Standard Operating Procedure
FEI Quanta 200 Scanning Electron Microscope

This document is intended as a guide to the operation of the FEI Quanta 200ESEM by certified users. It provides details on sample loading, bringing the microscope up to operating conditions, normal operation procedures, sample removal, and returning the microscope to a powered down state. This guide is not intended for novice users.



Please fill out the logsheet before beginning using the microscope.

1. Specimen Preparation and Handling

The Quanta 200 has three operating vacuum modes to deal with different types of samples**. High Vacuum (HiVac)** is the conventional operating mode associated with all scanning electron microscopes. High Vacuum mode typically requires that the sample be conductive or properly mounted and coated for conduction. Coating reduces beam penetration and allows for a sharper image; however, it may mask elements of interest for X-ray analysis. The two other operating modes are **Low Vacuum (LowVac)** and **ESEM**. In these modes the column is under high vacuum and the specimen chamber is at higher pressures of 0.1 to 30 Torr. Observation of outgassing or highly charging materials can be made using one of these modes without the need to coat the sample with a conductive material. In some cases a **PLA (Pressure Limiting Aperture)** cone placed on the conical objective maybe useful in **LowVac** mode because the gas in the chamber may cause "skirting" of the incident beam. **ESEM** mode is used primarily for any specimens that are considered "wet" and contain volatile components such as water or oil.

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. Computer User Interface (UI)

The SEM is controlled by means of a Windows-based user interface (UI) program, called xT Microscope Control. This program should normally be left running.

The primary SEM controls are accessed via the consoles on the right hand side of the screen. There are various tabs at the upper right hand side corresponding to different "pages". The **Work Page** (Figure 1) contains the "**Vacuum"** console, the "**Electron Column"** console, the "**Detectors"** console, and the "**Stage"** console, with **maps**, **coordinates**, and **tilt correction** tabs, and finally, the "**Status"** console. The **Options Page** (Figure 2) contains the "**Imaging"** console. The various pages are accessed by selecting the desired tab from the top of the console.

Images are displayed in the main window on the computer screen. The main window is divided in to 4 quadrants (quads). The bottom right quadrant, quad 4, shows an infrared camera view of the inside of the SEM chamber. The upper right quadrant, quad 2, is typically used to display the secondary electron image. Quadrant 1 (upper left) may be used for the backscatter detector, and quadrant 3 (lower left) may be used to mix images from quad 1 and 2.

The SEM can be controlled by making selections from the **Menu**, clicking icons on the **Toolbar** (Figure 3), or with keyboard commands (Figure 4). The mouse is also used to control various functions on the SEM. The controls are discussed in detail in the following sections.

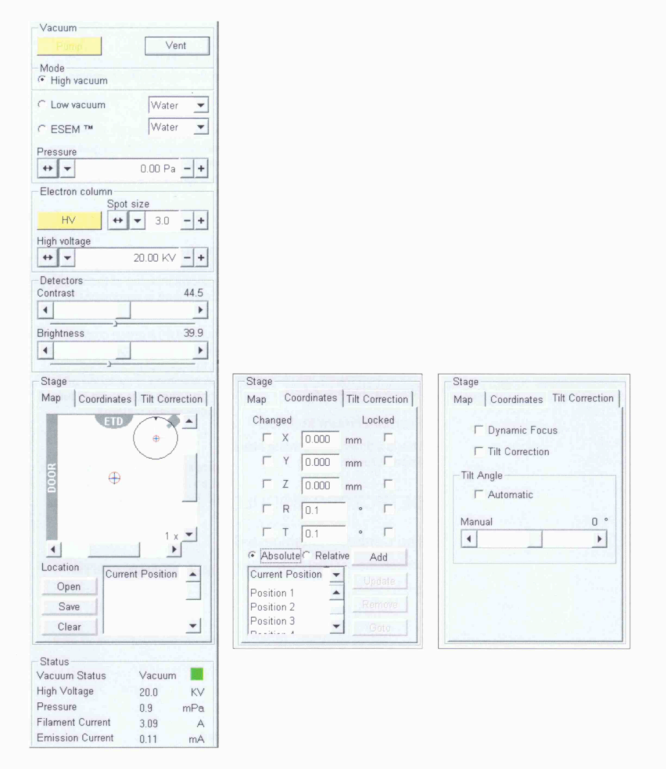


Figure 1. The **Work Page,** located on the right side of the xT Microscope Control window.

