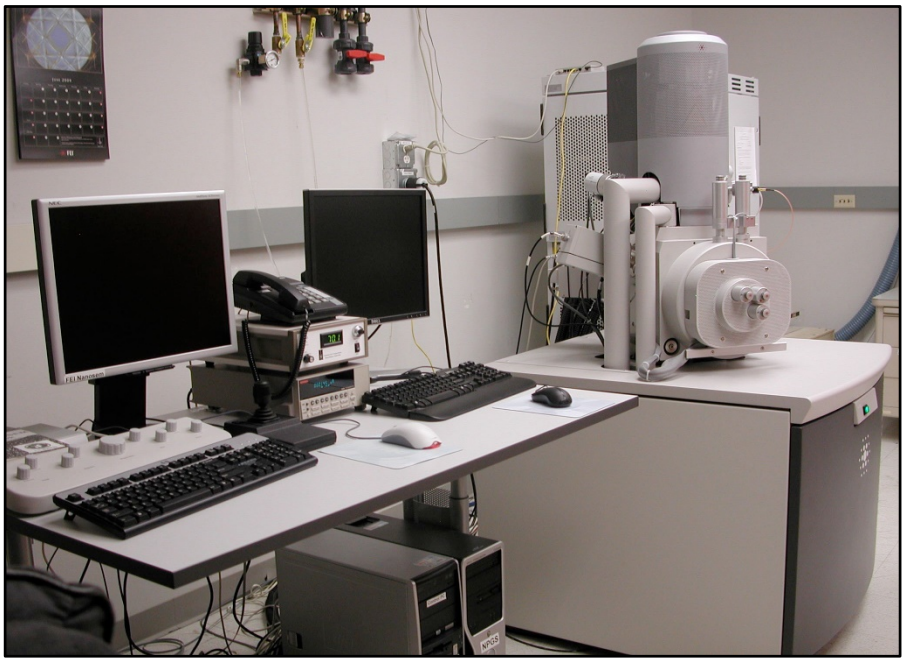
FEI 430 Nova NanoSEM



Introduction

The FEI 430 NanoSEM is a field emission scanning electron microscope with 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.

Characteristic	**Light Microscope**	**Scanning Electron Microscope**	**Transmission Electron Microscope**
Principle of Detection	Light passing through glass lenses	Scattered Electrons from field emission source using electromagnetic principles.	Transmitted electrons through thin sample from field emission source using electromagnetic principles.
Data Gathered	-Sample surface -Large area -2-D images about the morphology of any large structure	-Sample surface -Less than 1 mm area -Topography and chemical contrast of powders, IC chips, etched microstructures	-Internal composition and atomic arrangement -Approx. 1 micron x 1 micron -Internal structure of dislocations, tiny precipitates, grain boundaries and other defect structures
Sample Prep	None	Mount to sample stub. Insulating, biological, and powder samples require extra sample preparation.	Must be electron transparent (50-100nm thin) Sample viewing area limited by quality of sample prep
Expected Resolution	Microns	Several nanometers	Several Angstroms

Tool Capabilities

- Up to 5nm of Resolution
- Backscatter and Secondary Electron imaging with Immersion Lens Mode
- Working Distance: 5mm, Max HV: 30KeV with up to 5KeV stage bias
- Platinum Deposition
  Electron Beam Lithography see: NPGS Electron Beam Lithography Manual
  For EDS, EBSD, or FIB capabilities see: Oxford EDS, Oxford EBSD, or Scios FIB manual

Suggested Documentation

"A Guide to Scanning Microscope Observation" JEOL USA Electron optics documents

FEI System Operation Manual

www.fei.com/resources

Tool qualification policy

This document is intended to supplement but does not replace formal training and qualification by staff. In order to gain unsupervised access, the user must attend a 2-hour demonstration and then pass a qualification test. The lab member must demonstrate safe and knowledgeable operation of the microscope without any interference from the instructor. Please contact Rijuta Ravichandran (rravicha@ucdavis.edu) to set up a demonstration, practice, or potential imaging service work.

