

FEI 430 Nova NanoSEM Operating Manual

The FEI 430 NanoSEM is a field emission scanning electron microscope for high resolution imaging down to several nm features using its range of backscatter and through-lens detectors. This microscope can image samples ranging from small pieces to 4-inch wafers. It is also equipped with a platinum deposition tool and a NPGS electron beam lithography system.



Tool Capabilities

- 5nm Resolution
- Backscatter and Secondary Electron imaging with Immersion Lens Mode
- Working Distance: 5mm
- Beam Voltages: to 30 KeV
- Stage Bias up to 5KeV
- NPGS Electron Beam Lithography
- Platinum Deposition

Training to Become a Qualified User

Tool training for basic SEM operation requires a 2-hour, hands on session and a follow up a qualification test. The lab member must demonstrate safe and knowledgeable operation of the microscope to gain unsupervised access to the SEM. The first 10 hours of SEM usage must be during daytime, 8am to 5pm.

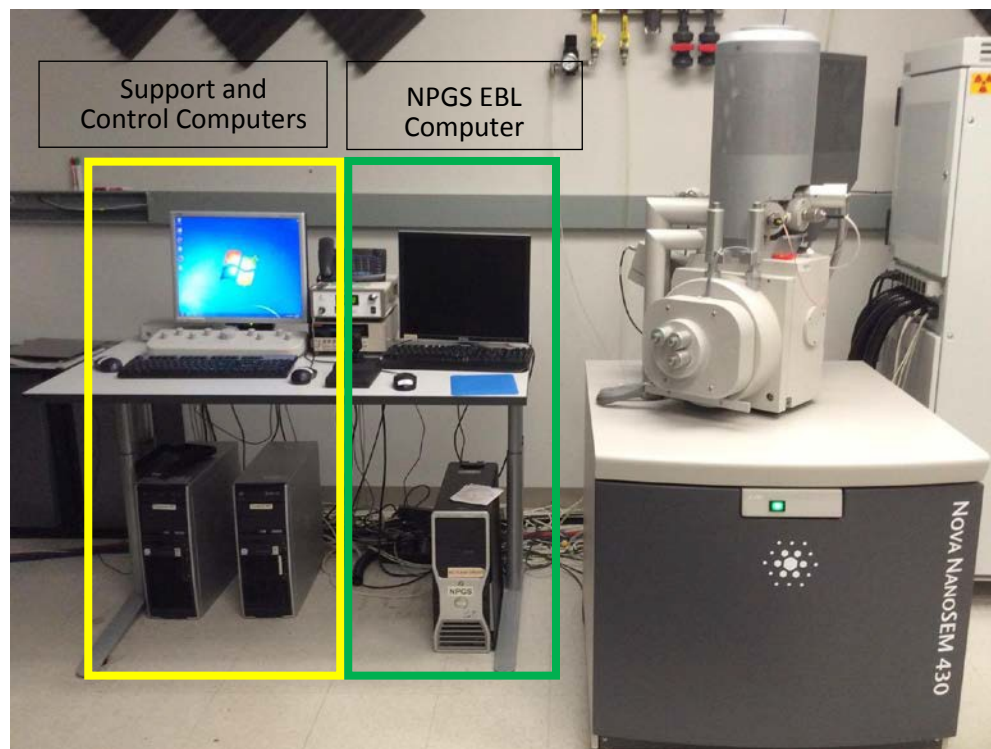
Training for platinum deposition and NPGS electron beam lithography are provided in separate training sessions.

Precautions and safety

1. Clean gloves are required at all times when touching:
 1. Your samples
 2. Sample Holders
 3. Any part of the SEM chamber
2. Always watch the CCD camera to ensure the stage and samples will not contact any chamber component when you are loading and unloading samples.
3. Never make large stage movements or tilt angles. Always watch the CCD camera during any stage movement to ensure the stage will not contact any chamber component.

System Hardware Overview

The NanoSEM 430 is controlled through three separate computer systems. The Support computer is used for file sharing, storing images and connecting to Badger. The Control computer runs the NanoSEM software. The Support and Control computers share a single monitor. A switch box is used to change between the monitor between the two systems. The NPGS computer is dedicated to the Electron Beam Lithography option.