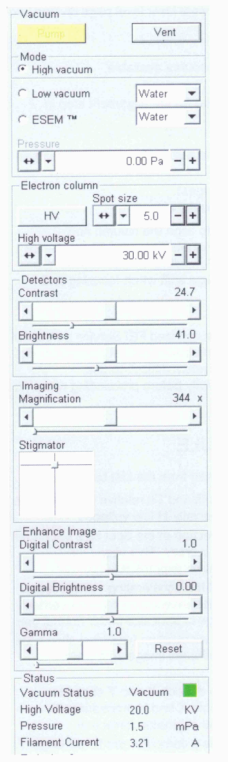


Figure 2. The **Options Page**, located on the right side of the xT Microscope Control window.

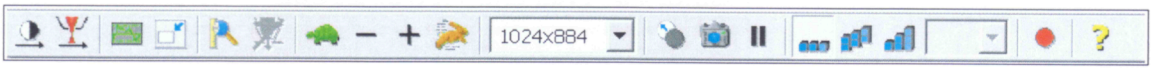


Figure 3. The **Toolbar**, located on the top of the xT Microscope Control window.

Key (+ Key)	Function
F1	On-Line Help (only switches ON)
F2	Snapshot
F3	Toggle Videoscope
Shift + F3	Home Stage
F4	Lens Alignment
F5	Toggle Quad Screen / Full Screen
Shift + F5	Toggle Center Cross
F6	Toggle Pause / UnPause
Shift + F6	Toggle Alignment rectangle
F7	Toggle Reduced area On / Off
F8	Degauss
F9	Auto Contrast and Brightness
F11	Auto Focus
Shift + F11	Auto Stigmator
F12	Toggle Compucentric Rotation.
Shift + F12	Toggle Scan Rotation
Ctrl + 0 - number	Centers X and Y stage axes to 0,0
Ctrl + F	Sets FWD to 10 mm
Ctrl + O - letter	Preferences dialogue
Ctrl + P	Prints to selected device
Ctrl + S	Save
Ctrl + Tab	Steps between Quads
+	Increases magnification
-	Decreases magnification
*	Rounds off magnification to nearest rounded number.

Figure 4. Keyboard shortcuts.

3. Start up Procedure

Users must wear latex 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. From the xT microscope control user interface (UI), enter your Username and Password. The general username is "**user**", and the password is "**user**".

3. The system will be in "**External**" mode. Under the "**Scan**" menu, select "**Mains Lock**". This will return control to the UI.

4. The column and specimen chamber are kept under 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. The program will display a prompt asking "Really vent the chamber?". Click yes to vent the chamber.

5. As the system vents, the **Vacuum Status Indicator** on the "**Status**" console will change from green, to yellow while venting, and finally to red when vented. At this point, the chamber door can be opened by pulling the door straight out.

6. If you are operating in **LowVac** or **ESEM** mode, e.g. if you have a non-conductive or wet specimen, see Appendix A for instructions on installing the appropriate detector and apertures.

7. 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 (see Figure 5). A small setscrew on the side of the stage should be gently tightened using a small hex wrench (see Figure 6).

8. Close the chamber door and click the "**Pump**" button on the **Vacuum** console in the computer interface.

9. Once the system has reached the working pressure, a green light will appear next to the vacuum pressure on the "**Status**" console. Check that the pressure is $5x10^{-5}$ torr or below. At this point the **accelerating voltage** may be turned on by clicking on the "**HV**" button on the "**Electron Column**" console.

