FEI Helios NanoLab 660 Dualbeam FIB-SEM Operation



Instruments Layout

Computers Layout



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Introduction

1. Check the status of FIB-SEM: the green light in front of the Helios is on (Figure 1).



2. On the SEM server computer, check whether xT microscope server and xT User Interface (xT UI) are running: if not double click the "FEI System Control" on the desktop to

Fi	gure 2		
🔒 xT microscope Server		2	
Server state RUNNING UI State RUNNING	Co Ma In	nsole devices otion NODE1 ition NODE2 laging	
Microscope Start St	top (Standby	
Show UI Sto	p UI	Advanced >>	

Figure 3

launch xT microscope server (Figure 2). Double click the blue area to show full view (Figure 3): Make sure that indicators for **Console devices**, **Motion NODE1**, **Motion NODE2** and **Imaging** are green in the right panel. Click "Start UI/Stop UI" button to start/stop UI. During starting UI a log-in window will pop-up for you to log in. <u>Username and password are both "supervisor".</u> Full view of xT UI (also called xT microscope control) is shown in Figure 4.

- 3. Check the vacuum: all three parts of microscope icon in the lower right corner of the main xT UI are green (Figure 4).
- 4. Check the CCD quadrant (must be live): the stage is 0° tilt; there is enough distance clearance between stage (no tilt) and the pole piece (stage must be below the 4mm line); retractable parts (GIS needle and Easylift probe) should be retracted (Figure 4). Note: GIS control is in Patterning [™] page and Easylift control is in Navigation [№] page.



5. Check both Ebeam and Ibeam quadrants, both Ebeam and Ibeam should be off: Beam On button in grey indicates that beam is off; Beam On button in yellow indicates that beam is on (Figure 5).



Note: Orbital icon indicates Ebeam and the ball

cluster icon 🕸 indicates Ibeam; The green bar above **Bean On** button indicates that beam is ready and white bar indicates that beam is not ready.

6. Check and make sure there are no unresolved errors in xTm Application Status message window in the CCD quadrant (Figure 6). The message window can be open through Tools > Application Status if it does not pop-up automatically. Note: Click Hide in Application Status window to bring CCD live view to front.

Note: DO NOT click Clear in Application Status window.

Messages Su	tem status	
Time	Description	Source
Friday		
17:38:42	The FIB source is not operational.	FIB Source
15:16:30	Simultaneous scanning in more quads is not allowed for requested scanning parameters (dwell time, bit depth, resolution). Try to pause one quad or adjust scanning parameters, i.e. increase dwell time, decrease bit depth or decrease resolution.	Imaging
14:24:15	The SEM aperture home procedure has been finished successfully.	SEM Apertures
14:24:08	The FIB aperture home procedure has been finished successfully.	FIB Apertures
14:23:49	The GIS Pt dep temperature has reached the set point 43.44 °C	GIS Patterning
14:23:47	The FIB source is working properly.	FIB Source
14:23:46	The FIB aperture cannot be selected: aperture home procedure is running,	FIB Apertures
14:23:39	The FEG source is operational.	FEG Source
Pop-up on M	lessage Severty:	[#]4.[0
O None	Error Warning Al	Clear Hide



SEM Operation:

Loading/Unloading Samples

Click Vent button in Beam Control page of UI to vent the chamber (Figure 7). Click Yes on the xTm: Vacuum Message dialog.



- 2. Venting the chamber for \sim 3 minutes, the chamber door is ready for open.
- 3. Open the chamber door slowly, and locate the sample stage. Attach samples tightly to proper pin stabs and fasten pin stabs to the stage (<u>Do NOT overtight the screw</u>). Adjust the height of sample stage to make sure the sample surface is close to Max marker of the gauge (Figure 8). Center and tight the cross piece of the stage with the two cylindrical pieces.

Note: Watch the live CCD quadrant during opening and closing the chamber door and be prepare to stop for unexpected incidents.



Figure 8

Close chamber door fully and click **Pump** button in Beam Control page of UI. Wait until all three parts of microscope icon in the lower right corner of the main UI turn green (Figure 4). It is recommended to wait until the chamber pressure is below <u>4e-5</u> mBar (Figure 4).