Control Screen Layout

Imaging is paused

Selected imaging window. Shown: ETD

Blue bar: Active window

Screen not paused

CCD window shows inside of chamber

Stage lowered

Software Control pages (see list below)

Microscope mode: High Vacuum

Microscope status

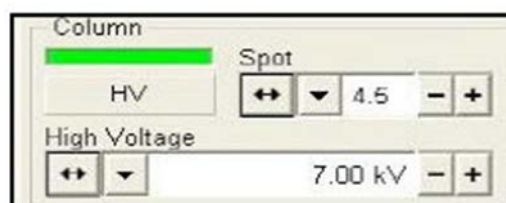
Page List	Main Features
Beam Control	Pump/Vent chamber, Beam On/Off, Column: Spot Size and High Voltage, Alignments, Microscope Status
Detectors	Detector Settings: Detector type and mode, Grid Voltage
Beam Deceleration Control	Apply beam deceleration voltage or stage bias
Navigation	Move stage position, apply tilt, and tracking Z-height
Patterning: Platinum Deposition	Platinum patterning: heater, patterning progress and controls
Processing/Measurement	Measurement tools, post processing: Digital brightness, contrast, and gamma
Alignments	Stigmator centering alignment

Tool Start-Up

1. Check the SEM logbook for any issues with the tools. If there is an issue, verify the problem has been added to Badger and the CNM2 staff has been notified.
2. Review Badger for Shutdown, Problems and Comments. Verify there are no lab member reservations for the duration of your session.
3. Enable FEI_NanoSEM_ncnc on Badger.
4. Make sure the green light on the front of the column cabinet is on.
 1. If the System Control Panel Power Button is yellow or off then do not use the SEM. Report the problem on Badger and contact the CNM2 staff.
5. Using the monitor selector switch, change to the SEM Control screen.
6. If the Microscope XT program is not open, find the XT server icon and double click to start the program.
 1. As soon as the program is open, a log-on window will pop-up.
 2. The application status window may pop-up. If it does, then alert staff with any issues that is displayed
7. Check the status window at the lower right corner of the screen.
 1. **Chamber Pressure:** $< 1 \times 10^{-5}$ mbarr
 2. **Gun Pressure:** $< 1 \times 10^{-7}$ mbarr
 3. **Emission Current:** Enter the emission current in the logbook.
 4. The Column icon is green for both the Gun and Chamber indicating both are under vacuum.



8. Verify the **Column HV** bar is bright green, indicating the electron emission source is ready.
 1. If this bar is white contact the CNM2 staff and put the system Shutdown in Badger.



9. Make sure the system is in **HIGH VACUUM MODE**.

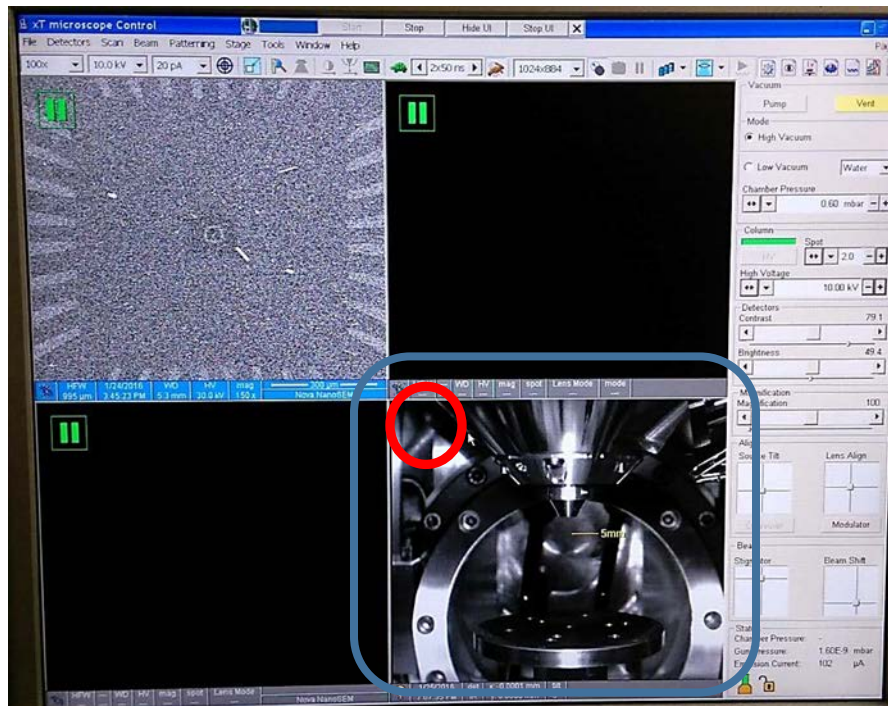
1. This is the standard configuration of the SEM and should always be in High Vacuum.