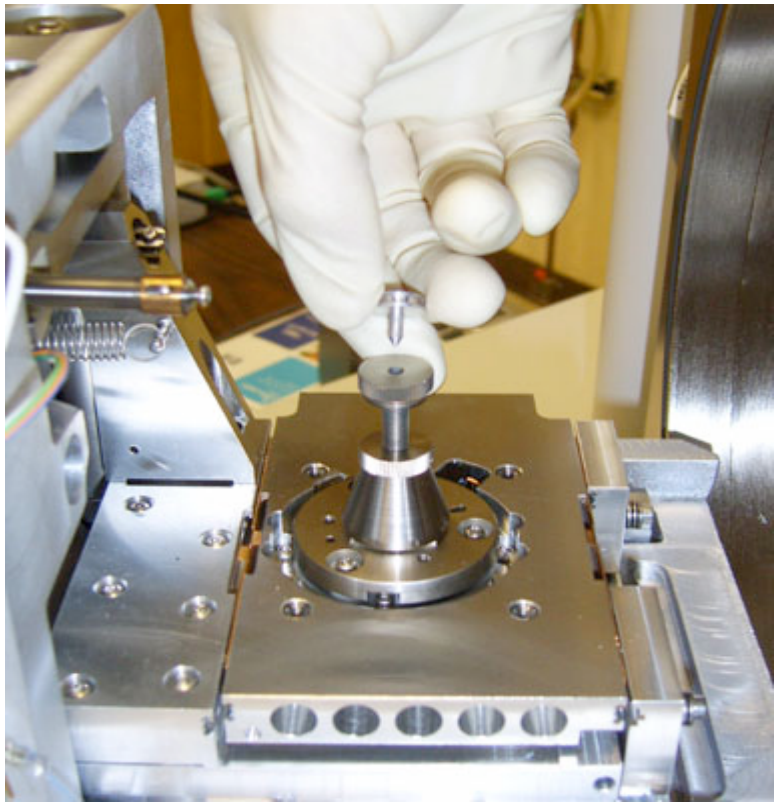


Figure 5. Inserting the specimen stub into the stage.

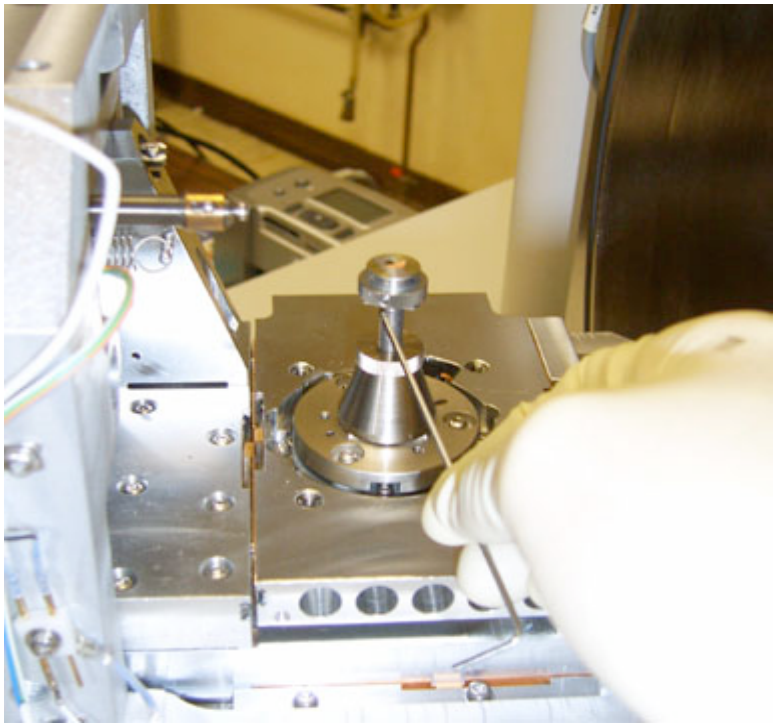


Figure 6. Tighten the setscrew on the specimen stage lightly, just enough to secure the stub.

4. 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, 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 (see Figure 7 for stage orientation and axes).

11. To optimize very high magnification imaging, the **stigmator** can be adjusted by holding the "**Shift**" key and the right mouse button simultaneously.