Ebeam Imaging

 Go to top menu Stage > Take Nav-Cam Photo to take a photo of sample layout. Nav-Cam photo allows user to quickly move to area of interest by double-click. The green cross centers at area of interest that is currently viewed by Ebeam. Note: Always watch live CCD quadrant when moving sample stage.

2. Click Ebeam quadrant, select proper HV (start with 2kv or 5kv), current (start with 25 pA), dwell time (300 ns for searching), resolution (start with 768 x 512) and filter mode (average 4 frames). Click Beam On
Beam On
button to turn it in yellow. Click Pause button
in Toolbar (Hotkey: F6) to start scanning and watch live image in Ebeam quadrant. Reduce magnification if needed and locate a visible feature on the sample surface. Adjust Brightness/Contrast knobs using SEM control panel or use auto contrast and brightness button

Note: Hotkey F5 switches selected quadrant to full screen view mode; Hotkey F7 activates reduced area view mode.

- 3. <u>Try the best to focus on sample surface feature until a mag > 2000 x</u>, then click Link Z to Working Distance button in Toolbar. Go to Navigation page , change Z coordinate of the stage to 6 mm, focus on the sample again, and click Link Z to Working Distance button again, then change Z to 4 mm, and focus on the sample. Note: it is a good practice to link Z to working distance if Z height and/or focus have changed.
- 4. Check stigmatism by changing focus back and forth to see if the image shows any stretch. If so, adjust focus until no stretch showing, and adjust Stigmator X and Y knobs on SEM control panel to minimize the astigmatism (showing sharpest image).
- 5. Take SEM images by using **Photo** button in **Image Acquisition**. You can change the setting in top menu **Scan** > **Preferences** > **Photo Preset**.
- 6. High resolution imaging in mode 2 (Immersion): switch SEM mode to Mode 2 (Figure 9). Repeat step 4 and step 5 to achieve sharpest image. Note: Response of image movement is much more sensitive in mode 2 than that in mode 1, so stay focused and be patient. Note: TLD detector for secondary electron (SE) will be automatically chosen to replace ETD detector in mode 2.
- When finishing, Click Beam On button to turn it to grey. Tilt the stage back to 0° if it's not at 0°. Click Vent and wait for ~3 minutes to unload the samples, after that, close the chamber door and click Pump.

Note: the following step 8, 9, and 10 are optional.

- 8. Direct alignment:
 - a. Click the icon 🗄 and direct alignment will show up as a pop-up window (Figure 10 left or right panel).



- b. Choose Beam page and click Crossover button, the Ebeam quadrant will show the beam crossover image. Use Xover Zoom sliding bar to zoom in/out crossover if needed. Move mouse cursor to Source Tilt square, click either lines or center icon and hold mouse left button to center the crossover in Ebeam quadrant. After you are done click the Crossover button again to return to normal Ebeam image. Adjust focus and stigmatism to get sharp view.
- c. Next click HV Modulator in Beam page, Move mouse cursor to Lens Alignment square, click either lines or center icon and hold mouse left button to minimize image movement in Ebeam quadrant. Use Amplitude sliding bar to change the amplitude of image movement.
- d. Now choose Stigmator Centering page and activate Modulator X, move mouse cursor over Stigmator Center X square, click either lines or center icon and hold mouse left button to minimize image movement in Ebeam quadrant, repeat this step for Modulator Y. Use Amplitude sliding bar to change the amplitude of image movement. Close direct alignment window after you are done.

Note: it is recommended to do direct alignment again if any one of Z height, HV, beam current has changed.

- 9. Multiple detectors set up: It is possible to use up to three detectors simultaneously in Ebeam imaging (quadrant 1-3, always keep quadrant 4 for chamber CCD). Most frequently used combination is ETD for SE, Mirror Detector (MD) for Backscattered Electron (BSE), In-column Detector (ICD) for BSE. If sample is not tilted, a two detector combination is often used: ETD for SE and retractable CBS detector for BSE. Shift + left click II to control scanning in all detectors simultaneously.
- 10. SmartScan can be setup by right click any one of 1 through 6 button in Scanning Preset (Figure 4). Right click any number (**1 through 6**) and choose **edit** to bring up smartscan setting window (Figure 11). Choose shorter dwell time (< 500 ns) and integrate 2ⁿ frame (normally 32 to 256) to generate the final image. This is an efficient strategy to avoid charge build-up for non-conductive samples. Another one is to choose low HV(1 kV, 500V, 350V) and beam current (25 pA, 7.7 pA, 1.1pA). Press **Ctrl** button and click on selected Smartscan button to acquire an image.

