

Standard Operating Procedure of Environmental SEM

Part I: FEI Quanta 200-HiVac Mode

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.



Chamber vacuum

- High-vacuum: $< 6 \times 10^{-4}$ Pa
- Low-vacuum: 10 to 130 Pa
- ESEM-vacuum: 10 to 2600 Pa

Resolution

- High-vacuum
 - 3.0nm at 30kV (SE)
 - 4.0nm at 30kV (BSE)
 - 10nm at 3kV (SE)
- Low-vacuum
 - 3.0nm at 30kV (SE)
 - 4.0nm at 30kV (BSE)
 - < 12 nm at 3kV (SE)
- Extended vacuum mode (ESEM)
 - 3.0nm at 30kV (SE)
- Accelerating voltage: 200V – 30kV
- Probe current: up to $2 \mu\text{A}$ – continuously adjustable

1. Specimen Preparation and Handling

- High-vacuum mode for imaging and microanalysis of conductive and/or conventionally prepared specimens;
- Low-vacuum mode for imaging and microanalysis of non-conductive specimens without preparation;
- ESEM mode for high-vacuum incompatible specimens which are impossible to investigate with traditional EM methods.

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 (Fig. 1).

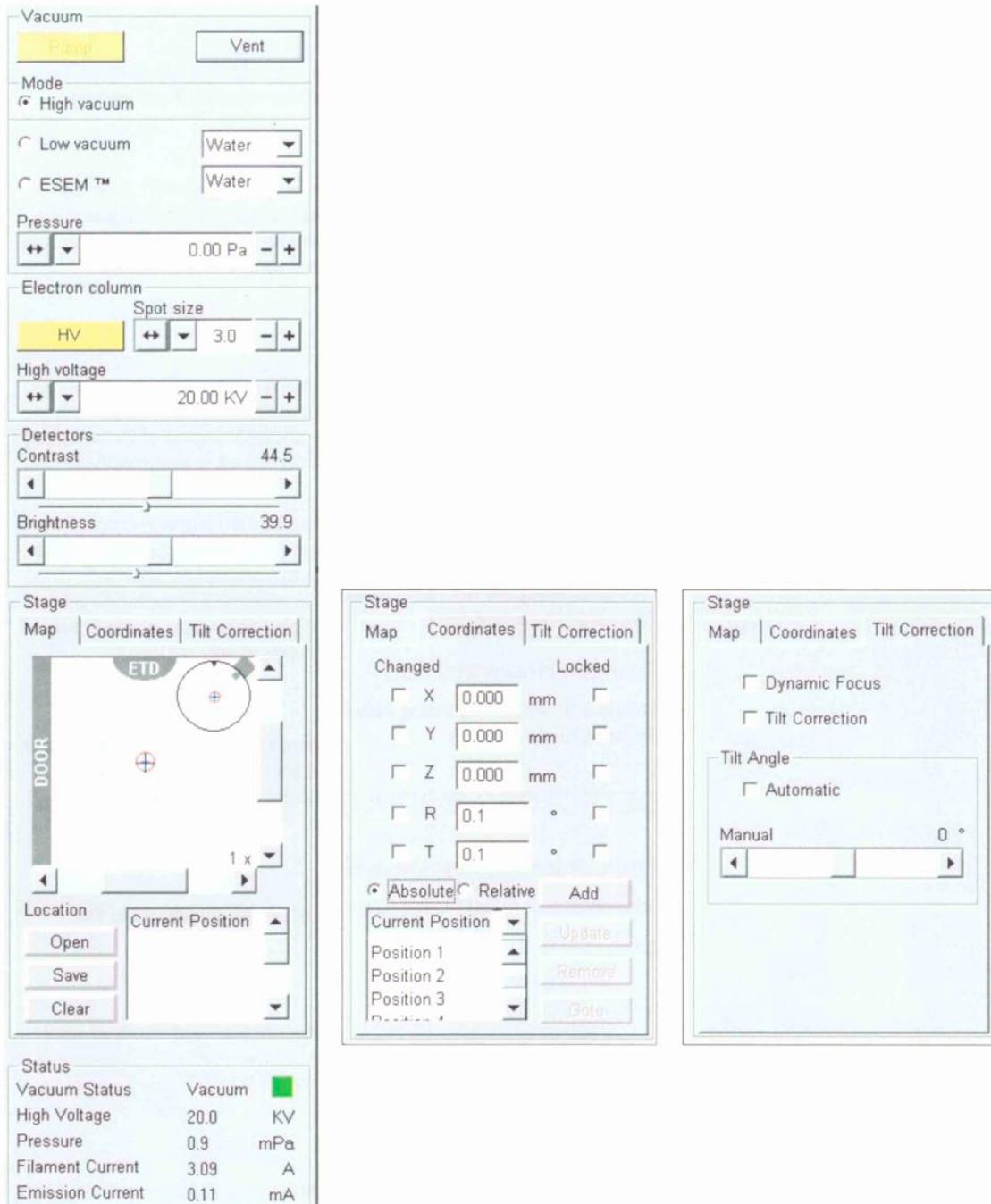


Fig. 1 The **Work Page**, located on the right side of the xT Microscope Control window.

Images are displayed in the main window on the computer screen. The main window is divided into 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.

3. Startup Procedure

**Users must wear latex gloves when handling detectors or loading specimens.*

1. Vent the specimen chamber by pressing the “Vent” button in the control program.
2. As the system vents, the **Vacuum Status Indicator** 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.
3. Mount your specimen to a specimen stub (Fig. 2), insert the pin into the opening in the top of the stage (Fig. 3).

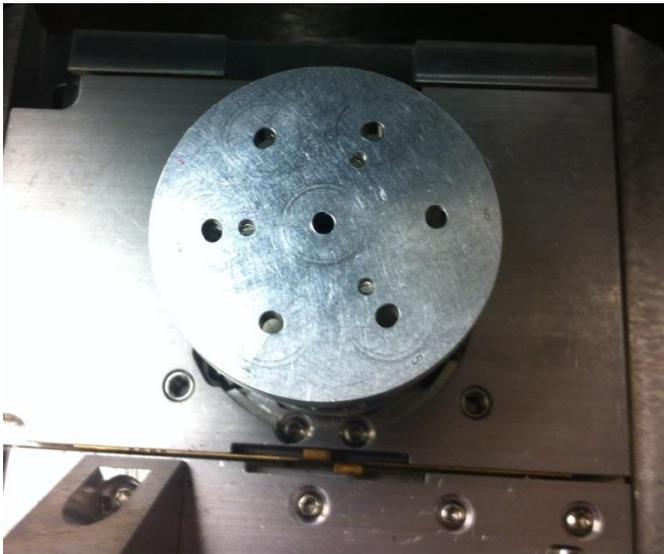


Fig. 2 Specimen stub

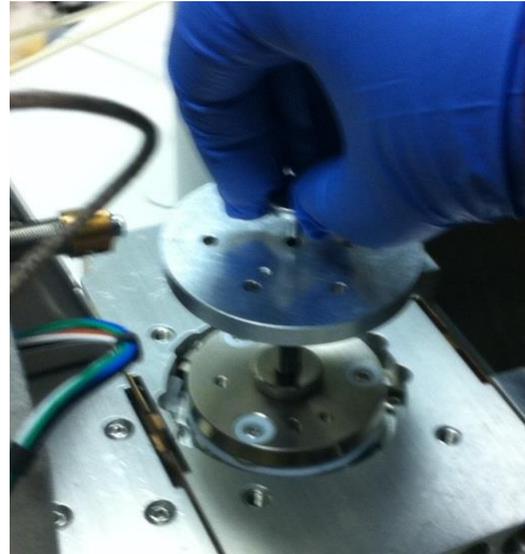


Fig. 3 Insert the specimen stub into the stage.