Precautions and safety

Please take into consideration the following when working around the microscope:

ALWAYS	NEVER
Wear gloves when touching anything that goes into the chamber: -Samples -Sample holders or mounts -Any part of the stage	**Touch items that make contact with the chamber with your bare hands or contaminated gloves:** -Do not touch face or clothes -Do not touch computer -Do not touch dusty surfaces
Exercise care when working around the stage, it is fragile	**Open or close the door too quickly**
Watch the CCD camera to ensure the samples and the stage is clear from the other microscope parts (i.e. pole piece)	**Make large stage movements, or tilt stage without watching the CCD camera in quad-screen viewing mode**

Sample Considerations

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

ALLOWED	NOT ALLOWED
-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	-Magnetic materials -Biological Samples (without prep) -**Outgassing materials**: -Uncured photoresist -Oils, solvents, or any liquids -Loose powders

Sample preparation

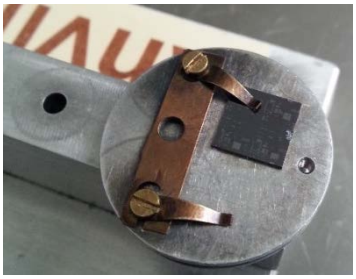
A set of sample preparation mounts, conductive tape, and tweezers are available for communal use in the SEM room. All of these materials must be placed inside the clear boxes to avoid dust and contamination. ONLY use vacuum compatible tapes provided by CNM2. Samples should be thoroughly degreased and 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



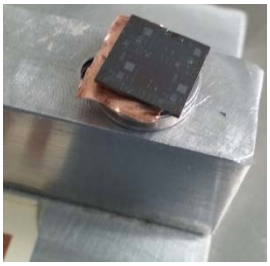
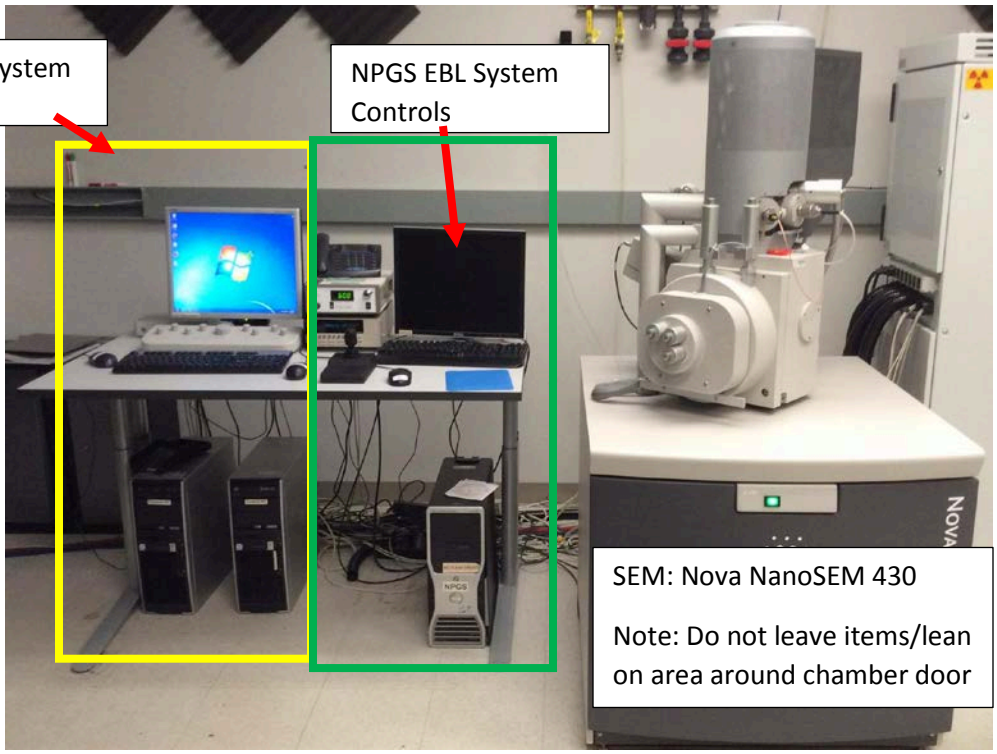
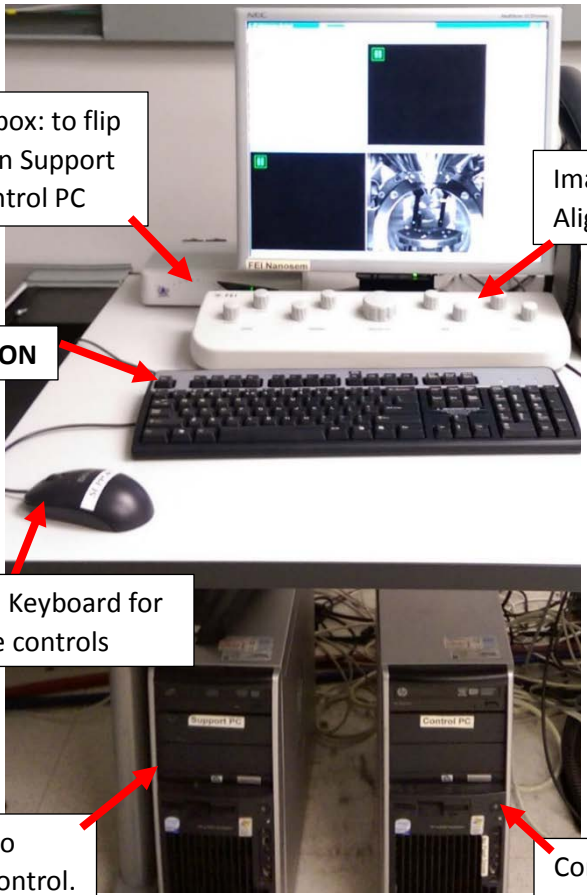