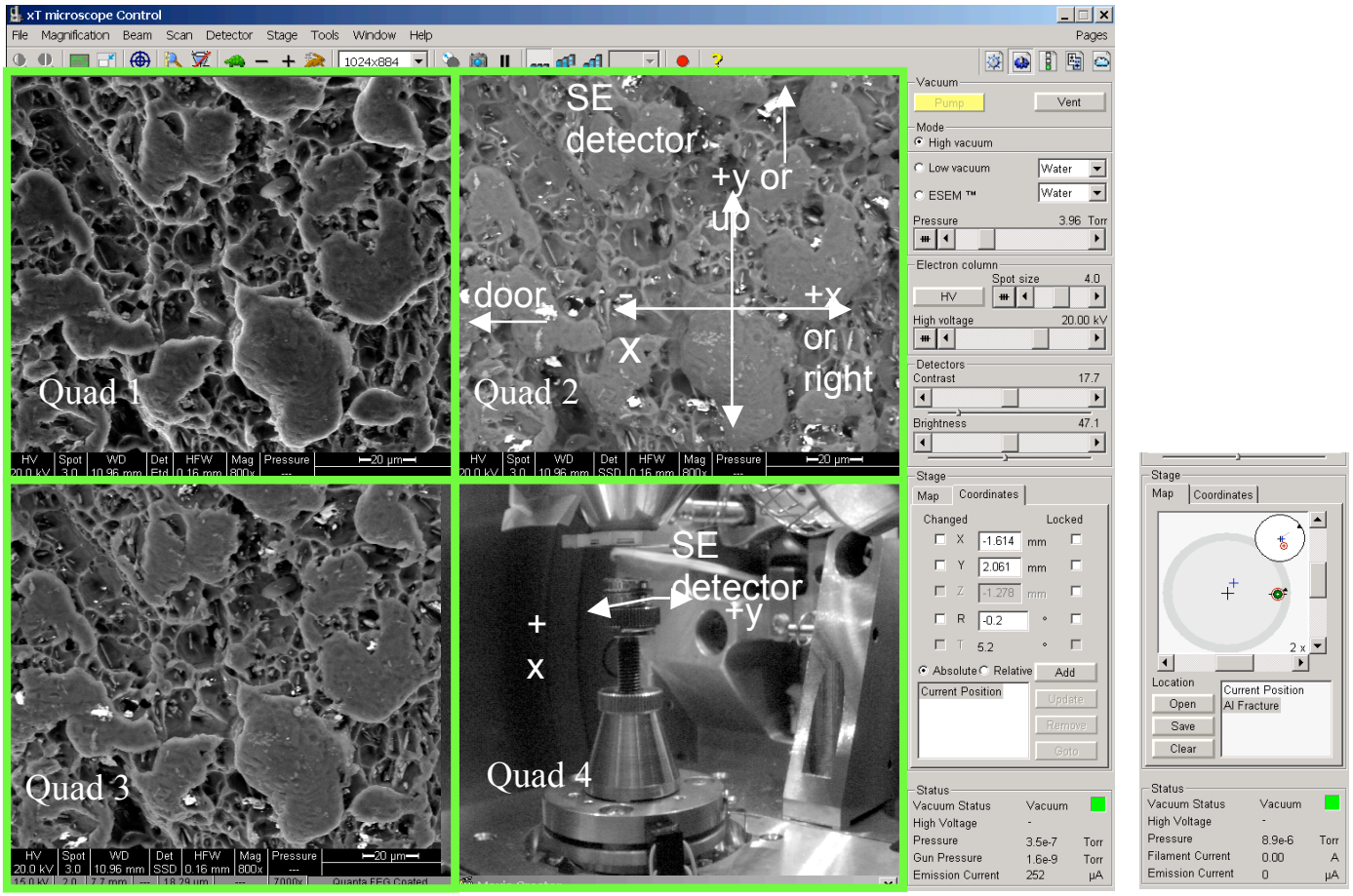


Figure 7. Stage Orientation and Axes.

5. 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, click the **filing cabinet** icon in the small toolbar at the top right of the screen to save into **xTDocu** database program.

6. There is a photo printer attached to the computer network so images may be printed directly by opening the image if not already shown and by clicking **Print** (crtl+p) under the **File** menu.

6. Transferring files

1. Click the **light bulb** icon in the small toolbar at the top right of the screen to toggle open the **xTDocu** database program window.

2. Once in the XTDocu window, select the images to be exported.

3. Select **Database** from the menu toolbar, then select "**Export Images**"

4. Images are exported to the support computer. Select the desired path, and click "**OK**". The images will be exported to the selected location.

5. If you do not have a folder, create one labeled with your project number and name. The default location is the "**Shared Data**" folder.

6. Access the support computer by clicking the "**1**" button on the KVM (keyboard-video-monitor) switch. Do not move the mouse, as this will prevent the switch from changing the input.