8. Close the chamber door and click the “**Pump**” 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 5×10^{-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.

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
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 = (Ctrl 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 Fig. 4 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.

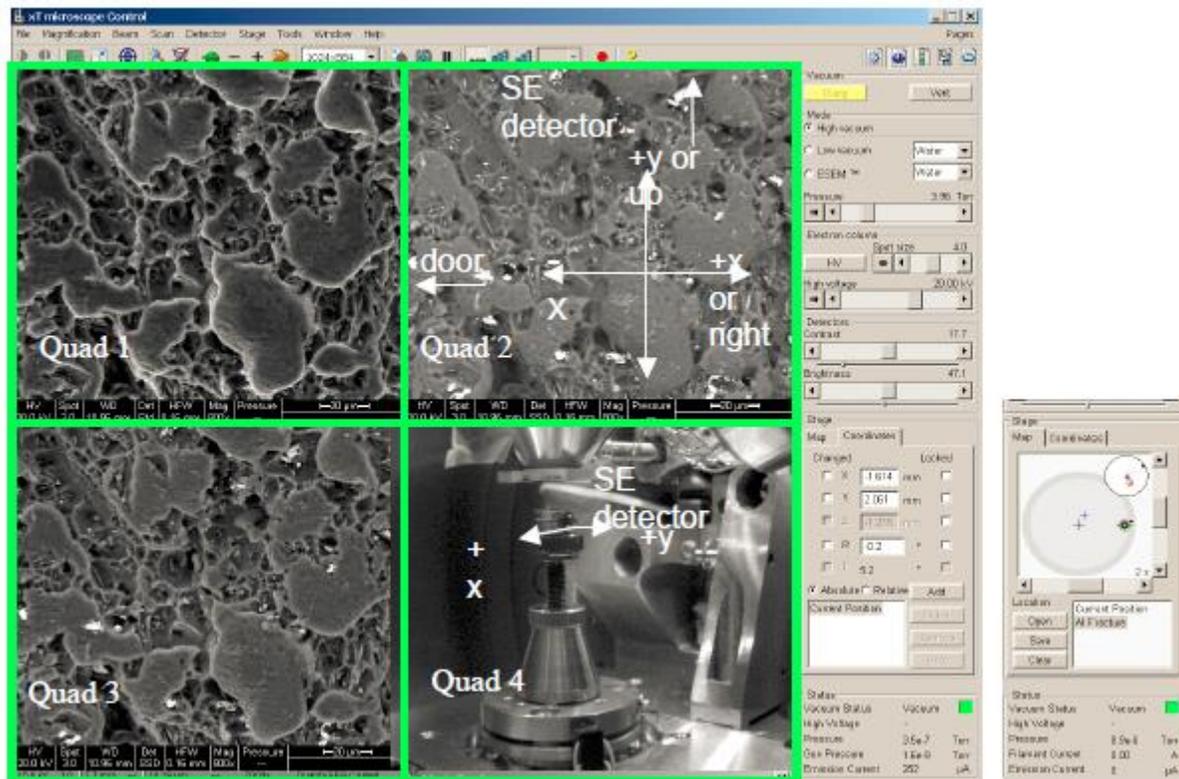


Fig. 4 Stage Orientation and Axes.

5. Image Capture

1. To capture an image click on the desired quadrant, 1, 2 or 3. To have good resolution in your saved image, the image size, located to the right of the rabbit button, should be 1024x884.
2. Now click the “camera” button on the toolbar to initiate a slow scan and capture a final image.

7. Shut-down Procedure

1. **Turn off** the accelerating voltage, and 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 gray box.
4. The specimen chamber can now be opened and the sample can be removed. **Remember user must wear latex gloves when unloading specimens or handling detectors.**
5. 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.
6. Wait until the vacuum indicator on the “Vacuum” console has turned green, a few minutes after pump down is commenced.