Loading a Specimen

If all parameters from the Tool Start-Up are correct then it is OK to load a specimen.

Please consult with staff before attempting to image cross-sections or tilted samples. These samples require specific precautions as tall samples can crash into the SEM pole-piece.

1. Verify the CCD camera is not paused and the stage is in the lowered position.



1. Make the CCD window active by clicking on the stage image.
2. Change between the paused state and the active state by pressing the F6 shortcut key.
2. **NOTE:** Always watch the CCD camera when opening and closing the chamber door!
3. Press **Vent** icon located at the top right corner of the screen.
 1. Confirm you want to continue with venting the chamber.
4. The chamber icon, located at the lower right will turn orange indicating the system is no longer in high vacuum.
5. When you hear a hissing sound from the chamber is at atmosphere and it is safe to open the door and load your specimen.
6. Keep a close watch on the CCD image, carefully open the specimen chamber.
7. Using clean gloves, place your specimen sample in the desired stage location.
8. Keep a close watch on the CCD image, carefully close the specimen chamber.

9. Gently hold the chamber door closed, press the **Pump** icon.
 1. Continue to hold the door closed until the door seals and the roughing pump initiates.
10. From the Stage pull down menu, select **Home Stage**.
11. Wait until the chamber icon changes to green, indicating the chamber is under high vacuum.

Preparing to Image a Specimen

There are many components to optimizing the resolution of an image. The specimen must be at the correct working distance, the correct beam parameters must be selected (accelerating voltage, spot size, aperture size) aperture alignment, beam stigmation, the imaging detector and its set-up parameters. In addition to the SEM, ultimate resolution is also dependent on the specimen, its preparation and how it is mounted.

Setting the Working Height

The optimum working distance and eucentric height for the NanoSEM is 5mm. There is a 5mm marker on the CCD image window to help establish the specimen height.

1. Verify the **Touch Alarm** is enabled (checkmark).
 1. Under the **Stage** drop down menu verify the **Touch Alarm Enable** is selected.
2. Wait until the specimen chamber has reached high vacuum and the chamber icon turns green.
3. With the mouse icon on the CCD image window active, press down on the center mouse wheel and drag the arrow upwards.
4. Continue to raise the stage until the top of your sample reaches the 5mm marker.

Setting the Electron Beam Parameters

1. Verify the Touch Alarm is enabled.
2. Select aperture #6.
3. Activate the SEM image, but selecting the window and using the F6 key to un-pause the image.
 1. The spot size and accelerating voltage depends on your sample and imaging needs. As you gain experience find the setting which work best for your specimen. To start, use the following initial settings.

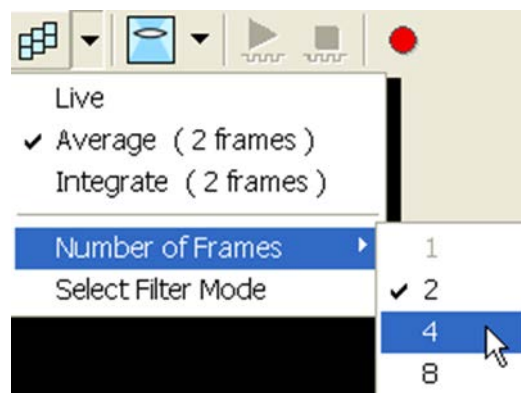


2. Spot Size: 5
3. Accelerating Voltage: 10 kV
4. On the right parameter panel go to the detectors menu and verify the **ETD** detector and **Secondary Electrons** are selected and the **Grid Voltage** is set to 250V.

