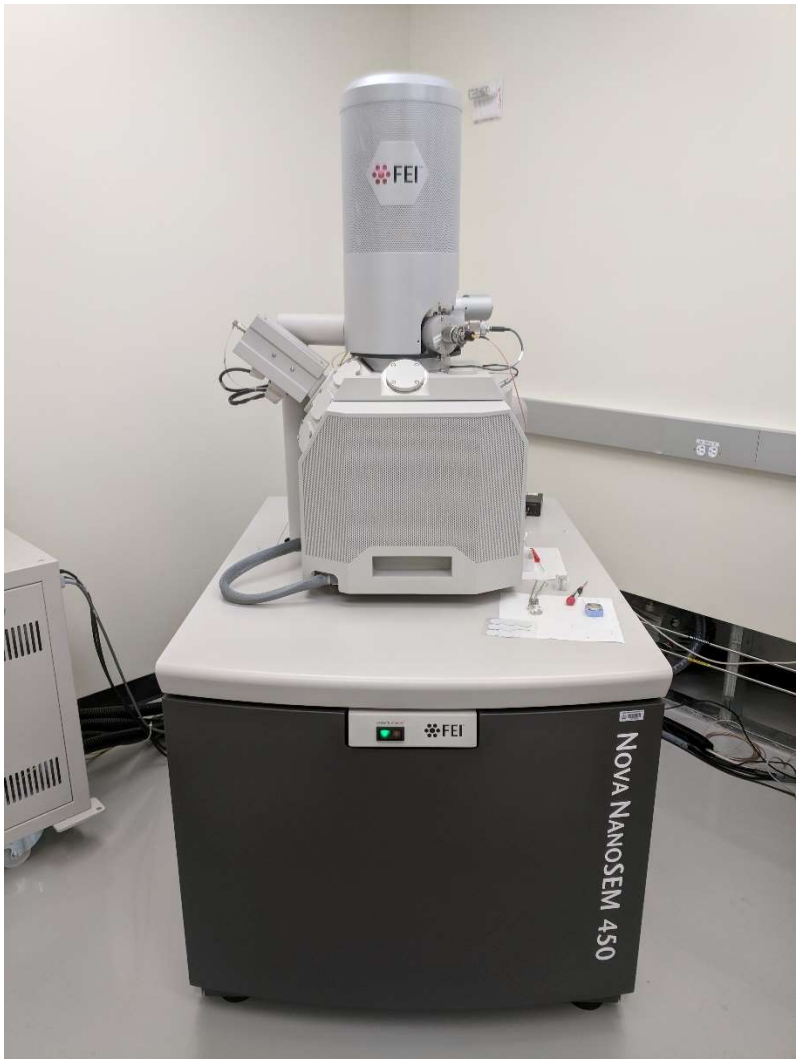
COLUMBIA UNIVERSITY
IN THE CITY OF NEW YORK
COLUMBIA NANO INITIATIVE / CENTER FOR INTEGRATED SCIENCE AND ENGINEERING

FEI (Thermo Fisher) Nova NanoSEM 450



These instructions are intended for reference only, and will *not* replace the thorough training required for proper system operation. Contact a clean room staff member with questions or to report a system problem.

Written by Dr. Philippe Chow

I: Accessing and loading the microscope:

1. Enable the tool *NovaNano SEM* in **BADGER**

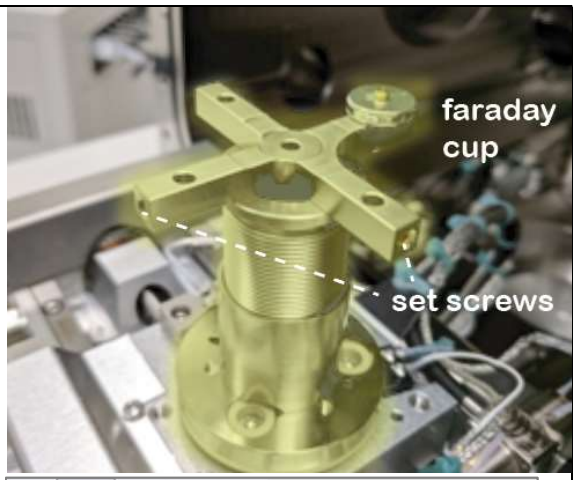
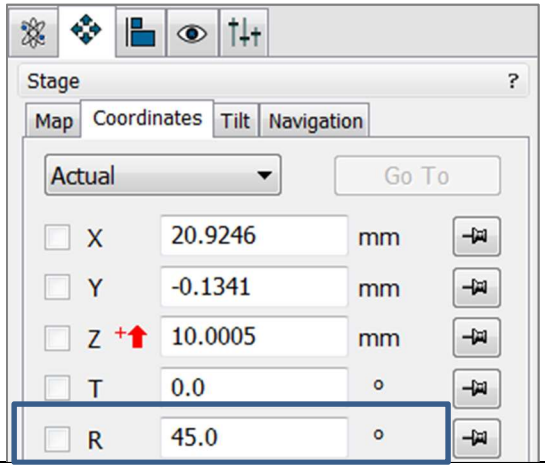
2. **Mount sample** onto one of the holders with either clips or double-sided Cu tape.

IMPORTANT:
Prohibited sample types include powders (nanoparticle samples may be dispersed on an acceptable substrate material) and magnetic materials (which may not be imaged in 'Immersion mode').

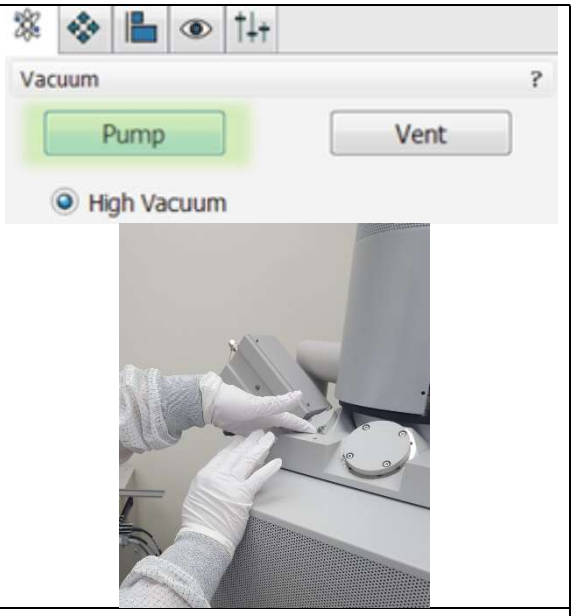
Standard sample holder

It is important to ensure *good electrical contact* between the stage and the surface to be imaged.

3. **Vent** chamber using VENT icon on right panel. Allow 2-3 minutes for the chamber vent.

4.	**Load sample:** • Carefully slide the door/slide assembly out. • Place holder stub onto the stage in one of the holes. The Faraday cup may need to be removed for larger holders and can be placed in its box. • Secure holder with set screw. • Do not manually move the stage! Use stage control if rotation is needed.	 
5.	Carefully close chamber door while observing IR camera in the lower right quadrant of the user interface to *ensure sample does not hit pole piece*.	

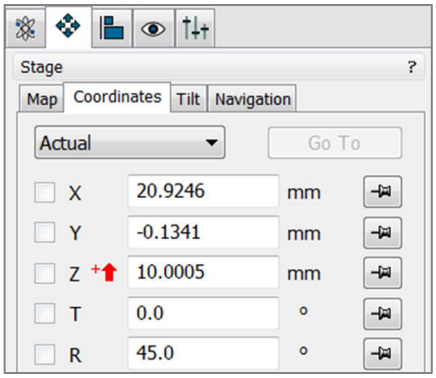
6. **Pump** chamber using PUMP icon.
Gently push on door to ensure that it seals.