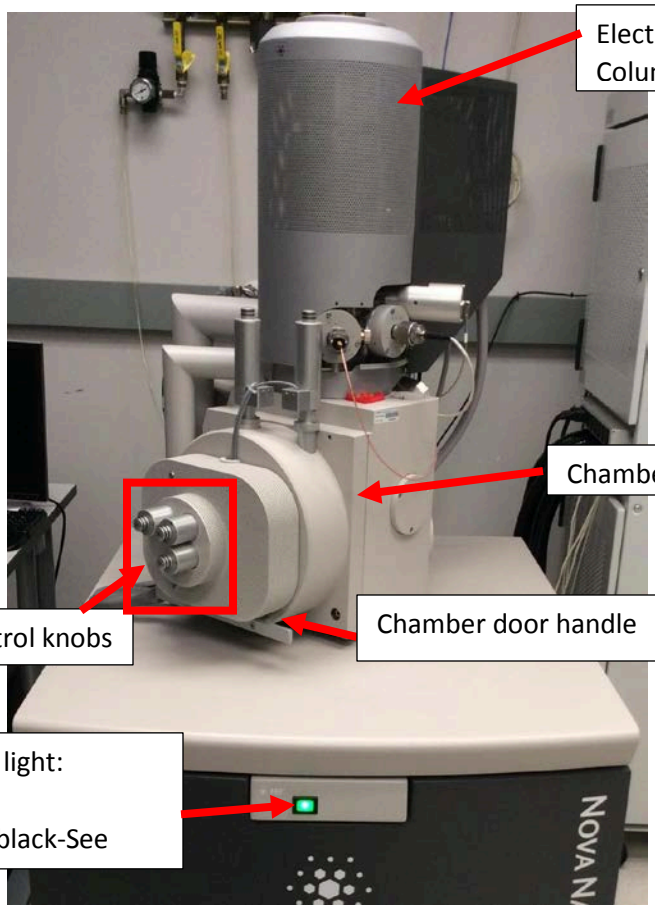
Fig 1. (Left) Sample mounted using pin-clip holder. Most samples can be mounted this way.
Fig 2. (Right) If samples are oddly shaped, too small, or too big for the pin-clip holder, then using adhesives is acceptable for these cases.

