Quanta 650F ESEM
Operating Instructions
Publications that made use of the EPIC facility instruments must include the following acknowledgements:

“This work made use of the EPIC facility of Northwestern University’s NUANCE Center, which has received support from the SHyNE Resource (NSF ECCS-2025633), the IIN, and Northwestern’s MRSEC program (NSF DMR-1720139).”

Find this information online at http://www.nuance.northwestern.edu/epic/publication-acknowledgements/index.html
I. 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 SHARED DRIVE! Then data can be migrated to the Quanta folder on the EPIC_SEM drive (S:\\). The SEM server is accessible through a computer in the lab. EDS and EBSD data can be saved on the D:\ drive then transferred onto your folder on the S:\ drive. You can transfer your data from the SEM server computer to a USB, etc. You should NEVER take your data directly from either the SEM or EDS 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!
II. 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. NOTE: There are four computer monitors for different applications on the microscope. The bottom right is the microscope PC.
4. Open the XT Microscope Server Icon on the desktop of the SEM PC and hit Start
5. Start UI once the server is running
   a. NOTE: The username and password are the same as above
6. Click OK when prompted to home the stage
7. Once stage is homed, press Vent to vent the chamber
   a. Allow chamber to vent, do not force door open!

III. Sample Loading
1. Always wear gloves when handling anything going into the SEM
2. Slowly slide the chamber door open
3. Select the appropriate sample holder for your experiment
4. Insert SEM stubs into the sample holder
5. 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 sec
   b. When the camera light goes out, swing it away from the sample
   c. NOTE: the camera will take 2 images before the process is complete
6. In the Beam tab, select the desired vacuum mode
   a. High Vacuum Mode
      i. For SE and 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

7. Close the chamber door and press Pump
   a. Low Vacuum users
      i. Make sure distilled water bottle is ¼ full
         1. Contact EPIC staff if water seems low
      ii. Select any accessories used, if any, when prompted

8. Once the Vacuum Status is green, the chamber has been pumped and the beam can be turned on.

IV. Operation and Alignment

1. There are 4 quadrants on the microscope UI. Click on the 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

3. Set the Spot Size (1 – 7)
   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. Rotate the large knob to align with the number aperture 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 on the tallest point on your sample to link your working distance and Z. **After you have linked, do not link again!**

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. Move up to 10 mm on tallest sample, refocus and re-link

11. Increase the magnification to at least 15,000x for alignment and focusing.

12. Focus on a small feature on the surface of your sample

13. Open the **Direct Adjustments** menu from the toolbar at the top of the control screen and select the **BEAM** tab

14. Click on **Lens Modulator** to finely adjust the aperture
   a. Select the **reduced area** button
   b. Increase scan speed to see translation in your image
   c. Select **amplitude** around 0.30
   d. Reduce translation in your image by clicking and dragging the x and y lines in the alignment box left/right (to minimize lateral movement) and up/down (to reduce vertical movement)

15. Click **Crossover** button and center the source image (bright spot) on the green crosshairs
a. If you cannot see the beam spot, increase the contrast  
b. If you cannot see a bright spot, increase your spot size

16. Unselect Crossover when finished with beam alignment

17. Turn the Modulator off and adjust the Source Tilt to achieve the brightest image
  a. This effect will be more significant for larger spot sizes

18. Select the Stigmator Centering tab in Direct Adjustments window
  a. Use Stig X and Stig Y modulators to minimize the image translation using slider windows

19. Close out of Direct Adjustments

20. Select the reduced area scan and center over a small feature

21. Focus and correct for astigmatism using X/Y Stigmator knobs on control surface

a. Focus the beam up and down with your fine focus and identify the two directions of stretching. Find the middle of the stretching and adjust one stigmator knob at a time until you achieve the clearest image.
V. Operation and Alignment

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. Drag and drop the image icon (the camera) to the scan speed you want to use for all images
         a. Select speeds for screenshots ( ) and 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
3. NOTE: When using Low Vacuum mode, slower scan speeds provide the best imaging

VI. Low Vacuum Mode

4. Low vacuum mode uses LFD (Large Field Detector) that is already installed in microscope
5. On right hand menu, select Low Vacuum Mode
   a) Make sure Water is selected as the chamber gas
6. 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
7. Select a chamber pressure up to approximately 1 Torr
   a) A higher pressure will reduce charge artifacts but decrease image quality
8. Use a slow scan speed while operating
VII. Backscattered Electron Detector

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

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

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

12. 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 come compositional contrast and topographic information from low angle BSE

13. **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

VIII. EDS with AZtec

14. Pause the chamber camera window (bottom right on SEM monitor)

15. Set the **Accelerating Voltage** to an appropriate level
   
   a) Usually 15-30 kV, but can be lower depending on your sample
   b) Typically about 2.1x the highest energy peak for the heaviest elements in the sample

16. Set the WD of the area to be analyzed at 10 mm (± 0.2 mm) away from the pole piece

17. Using a larger aperture and spot size may help to increase your signal

18. See AZtec manual for further instruction
IX. Shut Down and Sample Removal

19. Turn off the beam by hitting the yellow **Beam On** button

20. Click **Vent** once the beam is off

21. Wait for the chamber to vent completely

22. Carefully slide open the chamber door and remove samples

23. Put the system back to the default stage configuration:
   - a) Multi-sample holder installed
   - b) LFD detector installed
   - c) **High vacuum mode selected**

24. Close the door and select **Pump**

25. Wait for the system to return to high vacuum
   - a) Light at the bottom is green

26. End your reservation on NUcore
X. Frequently Asked Questions

27. The monitor is black
   a) Begin NUcore reservation

28. The stage is not moving or responding
   a) Solution: open the Stage menu and select Home Stage

29. Nothing is showing up on the screen
   a) Make sure the beam blanker isn’t selected. Unselect it by pressing the button
   b) Make sure the correct detector is selected in the detectors tab

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