Part II: FEI Quanta 200-ESEM Mode

I Install the GSED

With your gloved right hand, grasp the detector by the rigid connector end. Hold it with the detector head facing back towards you, and the yellow Torlon ring facing up. Insert the detector (gold fingers facing forward) into the connector located at the back of the chamber, behind the conical lens. This is made easier by inserting the right side of the detector in to the visible portion of the connector, then rotating the detector into position. A keyed position in the connector will prevent the user from inserting the detector up-side-down.

Next, place the yellow Torlon ring of the detector head under the lens insert and press the detector head up onto the insert. This requires little force and can be done with one finger. The yellow Torlon seal should be fully in contact with the lens.



Fig. 1 GASEOUS SE DETECTOR (GSED)

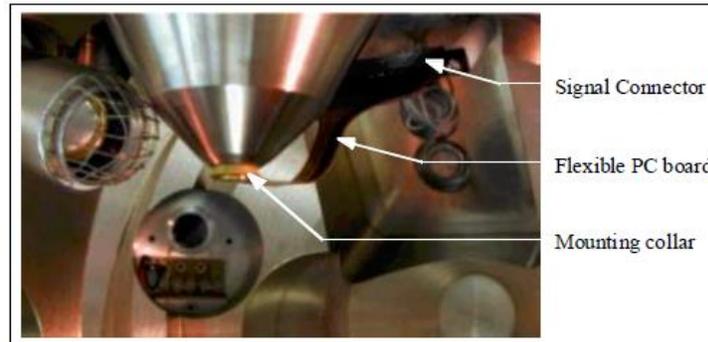


Fig. 2 GSED INSTALLED IN THE SEM

II Install the Cold Stage



Fig. 3 Cold Stage Module

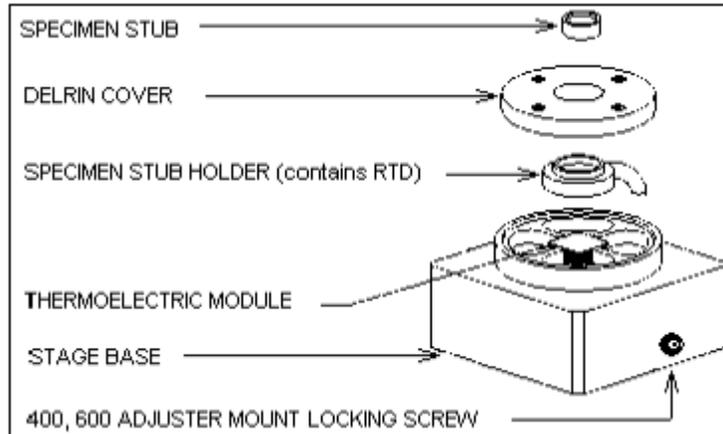


Fig. 4 COLD STAGE ASEMBLY

The primary constituents of the stage are the THERMO ELECTRIC MODULE, which alters temperature, and the RTD, which measures temperature. The RTD is located inside of the SPECIMEN STUB HOLDER, a plate into which the SPECIMEN STUB is inserted. The thermoelectric module and stub holder are held onto the main body of the stage using thermal grease; and the DELRIN COVER holds the entire assembly in place. When fully assembled, the RTD will be situated between the sample and the thermoelectric module, thus giving accurate temperature readings. The signal cable is permanently attached to the stage base, and connects to the inside of the Chamber Feedthrough Assembly.

The Cold stage is mounted onto the motorized stage through a Stage Adapter. The following instructions describe how to install the Cold stage. Vent the specimen chamber and open the stage door. Remove any other sample mount from the stage rotation head.



Fig. 5 STAGE ADAPTER AND SAMPLE MOUNT

Place the Stage Adapter on the rotation head of the stage and, through the center, screw in the Sample Mount supplied with the Stage Adapter piece. Make finger tight. The Sample Mount is not Click-fit so the **Rotation** control will need to be **LOCKED**.