1. A SEM image window must be the active window.



5. Check the chamber vacuum icon to verify chamber is green and under high vacuum.
6. Dwell time, resolution, and filter mode are adjustable parameters. As you gain experience find the setting which work best for your specimen. To start with, use the following initial settings.
 1. Dwell Time: **2x50 ns**. The time the beam spends on a single pixel per raster scan.
 2. Resolution: **1024x884**. The pixel density of the SEM image.
 3. Image: Average and Number of Frames 2. Clicking on the down-arrow next to the icon (filter mode) displays menu items Live / Average / Integrate, Number of Frames enabling to select number of averaged or integrated images



7. Verify in the Field Free Mode is selected.



8. Click on the **HV** icon to turn on the electron beam.
9. Decrease the magnification to the lowest magnification.
10. Use the Course and fine focus knobs to bring your specimen in to focus,
11. Adjust brightness/contrast using the **Contrast** knob, then the **Brightness** knob.
 1. An alternative way is to use the Auto Contrast Brightness (Short key: F9), the videoscope (Short key: F3) or the histogram under the Scan menu.
12. Move to the stage and find a specimen area with high contrast or the corner of specimen

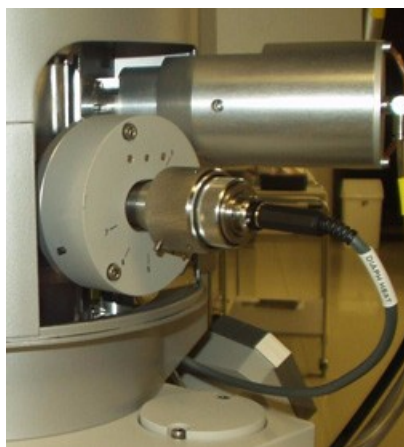
13. Use the Left mouse button and double-click on the feature of interest. This will automatically move this feature to the center of the SEM image.
14. Continue to increase the magnification and focus until you to get the best image in the 5kx magnification range. Optimization of the image is covered below.
15. Look at the CCD image (make sure it is not paused).
 1. If the specimen has moved away from the 5mm marker, bring the specimen height back to the 5mm marker.
16. **Link Z to FWD** sets the Z coordinate value to the actual Free Working Distance (FWD).
 1. Focus on the highest point of your sample.
 2. Under the **Stage** drop down menu select **Link Z to FWD**.
 3. If your specimen is properly focused, click the OK icon.
 4. Anytime the z-height of the stage is changed, or if you see the Link Z to FWD with red bars, click on the icon to reset the stage value.

Optimizing a Specimen to Image

Obtaining a high resolution image requires the correct alignment of the electron source, the column components and the electron beam (not available for the lab member). The lab member is responsible for the correct alignment of the Aperture, and Stigmator alignment. Optimum resolution may require multiple adjustments as the magnification is increased.

Aperture Alignment

Aperture alignment is during each SEM session and every time the aperture is changed. A poorly aligned aperture is evident when the image rotates as you go through focus. The goal of the aperture alignment is for the image not to move as the image goes through focus.



1. Focus on the specimen surface. The required magnification depends on the current alignment of the aperture.
 1. Very poorly aligned apertures, require lower magnifications during the alignment process
 2. High resolution images require the aperture alignment be carried out at very high magnifications.

2. Move a pronounced feature to the center of the image.
 1. Double clicking on a feature with the left mouse, brings the feature to the center of the image.



3. Select **Lens Alignment** by clicking the left mouse button on the toolbar icon.



4. The green target cross will appear at the center of the image.



1. Left mouse click and hold on the stable portion of the image. Move the 4-arrow icon to the center of the SEM image.
 2. Repeat this process at higher and higher magnifications until the center of the SEM image no longer rotates.
5. When the aperture is aligned, switch off the **Lens Alignment**. There should be no image shift when the focus control is used.