7. Locate your images. There is a shortcut on the desktop to the default location, the "**Shared Data**" folder.

8. Transfer your images to a USB drive or other removable device.

9. Switch back over to the SEM computer by pressing "**2**" on the KVM switch.

10. Toggle back to the SEM UI by clicking the **light bulb**.

7. Using the Back Scatter Detector (BSD)

1. The backscatter detector, shown in Figure 8, is used to image materials with high elemental contrast. The BSD is very delicate and expensive. Do not install the BSD unless you have been instructed and specifically authorized to do so.

2. Put on clean latex gloves. Carefully remove the BSD from its dock near the front of the SEM chamber, making sure not to touch the detector surface.

3. Again without touching the detector surface, install the BSD onto the end of the column inside the chamber. Hold the detector so that the torlon O-ring side is up, and the cutout is aligned with the EDS detector. Press straight up to seat the detector. Do not twist.

4. Once the BSD is in place, inset your sample, and pump down the chamber as usual.

5. To obtain an image, select quad 1. Activate the BSD by clicking on the "**Detector**" menu, and selecting "**BSD**".

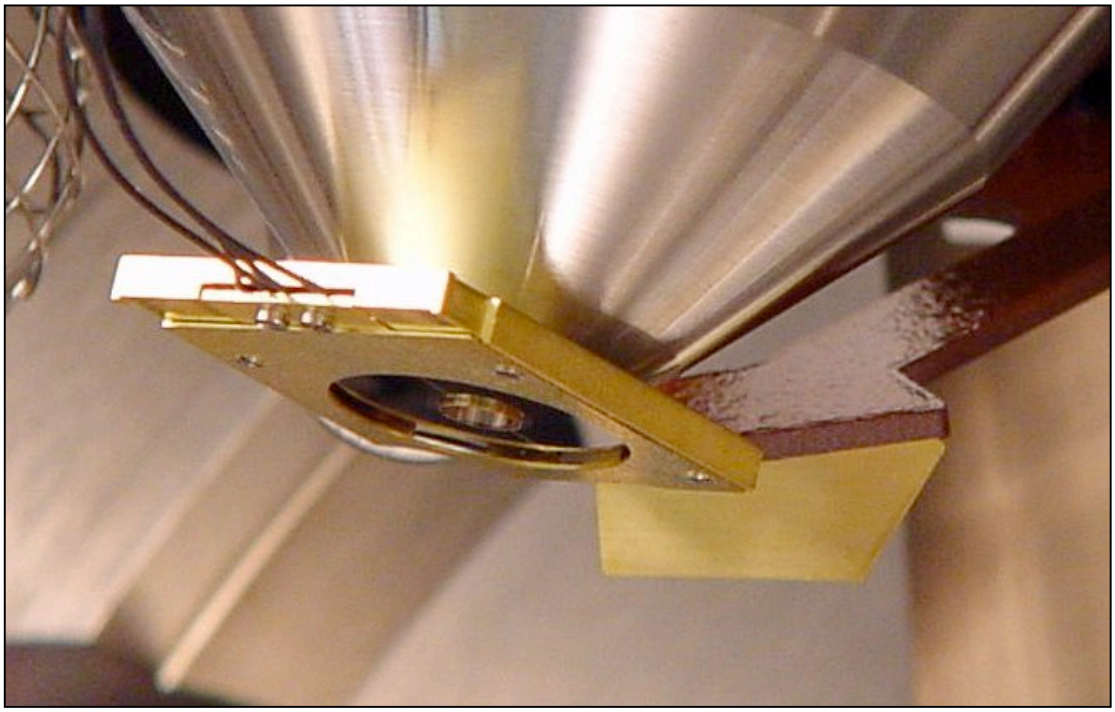


Figure 8. The Backscatter Detector

8. Shut-down Procedure

1. Reduce the magnification to its lowest value and turn off the accelerating voltage by clicking the "**HV**" 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. Once the system is fully vented, the "**Vacuum Status**" on the "**Vacuum**" console will display a red box.

4. The specimen chamber can now be opened and the sample can be removed. Use the 1.5mm hex key to loose 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.

5. If the **Back Scatter Detector**, **Gaseous Secondary Electron Detector (GSED)**, or any **PLAs** have been installed, they should be removed and placed back into package for storage.