Slacken off the set screw on the right of the base of the Peltier module using a metric 2 mm hex wrench, then place the module on and over the Sample Mount. The water hose connectors go towards the rear of the chamber. Tighten the set screw with the 2 mm hex wrench.



Fig. 6 COLD STAGE INSTALLED

III CABLE AND COOLING HOSE CONNECTION

(1) Connecting the stage to the Chamber Feedthrough Plate

Install the Signal Cable. This is the 9-pin D-type connector that is attached to the thermoelectric stage. The loose end of this connector should be installed on the corresponding connector on the inside of the feedthrough plate.

(2) Connect the Cooling Water Hoses.

The short set of hoses on the thermoelectric stage go to the inside of the feedthrough plate.

(3) Connecting the Chamber Feedthrough Plate to the Controller

One end of this cable will go to the outside of the Chamber Feedthrough Plate on the connector labelled PELTIER; and the other end to the rear of the PSC board in the detector rack.

IV Enable the water flow

Turn on power to the box. An alarm will sound, indicating that there is no flow through the box. Push and hold the START FLOW button. Keep holding this button down until all the air is out of the water lines; This can be seen as water flows through them. Once the lines are flowing clear, release the START FLOW button. Make sure that the FLOW OK light on the box is on. This indicates that water flow is working; i.e., that there are no leaks in the system. The FLOW OK light will remain illuminated until there is a leak, or if the STOP FLOW button is pressed. The STOP FLOW button can be pressed at any time to close the valves and shut off the water flow, for whatever reason. If STOP FLOW is pressed, an alarm will sound. The START FLOW button must be pressed and held again to re-establish flow through the system.

V Enable the cooling stage and PRESSURE VS. TEMPERATURE CONTROL

As a general rule, condensation is achieved by first setting the pressure to 4.0 Torr (540 Pa), bringing the sample to 5°C, then raising the pressure until water condenses on the sample, **making sure to keep the pressure below the condensation point 6.5 Torr (860 Pa)**. It is better to use pressure to control condensation, as opposed to temperature. There are two main reasons for this. Firstly, the heat pumping capability of the thermoelectric module is very small; therefore temperature is harder to control accurately. Secondly, by controlling pressure, it is easier to keep it below the condensation point, which will prevent water from raising the temperature of the sample. When water has to cool, it takes longer to reach the setpoint.

KEEPING THE SAMPLE WET

Water in the sample will tend to evaporate during the pump-down cycle. The simplest way to keep the sample wet is to add enough fluid to the sample during preparation so that it will still retain moisture after pump-down.

The **Cooling** button toggles to enable or disable the software control of the Peltier stage from the UI switches the manual mode on or off. When the cooling stage software control is enabled the button is **Yellow**.

VI DISCONNECTING THE WATER LINES

When one has finished using the Peltier stage, the water must be drained out of the tubing and the connectors inside of the chamber. The following instructions describe how to do this.

- Allow the stage to cool to room temperature.

- **IMPORTANT: Shut down the water chiller.**
- Disconnect one of the water lines inside the chamber and elevate it to keep water from dripping out. While holding it up, push START FLOW on the water flow box and hold it down until all the water is drained out of the tubes. Make sure the system is drained before disconnecting the other hose. This will prevent excess water from dropping into the chamber and slowing the time it takes for the chamber to pump down again.
- Connect the water line plugs into the Feedthrough plate.
- Once the stage has been drained and both water lines have been removed, blot out any remaining water from the connectors using a cotton swab or paper towel.

VII Removing the GSED

DO NOT pull the detector from the connector first, then pull down to remove the detector head from the insert. This will damage the detector. To prevent damaging the detector, it is important to remove it as described here. To remove the GSED, first remove the detector head from the lens insert. Do this by catching a fingernail or thumbnail (of the gloved hand) on the FRONT of the yellow Torlon ring and pull down. There is a shoulder machined into the Torlon ring which is specifically designed for this purpose. Next, pull the other end of the detector out from the connector.