	Basic Setup		
	Name	TV	
	Resolution	768x512	
	Dwell Time	300.0 ns	
	Bit Depth	16 bit	
Ξ	Filter Setup		
	Scan Interlace	1	
	Line Integration	1	
	Average	4	
	Frame Time	138.2 ms	
Ξ	Image Acquisition		
	Integrate	32	
	Drift Correction	Yes	
	Continuous Scan	No	
	Action	None	
	Acquisition Time	17.7 s	
Ξ	Shared Settings		
	Mains Lock	No	
	Start on Click	Yes	
N S	lame et this Scanning Preset	: name.	
	Import		286
	Export		Apply
	Default	ОК	Cancel

Figure 11

EDS operation:

O AZtec - test

- 1. Log in the EDS computer, password "supervisor", start Aztec program.
- 2. Click on the New Project button and select the folder to save the project.

0		X
Welcome t	to AZtec	IN STEV MENTS
Projects	Demo Data	Help
Wei		
SChoqu	ette	
Sam		
Lograssi	0	
Rao		
E New 1	Project 🕞 Ope	n Project
		Close
Show this screen at start-up		

3. Aztec defaults to a sputter coating of Gold for your sample. Make sure you uncheck this if you did not coat your sample.

File View Techniques Tools Help	• •
Mg O	Sends Help Q P
Specimens in 'test'	 → Qorman 1 → Qorman 2 → Size 2 → Size 3 → Size 4 → Size 5
Specimen Notes for 'Specimen 1'	of Site 1 *
Cick here to begin entering notes about your specimen.	Input Court Rate (pp) Output Court Rate (pp) Output Court Rate (pp) Dead Time Tree Recommended W0 50 mm Process Time 6 High Voltage 50 VV
Specimen Loading information:	F 🛓 🖌 (26239)

- 4. Make sure CCD quadrant is paused ^{III}; it provides a high noise to the EDS detector.
- Click on icon "EDS Detector Control" in bottom right of software and click on
 Operate in "Thermal", wait until the detector fully cooled, which will show "Cold" and the

blue light on the detector stops flashing. Then insert the detector by clicking In in "Position".

Thermal	Operating Status: Standby
Position	Operate Standby
Detector Cont	rol: port 207
5 Detector Cont Thermal	rrol: port 207

- 6. In SEM, use a high enough kV to excite the x-ray peaks you are interested in (typically 2x peak energy). For example, if you want to see a peak that appears at 6 keV, use 10 kV or 15 kV accelerating voltage (not 5 kV).
- Ideally, you want to increase your current so that the dead time is in between 20% to 60% when SEM is live (such as 1.6 nA or 3.2 nA). DO NOT let the dead time exceed 60%.
- 8. Adjust brightness, contrast, focus, stigmator alignment in SEM.
- In AZtec, choose what type of EDS scan you want to perform from the list in the top left ("Map" is recommended for general use):
 - a. Analyzer This will provide you with a general spectrum encompassing all the sample in the SEM



window (collect with the SEM live, not paused). This is ideal if you have a uniform sample and you are trying to identify what elements are present.

- b. Point & ID This technique allows you to grab an image of you sample and choose specific locations to acquire EDS spectra from. This is useful for verifying if certain locations contain elements that other locations do not
- c. Linescan This technique will provide you with a linear elemental distribution across your sample
- d. Map This technique will provide you with a 2-D elemental distribution of your sample
- e. Optimize This is used for calibrating the detector and you should not use it.



10. In Scan Image Page

, check the settings as following and press

Start to capture an SEM image in the area of interest.

Scan Image Settings		★ 🗵
Image Capture		
Image Scan Size:	1024	•
Dwell Time (µs):	10	•
Input Signal:	🗹 SE	
	BSE	
Number of Frames:		1
Frame Time (secs):	7.209	

11. In Acquire Map Data Page

check the settings menu:

- a. Process time dictates data binning such that small process times lead to faster scans but broader peaks. So choose a higher process time (5 or 6) if you are looking for elements which have peaks close together.
- b. For maps and linescans, choose "Until stopped" or "Fixed duration" with adequate frame count for your needs.