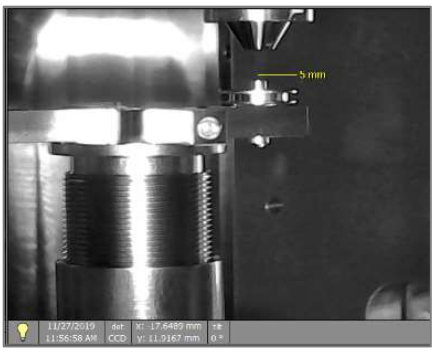


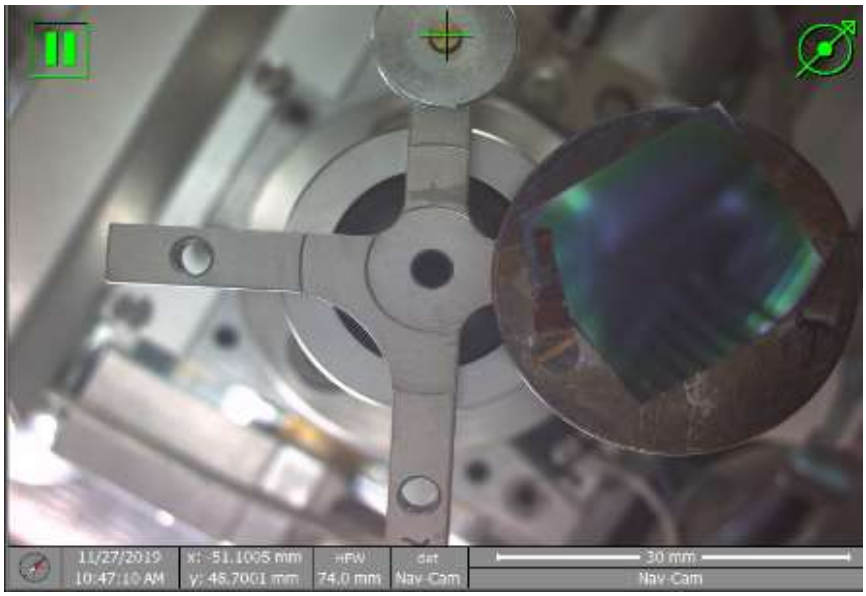
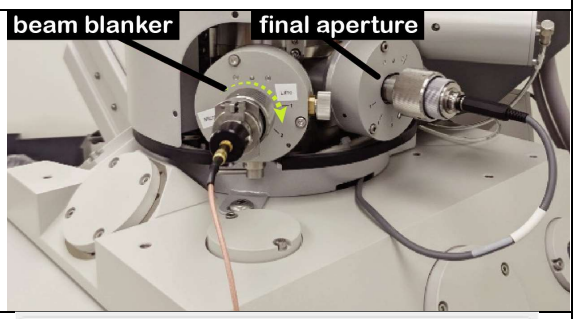
7. While pumping, **move the sample up** to the 5mm working distance line (see IR camera in Quad #4).

CAUTION: DO NOT CRASH SAMPLE INTO POLE PIECE!

- Ensure that IR camera (4th quadrant) is not 'paused'.
- Roughly navigate to bring your sample's tallest feature under the pole piece.
- Use the drop-down menu to select "Relative" motions (from "Actual").
- Set 'Z' motion to values of 5-10 mm and press "**Go To**". If sample is nearing the pole piece (i.e. visibly moving past the 5mm line), click '**Stop**' immediately.
- Adjust so that the top of your sample is roughly aligned to the 5mm line (see yellow line in chamber scope).





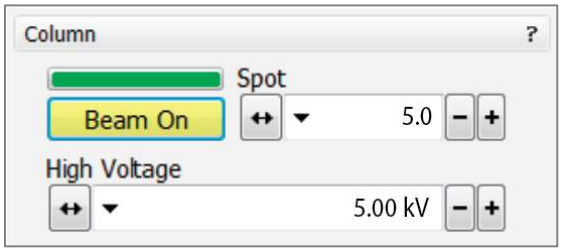
8.	**Take a NavCam photo** of the stage (Ctrl + Shift + Z). The plan-view image of the stage will allow for easy sample navigation and is located in the lower left quadrant.  Example Nav-Cam photo
9.	Check that the <u>beam blanker</u> is switched to the "Microscopy" setting for imaging. The <u>final aperture</u> is set by the manufacturer. **Do not adjust this knob. Control of final aperture occurs via software.**	
10.	**Wait** for the chamber to pump down to a base pressure of at least 4e-5 Torr (on the vacuum tab).	

II: Optimizing imaging conditions:

It is important for users to have an understanding of the principles of microscopy as is relevant to their particular requirements (sample type, image resolution and beam/sample interaction constraints, etc.). The settings presented here serve as a starting point for users to find the optimal conditions for their work. We present some guidelines for image optimization in the appendix of this standard operating procedure.