6. Now that the sample has been unloaded shut the specimen chamber door. The system is kept under high vacuum when not in use, so click the "**Pump**" button on the "**Vacuum**" console. If the system is in **LowVac** or **ESEM** mode, the **environmental backing valve (EBV)** should be shut when prompted by the computer.

7. 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.

8. Under **File**, log off from the system. Shut off the computer monitor.

9. Remember to shut off the lights and close the door to the lab securely behind you when you leave.

9. Troubleshooting

9.1 Shut Down Procedure for the SEM

If the program becomes unstable (for example the xT Microscope Control program window is not updating, or the program crashes), you may need to shut down and restart the computer and/or the SEM. **If you are unsure about any of these procedures, contact the SEM Manager before proceeding (Anastasia Micheals 408-646-0138).**

1. To close the **xT Microscope Control program,** click **File,** and select **Exit (**or from the xT Microscope Server [Figure 8], click on **Stop UI**).

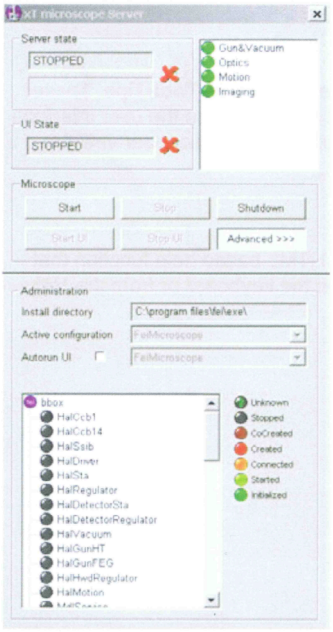


Figure 8. The xT Microscope Server window.

2. To close the **xT Microscope server**, click **Stop**.
3. Shut down the computer by clicking the Windows **Start** button, then selecting **Shut Down.**
4. Turn on the computer by pressing the power button.
5. Log on. The user name is "**user**" and the password is "**user**"
6. Double click the shortcut "**xT microscope Server**"
7. Press **Start** on the **xT microscope Server**
8. Press **Start UI**
9. Again enter "**user**" as both the user name and password
10. "**EXTERNAL"** may be displayed in green in each of the paused quadrants of the UI. This occurs due to a bug in the software. Reset the quadrants by selecting **Scan** from the toolbar, then **Mains Lock**. This should return control of the quadrants.

9.2 Opening the xT Docu Database

1. Hide the UI with the window bar, then open the xT Docu shortcut
2. Open a database located in "Archive on Support-d7935/Fei/. The current database is typically named "SJSU *semester year*"
3. Minimize XT Docu by clicking the **light bulb**

9.3 Blown Filament

If these is no picture with the HV on, check the **Status** console. If the filament or emission current reads zero, then the filament may be blown. Contact SEM manager for replacement.

9.4 Venting

The system is hooked up to a nitrogen dewar, which provides a clean, dry gas which is let into the SEM chamber during venting. The gas pressure should be not more than 1-2 psi, to prevent damage to the EDS system. If the dewar is empty, then the vacuum may not be released completely after the system has been vented. In this case, the nitrogen tank pressure will read zero (or close to zero), and the tank level may show empty (or close to empty).

In this case, take an adjustable wrench and remove the plastic hose from the gas regulator fitting. This will allow a path for room air to enter the chamber. Click "**pump**" and wait until the system begins pumping. Then, click "**vent**" to vent the system. The chamber door should open easily when the vent cycle is completed.

Do not force the door. Should you continue to have problems, contact the SEM Manager.

9.4 Help File

Further information is available in the help file. Press **F1** to access the online help file, or open the pdf file shortcut on the desktop.

10. References

10.1 Print references

Scanning Electron Microscopy and X-ray Microanalysis, Goldstein, J., Springer (2003).

Scanning Electron Microscopy: A Student's Handbook, Postek, Michael T., et al., Ladd Research Industries (1980).

Scanning Electron Microscopy, X-Ray Microanalysis, and Analytical Electron Microscopy: A Laboratory Workbook, Lyman, C., et al, Springer (1990).

