Publications that made use of the **EPIC** facility instruments must include the following acknowledgements:

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Find this information online at:

http://www.nuance.northwestern.edu/epic/publication-acknowledgements/index.html
A. Policies and Introduction

Reservations

Quanta 650F ESEM reservations are made using the NUCore online reservation system. Start your reservation before you begin using the instrument. When your session is complete, be sure to end your reservation in NUCore. If you need extra time on the microscope, we recommend ‘extending’ your original reservation, rather than making an additional reservation.

There is a hardware control system on the Quanta, so the system will not function unless you are logged in to NUcore. Tampering with or disabling the hardware control may result in revocation of your EPIC privileges.

Saving Your Data

During your session, you may store image data to your own folder within the EPIC_SEM drive (S:\). EDS and EBSD data must be saved on the D:\ drive then transferred onto your folder on the S:\ drive. The SEM server is accessible through the computer down the hall. You can transfer your data from this SEM server computer to a USB, etc. You should NEVER take your data directly from the SEM computers.

SEM Rules

1. Please wear gloves when handling any components and samples that will go into the SEM.
2. Do not install any software onto the SEM’s PC.
3. Do not insert any flash drives into any microscope computer!
B. Start Up
1. Log in to the NUcore system and begin your reservation
2. Log into each computer if they are not already on
   a. Username: supervisor
   b. Password: Quanta9925466
3. If not already open, click the XT Microscope Server Icon on the SEM PC and hit Start.
4. Start UI (User Interface) once the server is running.
   a. NOTE: The username and password are the same as above.
5. Click OK when prompted to home the stage, or open the Stage menu and select Home Stage.

C. Interface Overview
1. There are four computer monitors for different applications on the microscope. The bottom right is the microscope PC.
2. The XT Microscope Control User Interface is made up of:
   a. The Menu Bar, containing dropdown menus.
   b. The Toolbar, containing icons for frequently used microscope functions.
   c. Six microscope control tabs.
   d. Four viewing quadrants.
**Toolbar** (Important tools in **bold**):

- 100X
- 15.0 kV
- 4.0
- **Beam selection**
- Magnification
- Acc. Voltage
- Spot Size
- **Lens Alignment**
- Direct Adjustments
- Reduced Area
- Link 2 to PWD
- Auto Focus
- Auto Contrast/Brightness
- VideoScope
- **Scan Speed**
- Make scan speed slow
- Imaging Residuum
- **Beam Blank**
- Acquire Photo/Snapshot
- Direct measurement
- Import System Parameters
- **Record Movie**
- Imaging filter mode

**Control tabs:**

- **Beam Control**
- **Navigation**
- **Processing**
- **Detectors**
- **Temperature Control**
- **Alignments**
D. Sample Loading

1. Once stage is homed, press Vent in the Beam tab to vent the chamber
   a. Chamber icon will turn grey when fully vented, do not force door open!
2. Always wear gloves if handling anything going into the SEM.
3. Select the appropriate sample holder for you experiment.
4. Slowly slide the chamber door open.
5. Mount sample/sample holder onto the stage.
   a. Do not place powders directly onto the stage!
6. Swing out the stage camera over the sample holder.
   a. The camera will take an initial image. After this image has been acquired, hold down the green button for a few seconds.
   b. **NOTE**: the camera will take 2 images before the process is complete.
   c. When the camera light goes out, swing it away from the sample.
7. In the Beam tab, select the desired vacuum mode
   a. High Vacuum Mode:
      i. For SE/BSE imaging of conductive/coated samples
   b. Low Vacuum Mode (up to 1 Torr):
      i. For LFD and BSE imaging of nonconductive samples
   c. ESEM Vacuum Mode (up to 30 Torr):
      i. Uses GSED detector for nonconductive/wet samples
      ii. **NOTE**: if using ESEM mode, please consult the ESEM Manual
8. Close the chamber door and press **Pump**.
   a. Low Vacuum users:
      i. See section H page 10 for more information.
9. Once the **Status** icon is green, the beam can be turned on.