1. Set the "**Spot" = 5.0**, which corresponds to a final aperture diameter of 30 µm. Set the acceleration voltage to **5.00 kV** as a starting point.

Turn on the beam ('Beam On' turns yellow) and "un-pause" the 1st quadrant screen.



The microscope will be in its default 'Field-free' imaging mode, which uses the Everhartt-Thornley Detector (ETD) in secondary electron imaging mode. This is standard for lower-resolution imaging. If higher resolution is needed, Immersion Mode is recommended (see **Step 2.8**).

Check for visible features by adjusting magnification and focus. Adjust the contrast and brightness (manually or by using the 'Auto Contrast/Brightness' feature, located on the upper toolbar). 

Use the Stage control menu on the right-hand side to change the stage position, tilt and rotation. Sample navigation can also be performed by double-clicking on a position on the Nav-Cam photo (lower left Quad) or the scanning quad (upper left Quad).

2.	**Focus on your area of interest:** Locate a distinct feature at ~1000x magnification and obtain a rough focus using the coarse/fine adjustment knobs. If image is translating/shifting while focusing, proceed to **Step 2.4** for lens alignment. Use the Reduced area window*, which allows for rapid scanning. 
3.	**Adjust for astigmatism:** Adjust for the astigmatism aberration, which appears as a directional shape distortion while focusing (as opposed to a translation), by adjusting the X and Y stigmator knobs on the manual user interface. Tip: Adjust the X stigmator until image comes into better focus, then adjust the Y stigmator until image is further improved. Then adjust the focus knob to further improve. Repeat the stigmator adjustment until further improvements are not visible. If, while adjusting stigmator knobs, the image translates or shifts, then stigmator alignment is required. Proceed to **Step 2.5**.

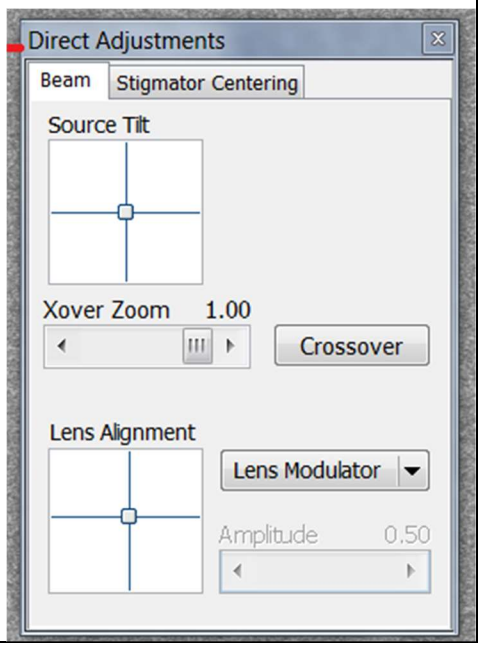
4. **Align the lens electronically:**

Open the Direct Adjustments window and select the Beam tab.

Zero the lens alignment by right clicking in the Lens Alignment crosshair box. See if focus has improved. If not, turn on the focus "Modulation", which will set the microscope to go in and out of focus. Adjust the Lens Alignment until the feature *translation* or shifting is minimized.

When satisfied, turn off the Modulation and adjust the *X* and *Y* stigmators on the manual user interface to obtain the clearest image. Repeat the cycle of focus, stigmator adjustment, focus, etc. until satisfied.

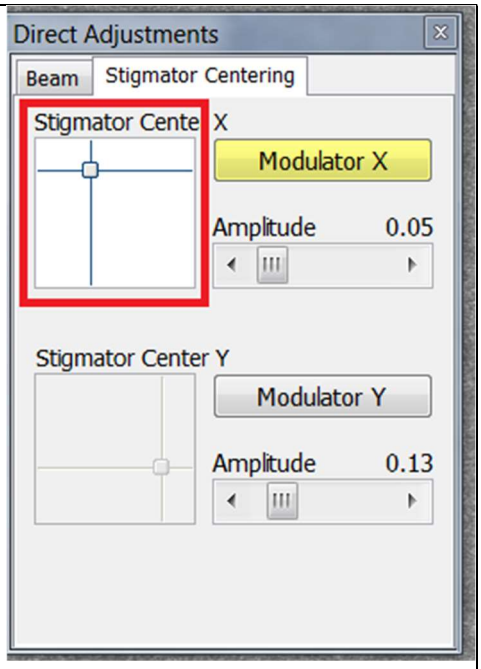
If the feature translates significantly during the *stigmator* adjustment, proceed to **Step 2.5**.