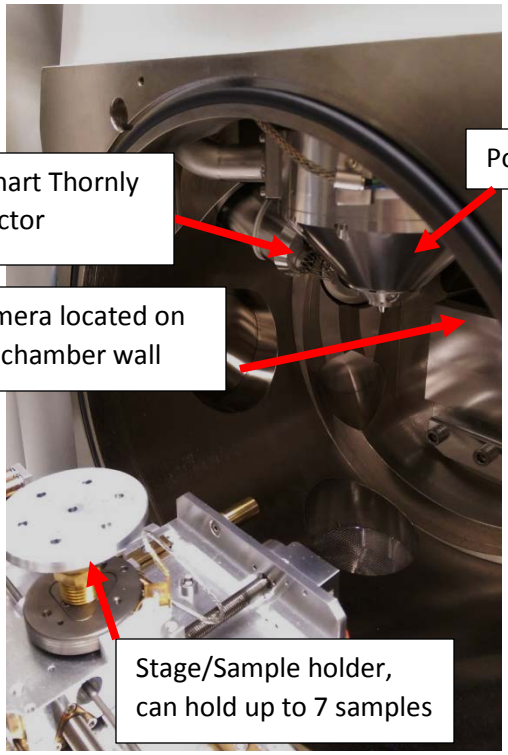
System Overview

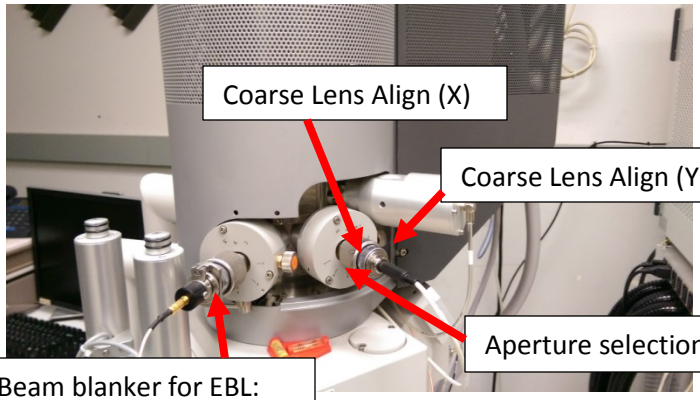
Hardware



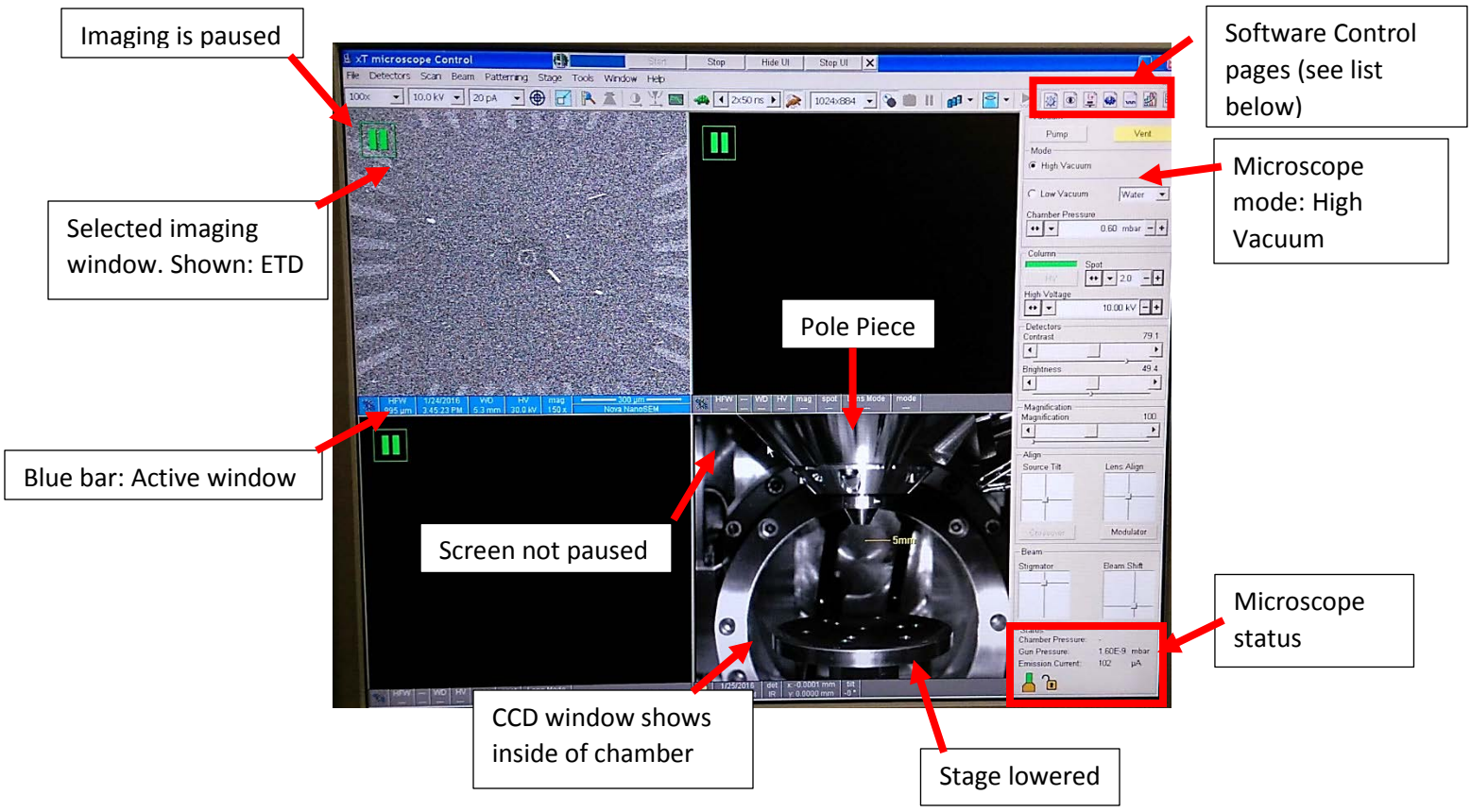



Electron Column
Chamber
Stage control knobs
Chamber door handle
Operating light:
Green-OK
Yellow or black-See
NOVA N


Everhart Thornly Detector
Pole Piece
CCD camera located on back of chamber wall
Stage/Sample holder, can hold up to 7 samples


Coarse Lens Align (X)
Coarse Lens Align (Y)
Aperture selection