- 12. Draw a rectangle, elliptical or freehand region from the left side menu to start acquiring EDS data in selected area. Note: pressing **Start** will collect EDS data on the whole SEM image region.
- 13. In **Construct Maps** Page , you can save the spectrum data by right click > Export > EMSA. It will save the x-ray energy (keV) and cps/eV in a text file.
- 14. Click **Report Results** expansion icon on the top right and choose "**Site Report**" to export a Word file containing all the images and spectrums of selected site.
- 15. Repeat step 10 to 14 for any other area of interest on the samples. You can add new sites in the Data Tree on the right or add new Specimen.
- 16. When finished, click to retract the detector (**Out** in "Position") and set it to **Standby** in "Thermal".



Focused Ion Beam (FIB) Application (imaging, milling, and patterning)

FIB alignments and imaging:

- If the Gallium ion source is not heated (The status bar above **Beam On** button is empty), click **Wake Up** button (Figure 4) in **Beam Control** page to start heating Gallium ion source until the status bar above **Beam On** button turns green.
- 2. Perform beam alignment in Ebeam (mode 1:field-free) and move stage Z to 4 mm..
- Zero beam shift for both beams in Beam Control page: right click Beam Shift square in Beam tab and select Zero (Figure 12).





4. Set Eucentric height: Center a recognizable feature by double click it in Ebeam quadrant at magnification in between 2000x and 3000x, fine tune focus and stigmatism to achieve sharpest image, link Z to working distance. Make sure Ebeam quadrant is live and tilt the stage to 2° while watch the centered feature in Ebeam quadrant. If the feature move away from the center of screen, adjust Z height up or down in navigation page [●] to bring the feature back to the center of the screen. Then Tilt stage to higher angle (e.g. 5°) and bring the feature back to center if it moves. Repeat this step each time with a higher tilt angle (2°, 5°, 15°, 30°, 40°, 52°) and bring the feature back to the center of stage to 52°. At 52° tilt angle fine tune focus and stigmatism and link Z to working distance

Note: redo eucentric height adjustment when moving to other sample locations.

- Choose HV(30 kV) and beam current (24 pA) for Ibeam imaging. Click Beam On button to switch on Ibeam (button turn yellow). Quickly adjust Brightness/contrast, focus, and stigmatism using reduced area (HotKey: F7).
- 6. Make sure that both beams center on the same area of sample (Coincident Point): center a recognizable feature in Ebeam, adjust beam shift of Ibeam in Beam control page (Figure 4) to center the same feature in Ibeam imaging (HotKey: Shift + mouse left hold and drag).

Note: Try checking **Dynamic focus** button in **Navigation**>**Stage**>**Tilt Correction** to see if the whole imaging area in Ebeam have the same focus.

In order to protect samples from being destroyed by Ibeam, choose smaller beam current (24 pA), shorter dwell time (100-300 us), and low resolution (768 x 512) for Ibeam imaging. Smaller beam current higher the resolution.

Note: choose Ibeam current >100 pA for milling and patterning.

Ebeam Pt deposition:

The beam (depending on whether you are doing Ebeam or Ibeam deposition) must be perpendicular to the sample surface during Pt deposition. Ibeam can easily destroy the top layer of samples of interests, it is necessary to add a thin layer of Pt layer by Ebeam before Ibeam deposition:

- 1. Ebeam deposition is to add a thin layer of Pt (0.1 um) to protect sample surface in Ibeam deposition process.
- 2. Return stage tilt to 0° .
- Draw rectangular pattern in Ebeam quadrant and choose Pt e-dep surface in Pattern>Basic>Application (Figure 13), pattern should be in green color, make sure the pattern is slightly wider than area of interest. Choose depth to be 0.1 um. Make sure the GIS is in Warm status (bottom panel in Figure 13).
- 4. Choose HV (2kv) and current (1.6-2.4 nA). Adjust beam brightness/contrast, focus, and stigmatism after changing HV and/or current.
- 5. Insert GIS needle by checking green box in **Pattern>Gas Injection** and watch live CCD quadrant.

