel fill factor to about 70% (under 100% is critical). Pixel fill factor can be set by changing the milling pixel resolution and/or changing the FIB image magnification
- Using a probe current of (or just under) 5 pA/μm^2 of deposition area

- Set probe dwell time to 0.4 µS for Pt and 0.8 µS for C deposition
- Make sure the GIS needle is safely inserted before deposition process starts

28. To execute the aforementioned daily adjustment procedure, click the tab to open the panel configuration bar, and click on the FIB Daily Adjust item. This will launch the following panel

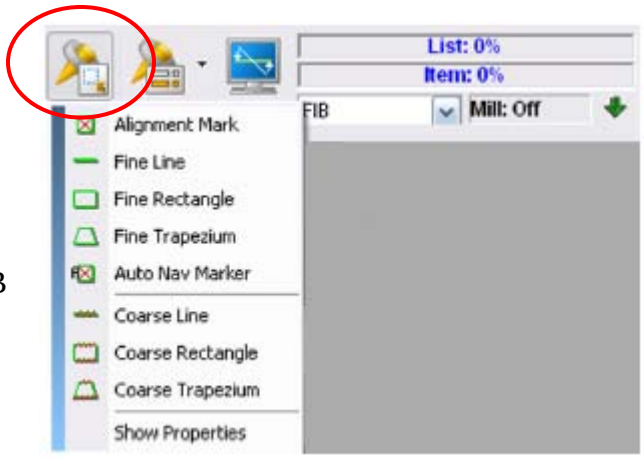


As previously mentioned, perform the precursor outgas procedure by clicking on the "Outgas" button. This starts a macro, which allows you to select a precursor to outgas and execute the procedure. If a precursor has NOT been recently out-gassed, an asterisk will appear next to the precursor's name. In this case, carry out the procedure. Otherwise, skip Outgas.

The condition of each FIB probe current determines not only efficiency, but also the quality of the milling process. In order to insure that the probes to be used are in the best working condition, click Select Probes and populate the popup list with the desired probes. Subsequently, click on the now-active Focus/Stig, Beam Position, and Aperture Align buttons, and carry out the procedure for each macro for all selected probes

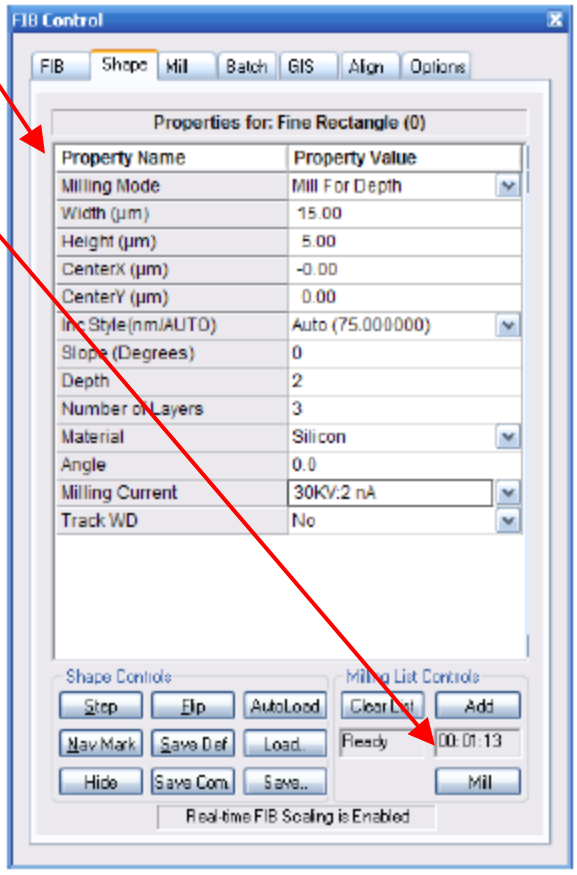
The dates to the right of each macro button indicate the last time the procedure was carried out. If the date is within one week, it is likely that the probes do not require adjustment. However, there is no guarantee that the previous routine included the currently desired probes

29. Once setup is successfully completed, choose a milling object by clicking on the drop-down button on the left side of the FIB toolbar