5. Align the stigmators electronically:

Zero the stigmator centers by right clicking in the Stigmator Center X and Y crosshair boxes. Check if shifting during stigmator adjustment has improved.

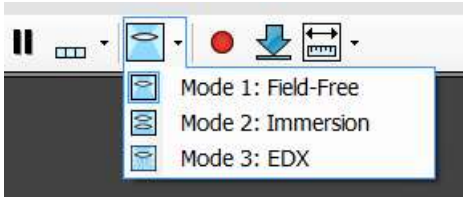
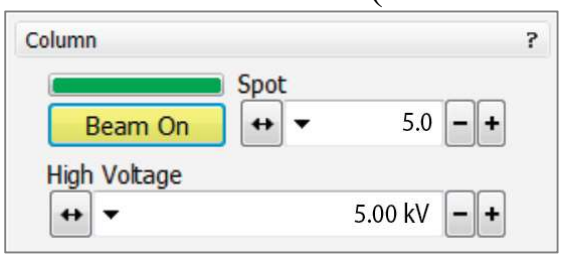
If not, click on the Stigmator tab and turn on the Modulator X. Adjust the modulation amplitude if the motion is jumpy. Adjust the Stigmator Center X until the feature translation disappears. Repeat for Stigmator Y.

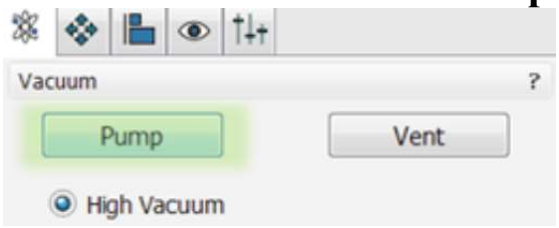


6. Link stage (not for e-beam lithography):

Once the stage is focused at a working distance of 5 mm, the stage can be linked so that the microscope can always know the distance from the sample to the pole piece. This sets an accurate WD and minimizes the change of damaging the pole piece.

Click the  icon on the upper toolbar when focused at roughly 10,000x magnification, and it will change to  , indicating that the stage is now linked. Sometimes, such as when switching imaging modes, or changing working distance, the stage needs to be re-linked. In this case, focus and perform the prior procedure again.

7.	**Capture and save an image:** Click on the Photo  or the snapshot  icon on the upper toolbar to scan a full frame and save the image. The scan duration can be modified as desired. Adjust the Dwell Time, Resolution and Scan mode (i.e. frame vs. line integration) as desired for your particular sample. Save files in the **USER IMAGES** folder shortcut. This folder is stored on the Desktop of the **Support PC** (not the Microscope PC) via local network connection. *Do not store images on the Microscope PC or they will be deleted.
8.	**Immersion Mode** is recommended for imaging small features (> 50,000x). Select this mode from the drop-down menu on the upper toolbar. Once selected, the immersion lens is switched on and the detector switched to the through-lens detector (TLD) in secondary electron imaging mode.  **NO MAGNETIC MATERIALS** are allowed when using this mode. **Align the lens and stigmators again**. This is a dual lens mode so getting ultra-high resolution requires another alignment. Perform the same focus/astigmatism procedure in this mode as in the field-free mode.
9.	**Turn OFF** the beam (reverse of **Step 2.1**). 

10.	**Vent** the chamber (as in **Step 1.3**). 
11.	**Unload** your sample (reverse of **Step 1.4**).
12.	**Close the chamber door and pump down** the chamber (See **Step 1.6**) 
13.	**BADGER LOGOUT:** Disable the *NovaNano SEM* in **BADGER** when finished.

III: E-beam lithography: *Nabity Pattern Generation System* (NPGS):

Overview: This section describes how to write a properly prepared pattern (i.e. *.dc2 files) using the NPGS e-beam writing system on the FEI Nova Nano 450 SEM. For information about pattern preparation, advanced writing features and pattern development, please refer to the NPGS documentation.