E. **Setting Imaging Conditions**

1. Click on a quadrant to activate that window. The active window will be highlighted in blue.
2. Set the accelerating voltage level (200 V to 30 kV).
   a. High accelerating voltage: better resolution, loss of surface sensitivity, and increased charging effects.
      i. Low Vacuum/ESEM: high voltage will minimize beam skirting in the chamber gas.
   a. Small spot size will provide better resolution, but decreased beam current.
4. Select desired objective aperture using the dial located directly above the chamber door on the SEM.
   a. Small aperture will provide higher resolution and larger depth of field, but decreased beam current.
   b. Rotate the large knob to align with the aperture number you wish to use.
5. In the beam menu click on the **Beam On** button.
6. Click in the upper left-hand quadrant of the SEM computer screen until the border is blue.
7. Open the **Detector** tab.
   a. Select the **EHT** detector to detect Secondary Electron signal.
   b. Select **CBS** to insert the **BSE** detector (the working distance must be at least 10 mm).
      i. **NOTE:** the top right quadrant can be used to
display the BSE image while the top left displays the SE image.

8. Find and focus on the highest point on the surface of your sample using the focus knobs.
   a. You can also focus by dragging the right mouse button.

9. Couple the Z-axis of the stage to the working distance once focused.
   a. **NOTE:** You MUST be focused on the tallest point of your sample before linking your working distance to Z. Do not relink if you move to another area of your mount.

10. Raise the stage by selecting the stage navigation menu and typing in the desired Z-height.
    a. Start by entering 20 mm – refocus at 20 mm and re-link Z and WD.
    b. Enter 10 mm, refocus and re-link.

**F. Beam alignment**

1. Focus on a small feature on the surface of your sample with at least 15,000x magnification for alignment and focusing.
   a. Select the **reduced area** button to scan over a smaller area over your feature.

2. Open the **Direct Adjustments** menu from the toolbar (or select from the Scan Menu) and select the **BEAM** tab.

3. Optional: Adjust the **Source Tilt**.
   a. Click and drag the horizontal and vertical lines one at a time to achieve the brightest image.
      i. This effect will be more significant for larger spot sizes.
b. Click the **Crossover** button and center the source image (bright spot) on the green crosshairs.
   
i. If you cannot see the beam spot, increase the contrast.
   
ii. If you cannot see a bright spot, increase your spot size.
   
iii. Turn off **Crossover** by reselecting it.

4. **Important:** Click **Lens Modulator** to adjust the aperture
   
a. Fast scan speeds make it easier to see translation in your image.
   
b. Select **amplitude** around 0.30.
   
c. Click and drag the horizontal and vertical lines one at a time to reduce translation in your image
      
i. Drag horizontal line up/down to minimize up/down movement
      
ii. Drag vertical line left/right to minimize left/right movement.
   
d. Turn the **Modulator** off when finished by selecting it again.

5. **Optional:** Select the **Stigmator Centering tab** in **Direct Adjustments** window.
   
a. Click on **Modulator X** and minimize the image translation using the sliders in the alignment box.
   
b. Repeat with **Modulator Y**.
   
c. Deselect **Modulator X/Y** when finished.

6. Close out of **Direct Adjustments**.

7. **Important:** Correct for astigmatism using **X/Y Stigmator** knobs on control surface.
   
a. Select the **reduced area scan** and center over a small feature.
b. Focus the beam up and down and identify the two directions of stretching. Find the middle point between the two directions of stretching (where it is in best focus).

c. Adjust one stigmator knob one at a time until you achieve the clearest image. Adjust focus if necessary and repeat with other stigmator knob.