Electron Optics, Grivet, P., Pergamon Press (1972)

10.2 Electronic references for Electron Microscopy

http://www.feicompany.com/Resources/StudentLearning/tabid/91/language/en-US/Default.aspx

http://en.wikipedia.org/wiki/Scanning_electron_microscope

http://mse.iastate.edu/microscopy/home.html

http://www.unl.edu/CMRAcfem/em.htm

http://www.unl.edu/CMRAcfem/em.htm

http://www.mwrn.com/microscopy/guide.aspx

10.3 Electronic references for EDS

http://www.edax.com/

http://www.mwrn.com/microscopy/spectroscopy/energy_dispersive.aspx

http://www.mee-inc.com/eds.html

http://www.geology.wisc.edu/~johnf/660.html

Appendix A – Low Vacuum Mode

Low vacuum mode allows a non-conductive or "wet" sample to be imaged by creating a water-vapor-containing atmosphere in the SEM chamber. The pressure allowed range from 0.8 to 1 torr. To operate in **LowVac** mode the **Large Field Detector (LFD)**, shown in Figure A1, must be installed. The **LFD** is inserted into the black connecter in the rear of the specimen chamber, as shown in Figure A2. The **LFD** is normally left in the microscope to enable the user to switch between low and high vacuum mode without venting the chamber. When using LowVac mode a **PLA cone** can be used, if desired. The PLA is installed onto the conical objective (Figure A3).

After installing the LFD and PLA and closing the chamber, select "**Low Vacuum**" on the **Vacuum** console area of the computer interface. Set the desired starting pressure (0.8 to 1.0 torr), and click "**Pump**". Before pumping down, the computer will ask if any cone accessories are being used. Click beside the picture of the accessory being used so that the system is aware of it presence.

The **environmental backing valve (EBV)**, located on the left side of the SEM column, must also be opened (see figure A4). The computer interface will display a prompt when it is time to open this valve.



Figure A1. Underside of the Large Field Detector (LFD).

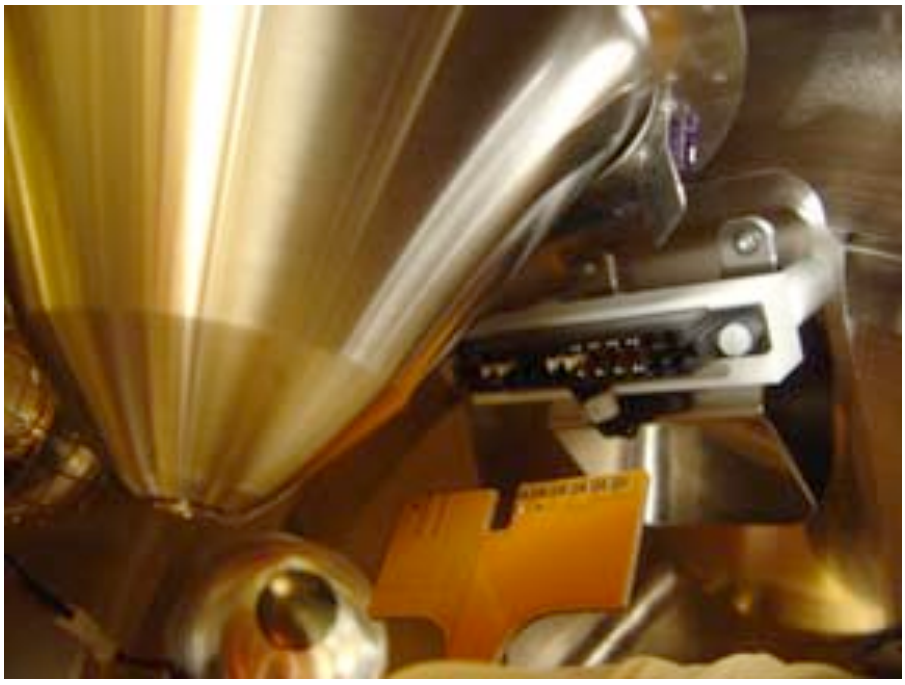


Figure A2. Installing the LFD or GSED into the connector in the rear of the chamber can be awkward; be sure to match up the slots in the card with the connector properly.