The NPGS tool gives the NovaNano SEM the ability to write patterns by systematically steering and blanking the electron beam, the latter of which is performed using the ScanService beam blanking module (always left in EXT mode).

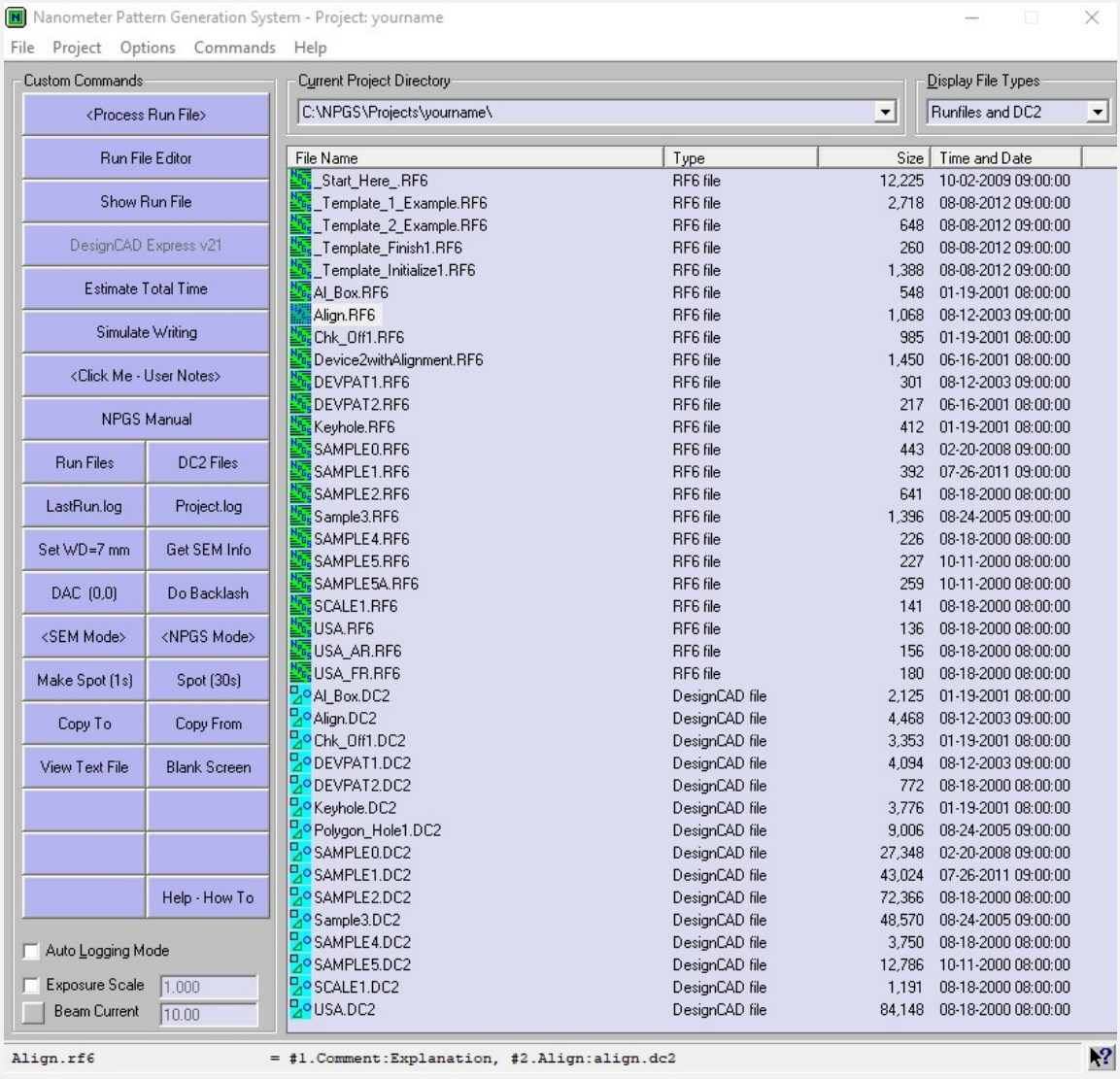


Figure 1: NPGS Menu user interface.