Beam blanker for EBL:
DO NOT TOUCH

Software

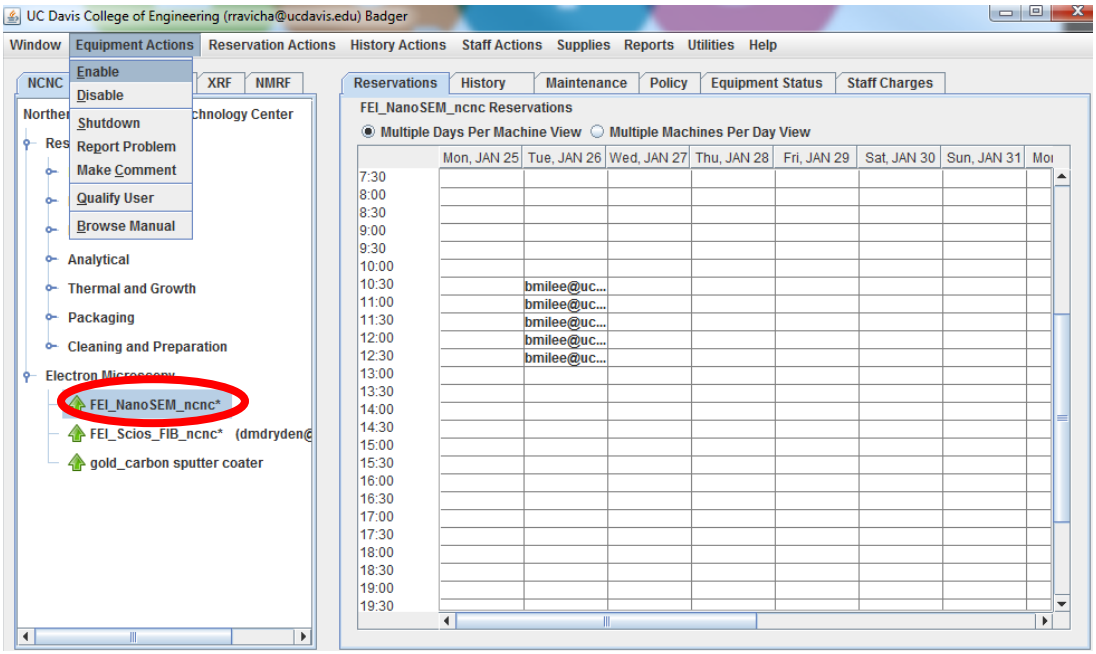


Page List	Main Features
Beam Control	Pump/Vent chamber, Beam On/Off, Column: Spot Size and High Voltage, Alignments, Microscope Status
Detectors	Detector Settings: Detector type and mode, Grid Voltage
Beam Deceleration Control	Apply beam deceleration voltage or stage bias
Navigation	Move stage position, 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