NOTE: The Lens Alignment and all alignments may require many iterations to gain the best possible image and the precession of the alignments become more critical as the magnification of the image increases.

Stigmator Alignment

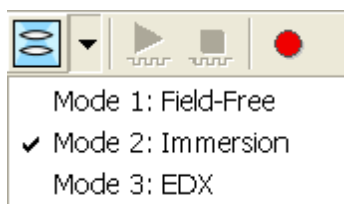
If an image elongates when going through focus, the beam requires stigmator alignment. A poorly stigmatized beam drastically decrease the resolution of an image. Stigmation is difficult and takes practice in getting the stigmation correct.

1. Adjust Focus and Contrast and Brightness to get the best possible image.
 1. It is often easier to start at a lower magnification and work your way up, similar to the Aperture alignment process.
2. Switch the image collection to the fast scan.
3. Adjusting the stigmation, is very similar to adjusting the focus.
 1. Adjust either the x- or the y-stigmator, trying to achieve the best image.
 2. Switch to the other stigmator and again adjust for the best possible image.
4. The higher your resolution and magnification requirements, the more often you will need to repeat the total alignment processes. For the best image, try to align at a higher magnification than what is required for your work.

Immersion Mode

The highest resolution for this SEM is using the Immersion Mode (also referred as the Ultra-High Resolution, UHR). This option is not available at lower magnifications. The default detector for this mode is the secondary electron detector. The detector is automatically selected when the immersion mode is chosen.

1. Prior to selecting the **Immersion Mode** you must have the sample must have the **Link Z to FWD** correct.
 1. Verify the sample is at the 5mm working distance and is in focus.
 2. Click on the Link Z to FWD icon.
 3. Verify the Link Z to FWD icon is blue, without any red features.
2. Using the mode pull down menu, select Mode 2: Immersion



3. Alignments from the Field-Free mode are not be carried over to the Immersion mode. You will need to go through the alignment process describe above in order to get a high resolution image.
4. You may need to repeat the alignment sequence multiple times to achieve the highest quality image.

Taking and Saving Images

1. Capturing an image is done by pressing the **F2** key. This uses preset scan settings to capture the image.
2. At the completion of the image capture a Microsoft save window opens.
3. All images are stored on the support computer in the Shared Data File.
4. Save your image in the correct data file.
 1. JPEG only, for best compatibility choose 8bit TIF
5. At the end of your session use the Support computer to retrieve any files you wish to remove.

Unloading Samples

1. Using the pull down menu, switch back to the **Field-Free** mode.
2. Center the stage (**Ctrl+O**).
3. Click on the **HV** icon to turn on the electron beam.
 1. Listen to hear the HV has been turned off.
4. Click on the **Vent** Chamber icon.
 1. The stage will lower down to safe position.
 2. Listen for the chamber to vent to hear when the chamber has reached atmosphere.
5. Carefully pull out the chamber door while watching the CCD to make sure the stage does not hit any chamber components.
6. With new, clean gloves, remove your sample.
 1. **Never touch anything inside the chamber other than your sample.**

7. Close the chamber door. While holding the chamber closed, press the **Pump** icon.
8. While the chamber is pumping down, update the SEM log sheet and clean up the area.
 1. **SAMPLES LEFT ON THE BENCH WILL BE ASSUMED AS ABANDONDED AND THROWN AWAY**
 2. There should be NO tweezers, samples, or wipes left around the microscope
9. Before leaving the area, make sure the **Chamber Pressure** is $<10^{-6}$ torr.
10. Log out of the SEM software, making sure to leave the server running.
11. Log off Badger, entering any Comments, Problems or Shutdowns you encountered.
12. Copy any images from the Support Computer to a flash drive, but never insert a flash into the Control Computer

Appendix

A) FEI Operation Shortcuts

Mouse Button Functions

Left button

- Click, drag and release to zoom into a selected area
- Double click to bring object to center of screen