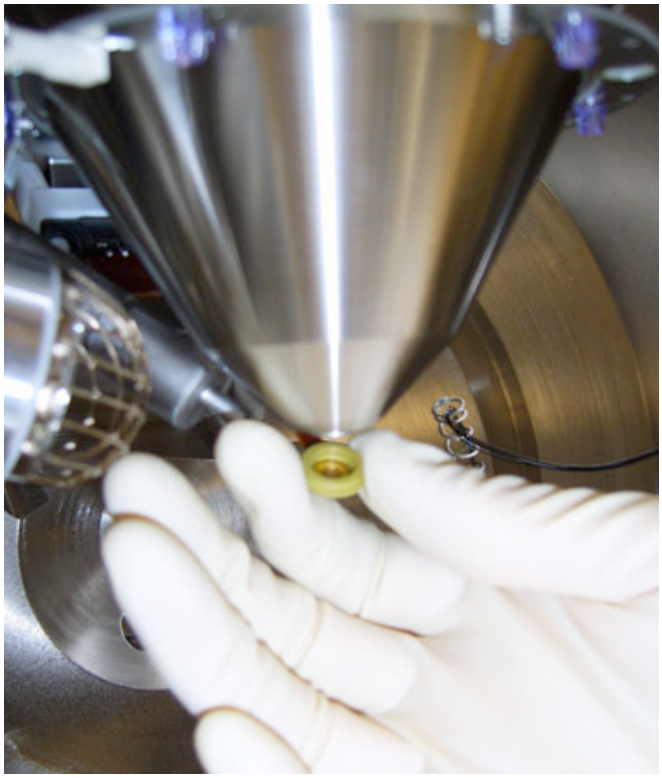


Figure A3. Inserting the GSED or PLA aperture into the conical objective opening where the beam is emitted.



Figure A4. Open the environmental backing valve (EBV) only when prompted by the computer system.

Appendix B – ESEM Mode

ESEM mode is used when a higher water vapor pressure than is allowed in low vacuum mode is needed. The pressure in this case can go up to 30 torr. When using **ESEM** mode the **Gaseous Secondary Electron Detector (GSED)** must be installed. The **GSED** has an integrated flexible pc board that must be inserted into the signal connector behind the conical lens (Figures B1 and B2) and an aperture that must be plugged into the conical objective opening were the beam is emitted. This aperture is inserted over the conical objective in the same way a PLA is in Low Vac mode (see figure A3 in the previous section).

When using **ESEM** mode, select "**ESEM**" on the "**Vacuum"** console area of the computer interface. The **environmental backing valve (EBV)** located on the left side of the column must also be opened (see Figure A4 in the previous section). The computer interface will display a prompt when it is time to open this valve.

Installing the Gaseous Secondary Electron Detector (GSED)

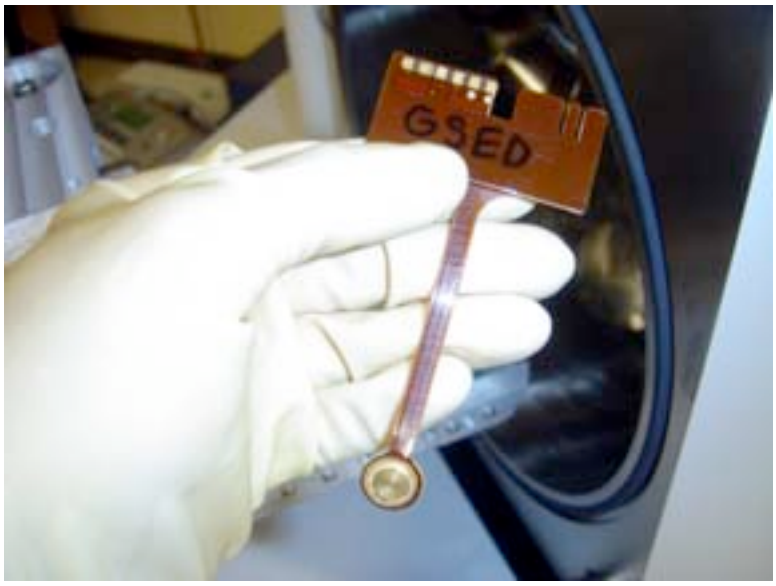


Figure B1. Gaseous Secondary Electron Detector (GSED).


Figure B2. Inserting the GSED card into the connector slot.