

FEI Quanta 600F sFEG ESEM Operation Instructions

For additional assistance, please contact the facility manager.

Please contact in an emergency:

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Quanta reservations are made using the EPIC FOM online reservation system. Please follow all EPIC facility rules for using this system.

Note: It is imperative that gloves be worn during all sample exchange procedures. If you cannot find any gloves, please ask!

You are asked to make a copy of your data on your own disk IMMEDIATELY after your session is finished. You may save your data on a CDR/CDRW, USB flash drive or transfer your data by FTP. The data may be deleted at any time without notice. EPIC is not responsible for any data loss.

System Startup and Sample Loading

1. Log in to FOM and log in to your reservation.
2. Log in to each of the three computers (from left to right, NPGS PC, Support PC and SEM Control PC) – Username: supervisor, Password: quantad7768
3. Double-click on the **XT Microscope Server** icon on the desktop and hit the **Start** button.
4. Once the server is running, click the **Start UI** button and enter the same username/password.
5. Click **OK** at the prompt to home the stage.
6. Once the stage is homed, press the **Vent** button to vent the chamber.
7. Allow the chamber to vent on its own (i.e. do not force the door open) – this should take about 5 minutes and the **Vacuum Status** light should be **red**.
8. Slowly slide the chamber door open.
9. Select the appropriate sample holder - the multi-stub sample holder should be installed by default. This can be swapped for the high resolution (single stub) holder or the Peltier stage (see cold stage section). The multi-stub holder has two parts (pedestal and platform), which can be removed by removing the allen screw in the center of the platform first and then repeating for the pedestal. If you replace multi-stub holder with another sample holder, please reinstall at the end of your session.
10. Insert your SEM stubs into the sample holder (the multi-stub holder has spring clips, the high-res holder has an allen screw to secure the stub).
11. Select the desired vacuum mode:

- a) High Vacuum Mode – For conductive/coated samples. E-T SE and BSE detectors are available.
 - b) Low Vacuum Mode (up to 200Pa) – For nonconductive samples. LFSE and BSE detectors are available.
 - c) ESEM Vacuum Mode (up to 4000Pa) – For nonconductive/wet samples. GSED or GAD detector must be used – these have a built-in PLA for high pressure use.
12. Installing detectors:
- a) E-T SE: None required, always installed.
 - b) BSE/GAD: The backscattered electron detector and gaseous analytical detector should be stored in the ‘parking lot’ just to the right of the objective lens with the wiring hooked on the side. To install, remove from the parking lot and clip the detector onto the end of the objective lens such that the wiring faces the front of the chamber and the notch is at the rear-left. Look in the camera to insure the detector is mounted flush and not tilted. The wires from the detector should be secured on the hook on the right side of the parking lot to make sure they do not interfere with the stage movement. The BSE/GAD detectors are the same, with the exception of the PLA built in to the GAD. They use the same preamp circuit and you must switch the BSE/GAD switch on the right side of the chamber to select the desired detector.
 - c) LFSE: The large-field SE detector is used for low vacuum operation and the circuit board clips into the slot at the rear of the chamber. This detector should be installed by default, but can also be used with either of the PLA cones to extend the pressure range.
 - d) GSED: The gaseous secondary electron detector is used for the highest pressure operation and clips into the same slot as the LFSE detector. The other end of the detector then clips onto the objective lens and should be flush mounted like the BSE/GAD detectors.
12. If you are using Low Vacuum or ESEM modes, check to make sure there is sufficient distilled water in the beaker in the rear compartment.
13. Close the chamber door and press the **Pump** button – if you have selected Low vacuum or ESEM mode, you will be asked to select the accessory installed (PLA, GAD, etc.). You can switch between High, Low and ESEM vacuum modes at any time during operation without venting the chamber. Occasionally, when switching vacuum modes or venting there is a vacuum hardware error. If this happens, hit clear and hide, then click on the desired vacuum mode and the system should resume its operation.

General Operation and Alignment

1. Wait for the **Vacuum Status** light to turn **green**.
2. Set the High Voltage (HV) level – variable from 200V to 30kV. In general, a higher voltage will provide better resolution at the expense of some loss in surface sensitivity and increased charging effects. For low vacuum/ESEM operation, a high voltage will also greatly minimize the amount of beam skirting in the chamber gas.