Shift + Left button

- Beam shift

Right button

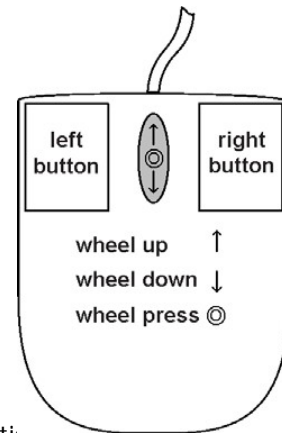
- Click and drag right/left to focus image

Shift + Right button

- Click and drag left/right and up/down to correct astigmatism

Wheel press

- Hold and drag in the desired direction for stage movement



Keyboard Functions

F1- Displays the FEI factory manual

F2- Starts slow scan for photo; save as box will appear after scan F3- Videoscope On/Off

F4- Starts Snapshot; F4 twice will active a fast scan F5- Single/Quad mode

F6- Pause / Unpause

F7- Reduced Area / Full frame mode Shift F12- Toggles Scan Rotation

Ctrl + R- Restarts slow scan for photo Ctrl + S- Save image

Ctrl + 0 (zero)- Centers X and Y of stage

+/- Increases/Decreases magnification 2x

* Rounds off magnification to the nearest round value

B) Sample Considerations

The following chart describes materials that are permissible within the microscope:

ALLOWED	NOT ALLOWED
<ul style="list-style-type: none">-IC chips and MEMS devices-Cured photoresist, PDMS, or other polymers-Ceramics, glass, or insulating materials-4-inch wafers to pieces-Side and tilted views	<ul style="list-style-type: none">-Magnetic materials-Biological Samples (without prep)-Outgassing materials:<ul style="list-style-type: none">-Uncured photoresist-Oils, solvents, or any liquids-Loose powders

Sample preparation

A set of sample preparation mounts, conductive tape, and tweezers are available for communal use in the SEM room. All of these materials must be placed inside the clear boxes to avoid dust and contamination. ONLY use vacuum compatible tapes provided by CNM2. Samples should be thoroughly degreased and dried to eliminate outgassing from organic contamination and water. Samples can be cleaned with solvents such as isopropanol and then dried using compressed nitrogen. Loose surface particles from cleaving wafers can also be removed by blowing the surface with nitrogen.

Most samples should be mechanically clamped using the copper pin-clip sample holder (**Fig. 1**). If the sample is too large or too small for the pin-clip holder, then these samples can be mounted using adhesives (**Fig.2**). Large pieces and crushed pellet samples may be mounted onto a stub with adhesives or paste. **Please contact staff for direction on mounting powders or imaging biological samples.** Silver paste must be left to cure overnight or for one hour with the use of a sample heater. For good measure, there is pressurized nitrogen available to remove any dust or loose debris from the sample's surface.

45/90-degree angle sample mounts are available for side and tilted sample views.

Samples that have insulating properties will create charging effects that will cause image distortion or drift. These effects are caused by electrons building on the sample's surface. Therefore, it is necessary to create a conductive path for these electrons to prevent them from accumulating on the sample's surface. This is done by using conductive holders, adhesives, and in addition sputter coating a thin layer of conductive material on the surface.

It is highly recommended to sputter coat insulating samples (oxides, cured polymers, etc) with a thin layer of conductive material. First mount the sample either with a pin-clip holder or on top of stub with conductive adhesives (Fig. 1,3). Then use the QuorumTech150 to coat a thin layer (1-10nm) of carbon or gold. After the sample has been coated it can go directly into the SEM for imaging.

See: *QuorumTech150 manual for more information on its operation*

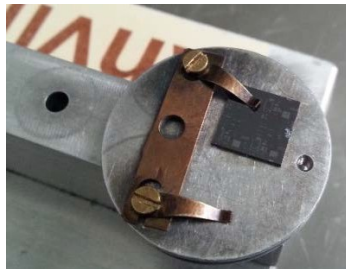


Fig 1. (Left) Sample mounted using pin-clip holder.

Fig 2. (Right) If samples are oddly shaped, too small, or too big for the pin-clip holder, then using adhesives is acceptable for these cases.