Operation

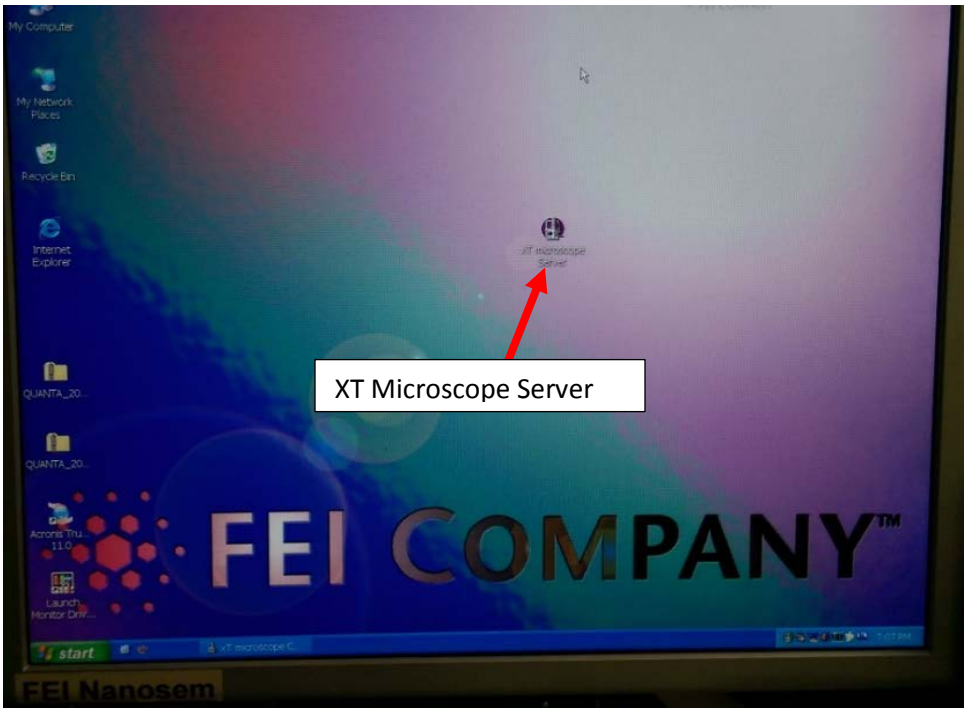
System pre-check

Make sure you have logged into Badger and have checked the logbook and Badger for any issues with the tools. Then Enable the tool on Badger.

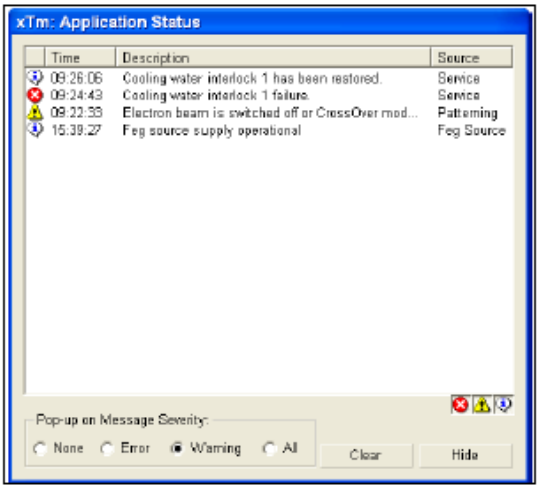


Make sure the green light on the front of the machine is on. If it is yellow or turned off then do not use the machine and report the problem to staff.

If the Microscope XT program is not open already, find the XT server icon and double click to start the program.

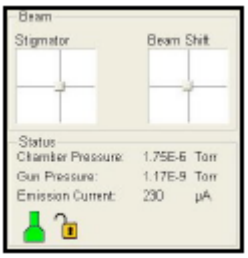


As soon as the program is open, the log-on window will pop-up.

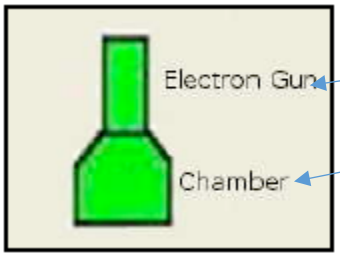


After you have logged into the program, the application status window may pop-up. If it does, then alert staff with any issues that is displayed (I.e. Cooling water interlock failure)

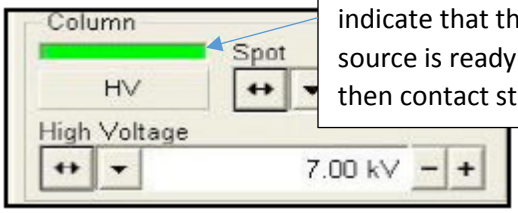
If there are no issues, then check the status window in the bottom right of the screen.



Chamber pressure: 1x10^-6-1x10 ^-5
Gun pressure: 1x10^-10 to 1x10^-7
Emission current: 100uA, or check previous log book entry (+/- 10% of this value)



The bright green symbols for both electron gun and chamber indicate that the microscope is at high vacuum.



Check for the bright green bar to indicate that the electron emission source is ready. If this bar is white then contact staff

<u>Start-up and Loading Samples</u>

If all steps from the pre-check list are completed without concern, it is OK to continue.