1.	**Perform Section I** of this document to properly access the Nova Nano SEM and load your sample for e-beam lithography. Note 1: For best reproducibility, the Faraday cup (see **Step 1.4)** should be placed in any one of the slots on the holder. Note 2: In order to obtain the correct focus in e-beam lithography. The microscope will need to be correctly in focus on the sample on which you intend to write. Therefore, it is necessary to have some feature, away from the writing area of interest, to serve as a focus reference. This could be a scratch (made with a razor or scribe), or a metal thin film feature (with high Z-contrast), a deep etched trench or metal nanoparticles dispersed on the resist surface.
2.	**Perform Steps 2.1 through 2.5** of this document to properly optimize the microscope on an area on your sample (WD = 5mm). If your sample is flat, optimize the image on the focus reference feature (i.e. the scratch The recommended accelerating voltage and spot number for standard e-beam lithography are **30.00 kV** and **5.0**, respectively. Do not link the stage as directed for imaging in **Step 2.7**.
3.	**Navigate** to the Faraday cup location and adjust focus as necessary on the hole in the center of the stub. Zoom in to high enough magnification such that the field of view is entirely in the Faraday cup hole, or use Spot scanning mode.

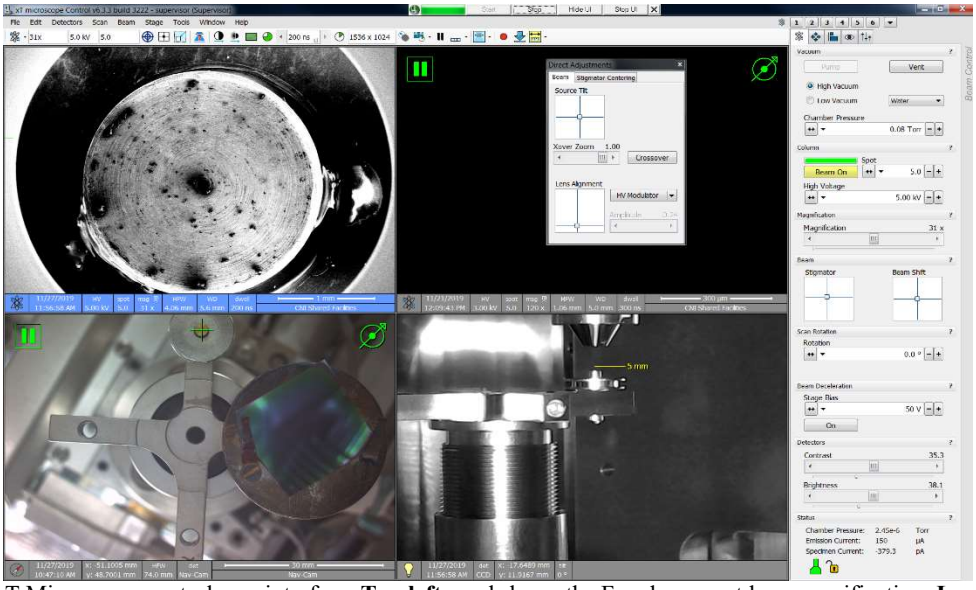


Figure 2: xT Microscope control user interface. **Top left** quad shows the Faraday cup at low magnification**. Lower left** quad shows the Nav Cam image for sample navigation and the **lower right** quad shows the IR chamber camera video.

Observe and note the specimen current on the bottom right-hand side of the side panel. Allow enough time for the current to stabilize. This is the beam current (pA or nA) that NPGS will use to calculate the appropriate dwell time (μs) for a prescribed e-beam dose ($\mu C/cm^{-2}$).

4. **Log on** to NovaNano SEM Support PC (SPC) which runs the NPGS software (user: *supervisor*, password: *NanoSEM450*).

5. **Launch NPGS Menu.exe** from the Desktop:

The software will launch and a DOS terminal will open, which runs an automatic calibration routine for the NPGS board's DACs. Allow the calibration to finish before performing and commands via the software.

You will notice that the beam is automatically blanked during this procedure, as the SPC starts controlling the microscope.