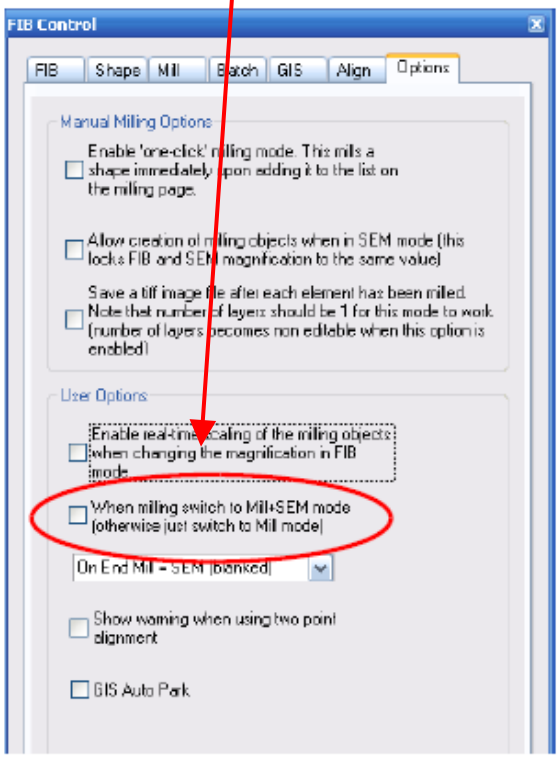


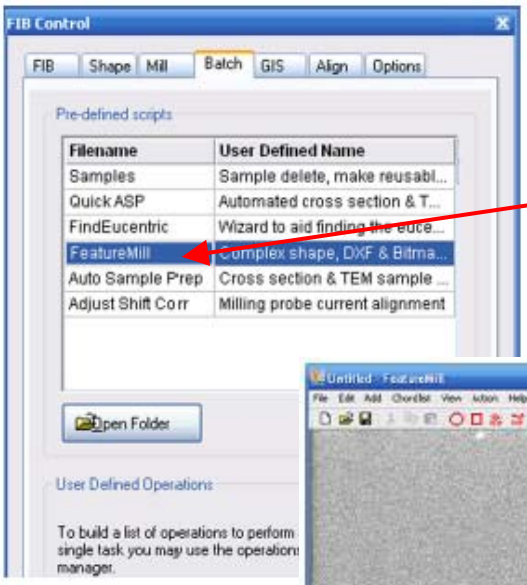
30. Select the desired shape type, and click and drag the cursor on the FIB or SEM image to draw and size the shape on the image

31. Upon drawing the milling object, the Shape tab on the FIB Control panel will become active. Use this GUI to input the desired values for the defined milling object. Up to 16 separate objects can be added to a milling list.
Clicking Mill will begin milling each individual object in the order they were added to the list



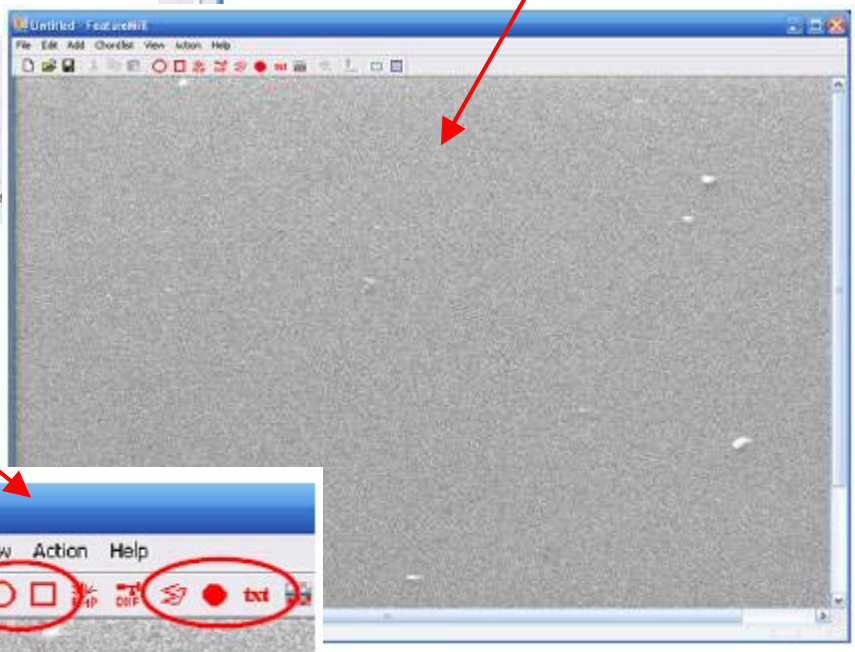
32. The milling process can be imaged and monitored by checking the box for Mill+SEM mode in the Options tab of the FIB Control panel





33. More complex milling objects can be defined by clicking on the Batch tab of the FIB Control panel

34. After clicking the Batch tab, click on FeatureMill. This will open an untitled Feature mill image window and the current active FIB image will be captured and displayed in this window



You can draw any of the text and/or geometric shapes available

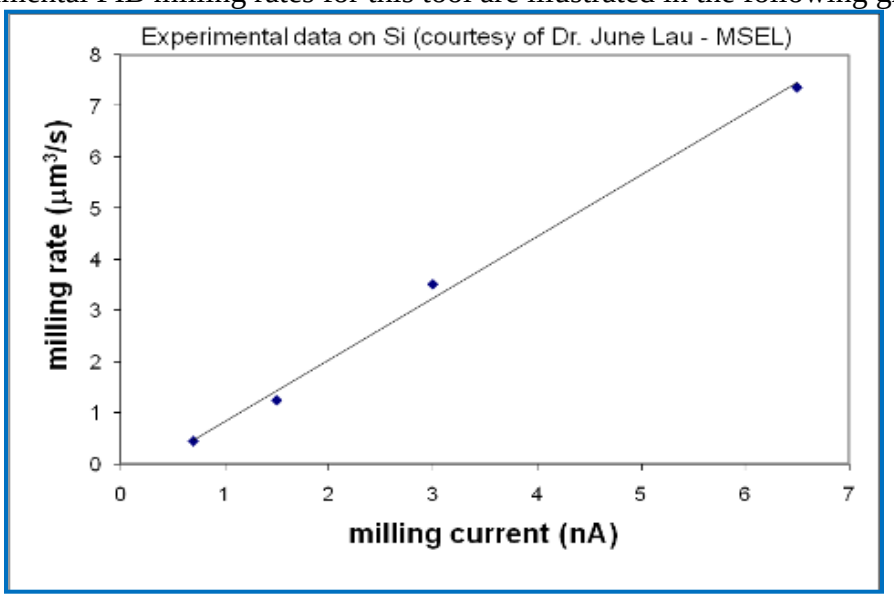
You may also select a BMP or DXF CAD file to import