- Press “Vent” in the top right corner of the screen, a message will pop-up for the user to proceed with venting the chamber.

MAKE SURE THE SYSTEM IS ON “HIGH VACUUM MODE”

The bright green section of the chamber icon will turn orange to indicate that the system is no longer in high vacuum. Once you hear the hissing sound from the chamber, it is ok to open the door and load in the samples. **MAKE SURE CCD CAMERA IS UNPAUSED BEFORE PROCEEDING.** (Short key: F6)

Always watch the CCD camera when opening and closing the chamber door!!

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

- Press “Pump” to bring the chamber back under high-vacuum. Place one finger on the door until the door seals and the roughing pump initiates.
- While the chamber is pumping down, raise the stage such that the surface of the sample is in line with the 5mm marker. To do so, press down on the center wheel and drag the arrow upwards.

After the sample has been raised, click on the imaging window and continue to set up the imaging parameters.

- Select the appropriate **spot size** and **accelerating voltage** for your sample

- Go to the detectors menu, make sure the system is using the ETD detector in secondary or backscatter electron mode and that the grid voltage is set to 250V.

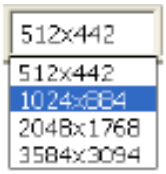
Once the chamber is under high-vacuum (check microscope status icon), you will be able to turn on the beam and begin imaging.

<u>Imaging</u>

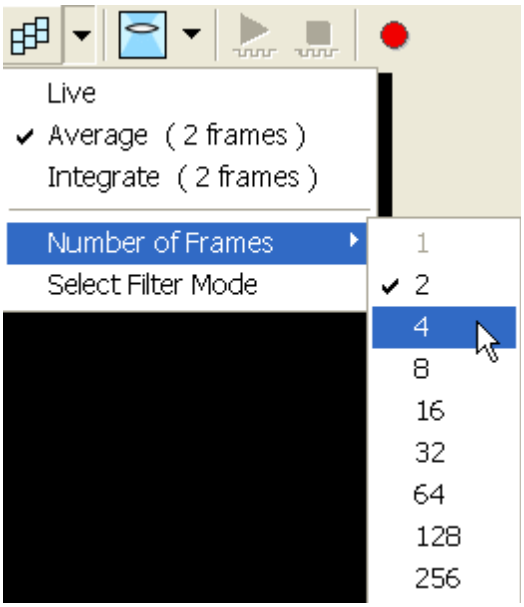
- Turn on the Beam by pressing the “HV button” —you will start in field free mode

- Adjust the **dwell time**, **resolution**, and **filter mode** (number of frames)

Dwell Time (tdwell): The time the beam spends on a single pixel per pass. Changing this influences the Total Depth and Total time, assuming a constant Number of passes.



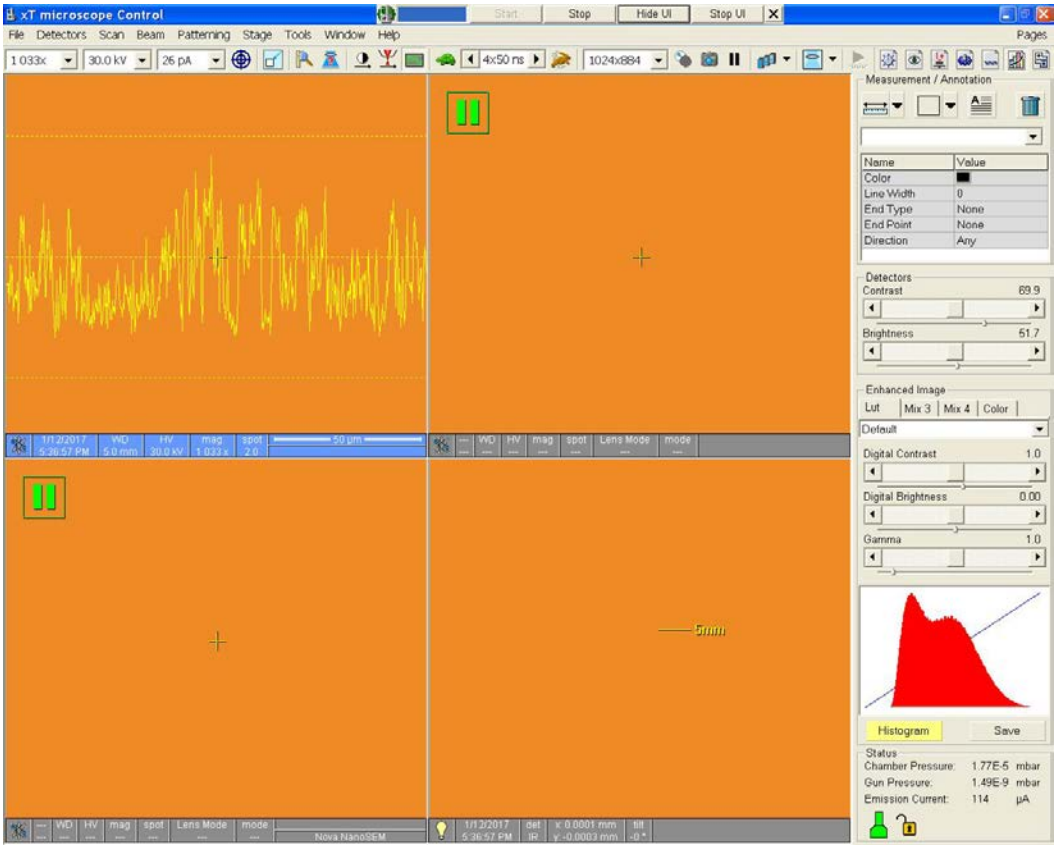
Resolution: no. of points or pixels, Width x Height (image resolution)