6. **Create your user project folder:** *skip if folder already created*

You must create a project folder for your user account. The location of your project folder should be: **C:\NPGS\Projects*yourname***.

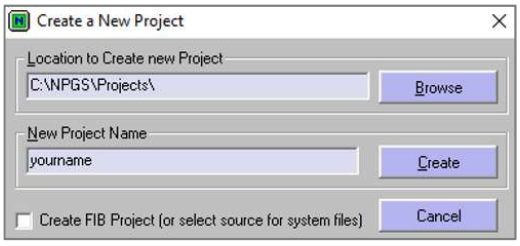
It is critical to copy ALL of the necessary system files from the directory: **C:\NPGS\Projects\Samples** to your project folder. These files contain important instructions for controlling the microscope via the support PC. The simplest way to perform this is by making a copy of the **…\Samples** folder and renaming it to …***yourname***.

- Navigate in Windows explorer to **C:\NPGS\Projects**
- Make a copy of the **Samples** folder
- Rename it to ***yourname***.

This action will also copy example CAD files (*.dc2) and run files (*.rf6), which can be used as references for your work.

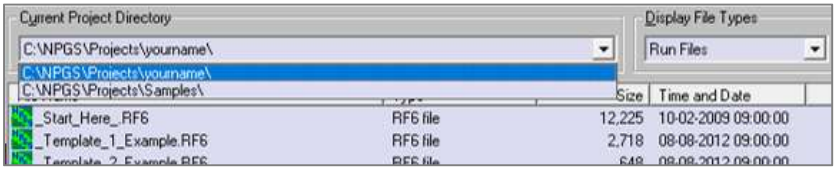
Your project directory may also be created from within the NPGS Menu:

- **Project > Create New Project**
- **Enter the name of your folder and click 'Create'**



Note: Creating a new project folder alone automatically loads the system files needed for operation. However, several fields within the files themselves are left blank and the writing will simply not work. Ensure that you copy all of the original files from the **…\Samples** folder by performing the following steps:

- Click File ►Copy File(s) to Current Project
- Select **C:\NPGS\Projects\Samples** and click **OK**
- In the top right corner, select 'All Files' from the drop-down menu 'Current File Types Listed'
- Click '**Select All**' on the lower left corner to highlight all files.

	• Click '**Copy Files**'. Unfortunately, you will be prompted to overwrite all conflicting files, so it's better to do this through Windows Explorer.
7.	**Select your project folder** from the drop-down menu at the top of the NPGS window: 
8.	**Position your sample** for writing using the stage Navigation features on the microscope PC (i.e. Nav-Cam navigation). Sometimes, the communication between the SPC and microscope (FEI's DCOM client) makes it impossible to switch between quads in the user interface. In this case, click "**NPGS Mode**" and then back to "**SEM Mode**" in the NPGS Menu to force normal SEM operation. Ensure that, when at the writing position, the surface of the e-beam resist will be in focus at a recommended working distance of 5 mm. It is important not to expose the area of interest by focusing at too high a magnification or by dwelling on the area for an excessive period of time. Once positioned as desired, zoom out to minimum magnification and 'pause' the scanning in the upper left quad. Notice that the specimen current goes to zero.
9.	**Place the SEM in External Control Mode** by clicking the **NPGS Mode** button on the left-hand side of the NPGS Menu window. Ensure that the quads on the microscope PC all say 'External' indicating external control by the NPGS software.

10.	**Locate your desired run file (*.rf6)** in your project folder through the NPGS Menu browser. Right-click and modify the file as needed. Be sure to update the beam current in any Pattern steps to the value measured in **Step 3.3**. Click **Save** and exit the run file editor. Optional: on the left side of the NPGS Menu, click '**Simulate Writing**' to check for potential errors with your run file. Y
11.	**Perform your exposure** by clicking **<Process Run File >** on the left-hand menu or by right clicking on your run file and selecting **Process Run File**.
12.	**Place the microscope in SEM mode** by simply closing the NPGS Menu software when finished. Alternatively, SEM Mode can be activated with NPGS left open by clicking **SEM Mode** on the left-hand menu. However, always close NPGS software when finished with writing.
13.	**Unload your sample** using **Steps 2.9-2.11.** **Pump the chamber** and log out using **Steps 2.12** and **2.13**.