The scale/size of the object can only be defined by overlaying a grid pattern of a specified pixel density and calculating the size using the known image pixel size.

Note: The system is capable of advanced lithography through a Raith ELPHY Quantum system. However, the separate lithography system's operation is quite complex and instruction for that system is available separately, and beyond the scope of this document

III. Other/General

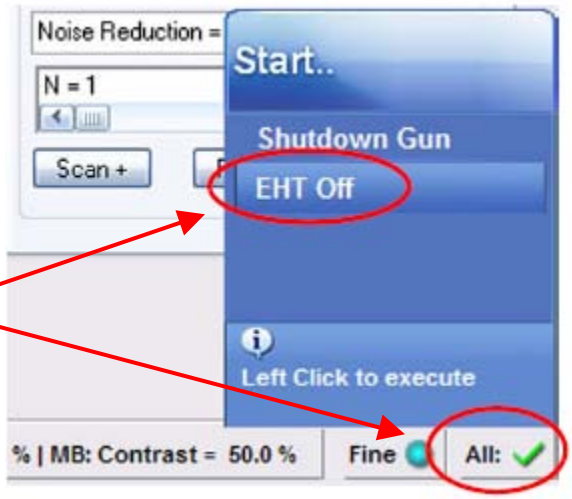
- Experimental FIB milling rates for this tool are illustrated in the following graph



- When SEM imaging is important, make sure that the SEM aperture is well aligned, and that focus and astigmatism correction are properly adjusted. To align the SEM aperture, press the Wobble button (13) on the Hard Panel, and eliminate image movement by adjusting alignment knobs X (4) and (5). Press Wobble once more to turn off
- Occasionally you will hear a popping noise (FIB gun valve closing) if the FIB scanning is idle for more than 5 minutes
- Tilt correction on the SEM image (to correct Y-direction image foreshortening due to tilt) can be implemented by double-clicking the item on the data zone and inputting the appropriate value
    - Enter 54 for vertical measurements on the sample's tilted surface
    - Enter 36 for vertical measurements on a milled cross section surface (90° to the 54° tilted surface
    - Horizontal (X-direction) measurements are not affected by tilt

IV. Unloading Sample

1. **Turn Camera on, and using the Z stage control, lower the stage so that the sample is clearly below the SEM and FIB lenses.** Click the "All" button on the lower-right section of the SEM Control panel, and left click on the EHT off popup to shut off the electron (SEM) beam



2. ON the FIB Control panel, go to the FIB tab and click the “Off” button to shut off the FIB

3. If the airlock exchange rod was removed during the specimen loading procedure, remove the Teflon plug form the airlock and replace it with the rod

4. Click Exchange button (15) on the Hard Panel, and reply yes to the popup

5. Press CLOSE button (to release) press PUMP button on the airlock control panel, and wait for the proceed light to illuminate

6. Press OPEN and wait for the airlock gate valve to open (the chamber camera view will get brighter when the valve is fully open)

7. Insert exchange rod into chamber and attach it (screw-on) securely to the sample holder. If the rod does not slide easily, loosen the setscrew knob to allow the rod to slide

8. Retract the holder onto the airlock dovetail stage, completely unscrew the rod from the holder, and fully retract the rod until the ROD STATUS light on the airlock panel stops flashing

9. Press OPEN Press (to release) and press CLOSE on the airlock panel

10. Wait for airlock gate valve to fully close and Press PUMP (to de-activate pump)

11. Press PURGE and open the airlock when pressure reaches atmosphere. Press PURGE at this time to turn off N2 gas flow

12. Remove sample form airlock, close airlock, and press PUMP. On the airlock menu, click Resume Exchange (or press Resume button [15]) and reply No to the popup: “Move Stage to Center of Chamber?”

13. Click File on the menu bar, and select Log Off

14. Confirm your choice in the ensuing the popup

15. Click the red X on the EM Server window to shut down the server

16. Click Start and log off the PC *Note: If you do not log off, Windows will lock the PC after a few minutes of inactivity and the next user will not be able to use the tool without*

17. Disable the tool in Coral from the Sun Ray client terminal. Alternatively, launch Coral on the instrument PC and disable the tool there before logging off