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

- Lowest magnification
- Move to the location of the sample and find area with high contrast or to the corner of sample
- Full screen (Shortkey : F5)

Adjust brightness/contrast using the videoscope (Short key: F3) and/or using the histogram under the processing menu. Watch out for bright and dark peaks.



- Start adjusting the focus with the knobs or the right button on the mouse. After reaching 2-5Kx mag, if you find that you are far away from the 5mm marker or ideal working distance, then link the working distance. To do this, you must press the "Link Z to FWD" button on the toolbar and then keep an eye on the z-height using the navigation menu.

- Stigmator and lens align after increasing the magnification. If there is no misalignment seen, increase the magnification and continue to adjust the lens align, focus, and stigmator.
- After being completely aligned and focused at 40-50kX or higher magnification then link the working distance one more time, fine tune the stage height if necessary. If between 4.8-5.2 mm, then working distance should be ok for most uses.

Immersion Mode (Optional)

Immersion mode provides the opportunity of high resolution imaging especially for features smaller than 1 micron and light weight elements. Larger features often don't require going into immersion mode. If immersion mode is desired, the following is necessary:

- Must having the working distance linked (Link Z to FWD)
- Variables must be in sync: Working distance, Accelerating Voltage, and Magnification (Generally, we have the working distance set to 5 mm, and the mag above 2000x)
- Very slowly adjust the fine focus knob until you see an image. There will be an image shift and the image will be defocused, but most lens align and stigmator alignments are not lost.
- Adjust the brightness and contrast again using the videoscope and/or histogram.
- Refocus and realign the images as needed while increasing the magnification.

Taking a picture

After aligning the beam, in order to take a picture at the desired region, please take note of the following:

- Go 2-3 times the magnification at which you want to take a picture and align the beam through lens align, focus, and stigmator. (Recommended)
- Press F2 to take a slow scan photo, and it will start a slow scan with the default scan settings.
- During photo scan you can adjust Brightness/Contrast, and other scan settings as necessary. If you have adjusted any of these settings, then retake the photo with the adjustments.
- At the end of the scan a dialogue box should open to save image to shared data folder
- If samples have a lot of charging, then switch to integrate mode
  - Trade-off between noise and frame rate
  - File -> Save as -> Save into shared data folder

Saving images

- Must be in shared data folder
- JPEG only, for best compatibility choose 8bit TIF
- Switch to the Support PC in order to move the files off the computer

Unloading Samples

- Switch back to field-free mode
- Turn off Beam (wait for sound!)
- Vent Chamber (stage will start lowering down to safe position)
- Pull out samples, and immediately pump down the chamber
- While the chamber is pumping down, clean up your area
  SAMPLES LEFT ON THE BENCH WILL BE ASSUMED AS ABANDONDED
  There should be **NO** tweezers, samples, or wipes left around the microscope
- Home stage (Shift + F3) when all samples are removed from the chamber
  MAKE SURE SYSTEM IS UNDER HIGH VAC BEFORE WALKING AWAY
- Log off the system
- Leave server running
- Log off Badger