3. Set the **Spot Size** (1 to 7). A smaller spot size allows for better resolution, but decreases beam current.
4. Select the desired **objective aperture** – 1 (largest) to 7 (smallest). A smaller aperture allows for better resolution and higher depth of focus, but lower beam current.
5. Press the **HV** button to turn on the beam.
6. Each quad can display a different detector or mixed detector signal. Select a quad by left-clicking and then choose the desired detector from the Detector menu.
7. Adjust the brightness and contrast – you should be able to make out the center of the sample holder.
8. Use the **Stage X/Y** control to locate your sample. This can be done manually with the manipulators on the chamber door or through the software by middle-clicking in an image quad and dragging the mouse in the desired direction. The further you move the mouse, the faster the stage moves. The sample orientation on the screen is oriented with respect to the E-T detector (i.e. rotated 90° CCW from the actual orientation).
9. Focus on the highest point on the surface of your sample using either the control surface or by clicking the right mouse button and moving left and right.
10. Couple the Z-axis of the stage to the working distance by pressing the **Couple Z to FWD** button (Shift + F9).
11. Raise the stage by middle-clicking in the camera quad and moving the mouse up until the desired working distance is achieved (the system should automatically focus as you raise the stage).
12. Find a small feature on the sample surface to use for alignment and focus on the feature at high magnification (>15,000X). Magnification can be adjusted by the wheel on the control surface or by the +/- keys on the keyboard.
13. From the Startup Page (first tab in upper right corner), zero the Tilt and Lens Alignments under Source Control by right-clicking and selecting zero.
14. Turn the **Modulator on** and adjust the **X/Y knobs** on the objective aperture set to minimize translation of the image (coarse aperture alignment).
15. Fine adjust the aperture alignment by adjusting the **Lens Alignment** to completely stop image translation. Left-click in the 2D Lens Alignment box and move the mouse up/down (to minimize lateral movement) and left/right (to minimize vertical movement).
16. Turn the **Modulator off** and adjust the beam **Tilt** to achieve the brightest image (this effect will be more significant for larger spot sizes). Alternately, the beam Tilt can be adjusted by pressing the Cross Over button and centering the source image about the green cross hairs.
17. Repeat steps 13 to 15 if necessary.
18. Correct for astigmatism by either using the **X/Y Stigmator** knobs on the control surface, left-clicking in the 2D Stigmator box or holding down the shift button and the right mouse button simultaneously. It is easiest to correct for astigmatism by switching between X , Y and focus until the sharpest image is obtained.

Digital Imaging with XT Docu

1. XT Docu is the default image capture and manipulation software used with the Quanta. An image database and folder in the Shared Data directory will be set up during your training session. All images should be stored on the SEM support PC – the Shared Data folder is mapped to the SEM control PC.
2. Once the XT Microscope Server is running, the XT Docu program can be loaded by double-clicking and selecting your database.
3. There is a floating tool bar associated to allow easy transition between the SEM UI and XT Docu. Clicking on the light bulb in the tool bar switches between the applications.
4. To capture an image, select the desired image quad and click the snapshot button (F2). The snapshot scan speed preset can be changed by selecting preferences under the Scan menu (Ctrl + O).
5. When the image is frozen (Pause icon shown), click the ‘camera’ button in the XT Docu toolbar, followed by the ‘file cabinet’ button.
6. Enter the desired file name and click on ‘Insert’ to transfer the image to the database.
7. Double-click on an image in the database to open it up in the full-screen view.
8. The data bar can be displayed, removed or customized under the Database menu.
9. If you just want a scale bar and nothing else, this can be accomplished under the Image menu.
10. Images stored in the database must be exported to a .tif file to view in other software. From the full-screen view, right click at the top of the image and select Save As. Multiple images can be exported through the export option under the Database menu.

Digital Imaging and EDS with Inca

1. Make sure the switch on the blue box on the right side of the desk is set to SEM.
2. Open the INCA software by double-clicking on the desktop icon.
3. There are three main subsets to the program:
 - a) Analyzer – allows for EDS acquisition but no imaging/scan control
 - b) Point & ID – the main image acquisition program, which also allows for site specific EDS analysis
 - c) Mapping – allows for EDS maps and linescans using a data mining approach (i.e. a full spectrum is stored from each pixel)
4. Within each project file you may have multiple samples. For each sample, you may have multiple sites of interest – each site of interest is an image with various associated spectra.
5. Basic instructions are presented below, but more detailed information can be readily obtained from the bubble help (upper right corner of the screen).
6. Image capture:
 - a) Set the SEM to a slow scan rate (i.e. Scan Speed 3 or 4)
 - b) Click on the Image Setup menu
 - c) Select the desired image resolution, scan speed and data (8 bit is generally sufficient).

- d) Go back to the Site of Interest menu and click on the green button to acquire an image
7. EDS acquisition:
 - a) Click on Acquisition Setup
 - b) Set the collection time (Live Time)
 - c) Set the pulse processor (Process Time) to the desired setting. A lower process time allows for faster collection at the expense of energy resolution (i.e. wider peaks).
 - d) Set the desired energy range and eV per channel.
 - e) Click on X-ray Acquisition.
 - f) Select region(s) of the sample to analyze. In Point & ID this is done by using the point, box, etc. tools, but in Analyzer mode there is no scan control so the system collects X-rays from wherever the SEM is directing the beam.
 - g) Click on Confirm Elements to verify the peaks.
8. EDS mapping:
 - a) From the mapping program, collect an image as per the image capture instructions above.
 - b) Select a region to map or draw a line to scan.
 - c) Click on the green button to begin acquisition.
 - d) Click on Element Setup to select the elements to display.
9. To save any images, maps or EDS spectra, right click and select Export.

Shut Down and Sample Removal

1. Shut of the High Voltage by pressing the **HV** button.
2. Lower the sample stage to a working distance of 20mm or greater.
3. Select **Center Position** under the Stage menu (Ctrl + 0).
4. Press the **Vent** button to vent the chamber.
5. Wait for the Vacuum Status light to turn **red**.
6. Open the chamber door and remove your sample(s).
7. Put the system back to the default configuration:
 - a) Multi-sample holder installed.
 - b) LFSE detector installed
 - c) BSE/GAD detectors in the parking lot and cables secured.
 - d) High vacuum mode selected
8. Close the door and hit the **Pump** button.
9. Wait for the system to return to high vacuum – Vacuum Status light is **green**.
10. Close the SEM UI.
11. Press **Stop** in the XT Microscope Server window.
12. When the system is stopped, right click at the top of the window and select **Exit XT Microscope Server**.
13. Close all software and log off from all three PC's.
14. Clean up the SEM room as a courtesy to the next user.
15. Log out from FOM.