G. Image Capture

1. Center and focus on the area of interest.

2. There are two ways to save images:
   a. Select your own scan speed
      i. Slow down the scan using the scan speed section bar on the top of the screen.
      ii. Click the pause button
      iii. Go to File > Save as > EPIC_SEM (S:) > Your NetID > Save
      iv. Save file name as: Name_001 to start numbering your images.
   b. Preset Imaging conditions
      i. Go to Scan > Preferences > Scanning
      ii. Select speeds for screenshots ([]) or images ([]).
      iii. On the main user interface, press the image capture button on the top of the screen.
iv. Save in your NetID folder on the EPIC_SEM (S:) drive.

H. Low Vacuum Mode

1. **Note:** Low vacuum mode uses LFD (Large Field Detector) that is already inserted in the microscope.

2. On right hand menu, select **Low Vacuum Mode**
   a. Make sure **Water** is selected as the chamber gas.
      i. make sure distilled water bottle is ¼ full.
      ii. Contact EPIC staff if water seems low.

3. Indicate whether or not an accessory is being used.
   a. The answer for this is typically “no accessory” but pressure limiting aperture cones can be used to extend the pressure.

4. Select a chamber pressure up to approximately 1 Torr.
   a. A higher pressure will reduce charge artifacts but decrease image quality.

5. **Note:** slower scan speeds are needed for Low Vacuum Mode.

I. Backscattered Electron Detector

1. Ensure your sample is at least 10 mm away from the pole piece.

2. Under the detector menu, select **CBS detector**.

3. Select **Insert** to insert the detector at the end of the pole piece.

4. Select the rings of the detector to be used.
   a. Inner rings: used for high compositional contrast collecting high angle BSE.
   b. Outer rings: collect some compositional contrast and topographic information from low angle BSE.
c. **NOTE**: a combination of rings can be used to collect both compositional contrast and topographic information. The BSE detector can also help reduce charging artifacts by deselecting the inner rings.

**J. EDS with AZtec**

1. Pause the chamber camera window (bottom right on SEM monitor)
2. Set the **Accelerating Voltage** to an appropriate level.
   a. Usually 15–30 kV, but can be lower depending on your sample.
   b. Accelerating voltage should be ~2.1x the highest energy peak you expect.
3. Set the WD of the area to be analyzed to 10 mm (± 0.2 mm) away from the pole piece.
4. **Note**: Using a larger aperture and spot size may help to increase your signal.
5. See separate AZtec manual for further instructions

**K. Shut Down and Sample Removal**

1. Turn off the beam by hitting the yellow **Beam On** button.
2. Click **Vent** once the beam is off.
3. Wait for the chamber to vent completely.
4. Carefully slide open the chamber door and remove samples.
5. Put the system back to the default stage configuration:
   a. Multi-sample holder installed
   b. LFD detector installed
   c. High vacuum mode selected
   d. Backscatter (CBS) detector retracted
6. Close the door and select **Pump**.
7. Wait for the system to return to high vacuum.
a. Chamber icon at the bottom is green

8. End your reservation on NUcore.

L. Frequently Asked Questions

1. The monitor is black.
   a. Make sure you have begun your NUcore reservation.

2. The stage is not moving or responding.
   a. Open the Stage menu and select Home Stage.

3. Nothing is showing up on the screen.
   a. Make sure the image isn’t paused.
   b. Make sure the beam blanker isn’t selected. Unselect it by pressing the button.
   c. Make sure the correct detector is selected in the detectors tab.
   d. If all else fails, see question 6

4. The image is scanning and pausing. It is not live scanning.
   a. Make sure that Live is selected under the scan options tab

5. Backscatter detector Insert is greyed out and cannot be inserted.
   a. Check that your Z did not become unlinked
   b. Restart server: On xT microscope Server, click Stop UI, then Stop (right above.)
6. Viewing quadrants are frozen and say “External.”
   a. Another software has control of the microscope, likely Aztec or the Ebl software.
   b. Check the NPGS software on the top left computer monitor. Hit SEM mode button several times.