Note: After inserting GIS needle, take a quick Ebeam snapshot (double click F6) to make sure there is no stage drift.

- 6. Click **Play** button **b** in toolbar to start deposition.
- After the Ebeam deposition is done, <u>retract GIS needle</u>. Take a quick Ebeam snapshot to make sure the Ebeam deposition works.

Ibeam Pt deposition:

Tilt the stage to 52° and take a snapshot in Ibeam quadrant using low beam current (24 pA). Find the Pt layer and adjust Ibeam brightness/contrast, focus, and stigmatism using reduced area if needed.

- 2. Draw rectangular pattern in Ibeam quadrant and choose **Pt Dep** in **Pattern>Basic>Application** (Figure 13), pattern should be in green color, make sure the pattern is slightly smaller than the existing Pt layer. Choose the depth to be 0.5-2 um.
- 3. Calculate the area of rectangular pattern in um^2 and choose proper Ibeam current to guarantee that the averaged beam density is 6 pA/um². In addition, the total deposition time of 5-15 mins indicates the correct choice of beam current. After choosing Ibeam current do a quick Ibeam focus and stigmatism.

Note: DO NOT use Ibeam current that is larger than the calculated max current.

4. Insert GIS needle, take a quick Ibeam snapshot to make sure there is no image shift. Click **Play** button 上 in toolbar to start deposition. Activate iSPY to monitor the process of deposition if you prefer.



Note: iSPY is optional, you can turn on iSPY for the first

several minutes and turn off iSPY after you have confirmed that the deposition is really happening.

5. After the Ibeam deposition is done, retract GIS needle and take a quick Ebeam snapshot to make sure the Ibeam deposition works.

Ibeam milling/patterning:

If the top layer of sample need to be protected from Ibeam damage during milling/patterning, do the Ebeam and Ibeam deposition first before milling/patterning.

1. Select pattern shape in the pull-down menu of **Pattern** tab (left panel of Figure 14) and draw it in the Ibeam quadrant (pattern should be in yellow color); choose proper parameters in **Basic** and **Advanced tab** in **Pattern** tab (right panel of Figure 14): Application (Si New for rectangle pattern, Si-multipass New for regular cross-section and **Si-ccs New** for cleaning cross-section), X Y Z size, ScanDirection, etc.

Choose HV (30 kV), choose proper beam current (> 0.43 nA), quickly check focus and stigmatism, click Play button in toolbar to start patterning.

Note: the Ibeam current choice depends on various patterning application. The following shows the most frequently used regular cross-section milling.

- (Optional) Monitor patterning process: check On button in Pattern>End Point Monitor>iSPY, choose proper time interval. iSPY takes Ebeam snapshot for every time interval defined.
- 4. Regular cross-section milling is frequently used. Always draw regular cross section pattern wider (x dimension) than area of interest. Cleaning cross section is useful tool to smooth surface of cross section and is good for milling ball-like structures. Current selection for milling:

Rough milling for large volume: 21, 47, 65 nA Regular milling for moderate volume: 0.79, 2.5, 9.3 nA Cleaning/polishing: 0.23, 0.43, 0.79 nA

Note: if choosing >21 nA to do rough milling, it is good practice to leave a safe distance (1-2 um) from the Pt deposition layer and then choose 0.23, 0.43, 0.79, 2.5 nA for fine cleaning.

Pattern	?	Pattern	
	Hide Hide		Hide Hide
Q.	4 • • •	•	4 + >
1 -Rectangle 1	~	1 -Regular Cross Se	ction 1 🗸 🗸
Basic Advanced P	rogress Sel. Mill EasyLift	Basic Advanced F	Progress Sel. Mill EasyLift
Name	Value	Name	Value
Application	SiNew	Application	Si-multipass New
Xsize	40.00 μm	Xsize	40.00 μm
Ysize	20.00 μm	Ysize	20.00 μm
Z size	15.00 μm	Z size	15.00 μm
ScanDirection	Bottom To Top	ScanDirection	Bottom To Top
DwellTime	1.000 µs	DwellTime	1.000 μs
Beam	lon	Beam	lon
Time	0:15:32	Time	0:04:12
Beam Current	48.87 nA	Beam Current	48.87 nA

Figure 14