Standard operating procedures for FEI Quanta FEG450

1. Specimen Preparation

The Quanta 450FEG has three operating modes: High Vacuum (HiVac), Low Vacuum (LowVac), and Environmental (ESEM). HiVac mode typically requires the specimens to be conductive or coated. LowVac mode can be used to observe outgassing or highly charging specimens without conductive coatings. ESEM mode is primarily used for wet specimens or specimens containing volatile components.

Specimens should be clean, fixed, and properly mounted before loading them onto the specimen stage. The specimen must be electrically grounded to the sample holder with conductive tape or paint to minimize specimen charging.

2. Specimen loading *(Users must wear powder free gloves when handling detectors or loading specimens*.)

(1) Fill out the Logsheet in the binder on the SEM computer table. Fill out the Date, User, Project Number, Start Time and Start-Up Status on the Logsheet. Refer to the previous entry on the Logsheet to confirm the SEM status.

(2) The column and specimen chamber are kept under high vacuum when not in use to prevent contamination so first the system must be brought to atmospheric pressure to open the specimen chamber. The system can be vented via the computer interface by clicking the "**Vent**" button on the "**Vacuum**" console.

(3) As the system vents, the **Vacuum Status Indicator** on the "**Status**" console will change from green, to amber while venting. After venting, the chamber door can be opened by pulling the door straight out.

(4) After mounting your specimen to a specimen stub, insert the pin on the underside of the specimen mount into the opening in the top of the stage. A small setscrew on the side of the stage should be gently tightened using a small hex wrench.

(5) The "ESEM" mode and backscattered imaging require installation of the **Gaseous Secondary Electron Detector (GSED)** or the **Back Scatter Detector**. Please contact staff for changing/installing the detectors, or installing the **Hot/Cold** or **Microtensile** stages.

(7) Close the chamber door, chose the desired vacuum mode (HiVac, LowVac, or ESEM), and click the "**Pump**" button on the **Vacuum** console in the computer interface.

(8) Once the system has reached the working pressure, a green light will appear next to the vacuum pressure on the "**Status**" console. Click the "**Beam On"** to turn on the electron beam.

3. Adjusting Working Distance, Accelerating Voltage and Spot Size

(1) Set the highest point on the specimen to a **working distance** of approximately 10 mm by adjusting the z-axis on the stage. The z-axis can be changed by clicking on the camera view window or in quad 4, then clicking and holding the middle scroll button up or down over the yellow bar which will appear on the screen.

(2) The **accelerating voltage** can be set between **0-30 kV** via the "**Electron column**" console however, **10 kV** will be adequate for most materials. For polymer and glass samples **2-6kV**

works well and for metals or highly conductive surfaces **10-20kV** will provide high resolution.

(3) To obtain an image, click on the desired quadrant, 1, 2 or 3, click the "unfreeze" button, and then click the rabbit icon on the toolbar for fastest render.
(4) Demagnify as far out as possible when setting up an image by pressing the "-" key on the number pad on the far right hand side of the keyboard.
(5) Adjust the **magnification**, **focus**, **stigmator**, **contrast** and **brightness** to desired levels as described below.
(6) Adjust the **contrast** and **brightness**, located on the "**Detectors**" console, to the desired levels, or press **F9** for the auto contrast brightness (ACB) function.
(7) Once the **brightness** and **contrast** have been adjusted, increase the **magnification** using the "**+**" key on the keyboard. Other magnification adjustments are
   - Higher/Lower **=** (+/- on num pad)
   - Coarse control = (Crtl key + mouse wheel up/down)
   - Fine control = (Shift key + mouse wheel up/down)
   - Round value **= (*** on num pad)
   - Select preset value from **Magnification** menu on the **Option** page
(8) **Focus** the image by holding the right mouse button and moving the mouse left or right.
(9) The "**Spot size**" on the "**Electron column**" console should be adjusted to improve the image quality; however, in turn the **brightness** and **contrast** will need to be readjusted. In general smaller spot sizes are used for high magnification/resolution while larger spot sizes are more suitable for low magnification and X-ray analysis.
(10) An area of interest can be moved to the center by locating the mouse pointer over it and double-clicking. To move the sample, the arrow keys, the center scroll button, the stage console, or the x, y and z knobs on the SEM chamber door can also be used.
(11) To optimize very high magnification imaging, the **stigmator** can be adjusted by holding the "**Shift**" key and the right mouse button simultaneously.

4. Image Capture

(1) To capture an image click on the desired quadrant, 1, 2 or 3.
(2) By clicking the "**-**" button in between the "**turtle**" and "**rabbit**" buttons on the toolbar the render speed can be decreased, thereby increasing the image quality shown on the screen and allowing for a better idea of how the final image will actually look. It is good technique to adjust the **brightness** and **contrast** a few times while switching back and forth between fast and slow raster speeds until the extremely bright regions are minimized and muddy looking regions show detailed contrast.
(3) To have good resolution in your saved image, the image size, located to the right of the rabbit button, should be 1024x884. This results in an image of around 900kB.
(4) Now click the "**camera**" button on the toolbar to initiate a slow scan and capture a final image.
(5) Once the image has been fully rendered, save the image into the user folder.

5. Shut-down Procedure

(1) Reduce the magnification to its lowest value and turn off the accelerating voltage by clicking the "**Beam On**" button on the "**Column**" console.

(2) Once the accelerating voltage has been turned off, click the "**Vent**" button on the "**Vacuum**" console to bring the system back to ambient pressure.
(3) The specimen chamber can now be opened and the sample can be removed. Use the 1.5mm hex key to loosen the set screw on the stage and remove the specimen stub from the socket. Remember user must wear latex gloves when unloading specimens or handling detectors.
(4) If the **Back Scatter Detector** or the **Gaseous Secondary Electron Detector (GSED), or hot/cold/microtensile stage** is used, contact the staff for removal of the detectors or stages.
(5) Now that the sample has been unloaded shut the specimen chamber door. The system should be kept under high vacuum when not in use, so click the "**Pump**" button on the "**Vacuum**" console.
(6) Wait until the vacuum indicator on the "**Vacuum**" console has turned green, a few minutes after pump down has commenced, and record the pressure on the logsheet.
(7) Remember to shut off the lights and close the door to the lab securely behind you when you leave.

References:

http://www.sjsu.edu/people/anastasia.micheals/courses/MatE143/s1/SOP_QUANTA.pdf