

Hitachi S-4800 FE-SEM

Operation Instructions

For additional assistance, please contact the facility manager.

Please contact in emergency:

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S-4800 reservations are made using the EPIC FOM online reservation system. Please follow all EPIC facility rules for using this system. There is a hardware control system on the 4800, so the system will NOT function unless you are logged in. Tampering with or disabling the hardware control may result in revocation of your EPIC privileges.

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

About saving your data: During your session, you may store image data and EDS data directly to the SEM Server (U:). The SEM server is available on the middle computer in the specimen prep lab. You can transfer your data from this computer to a USB etc. You should never take your data directly from either the SEM or EDS computers.

System Startup

1. Log in to FOM and log in to your reservation.
2. Assemble the sample holder and load your sample stubs onto the holder. Verify the following:
 - a) Sample height is flush with the height gauge (the sample can be shorter than this, but NEVER taller than the gauge – this is the “Standard” height)
 - b) The screw does not stick out below the bottom surface of the holder.
 - c) Sample stubs are secured with set screws.
3. Verify that the Hitachi PC-SEM software is running.
(Note: To log into the PC and PC-SEM software, the username and password are set by default to “semuser”)
4. Switch the **Camera On/Off** button below the chamber scope to the left of the PC monitors. (Note: You will get no EDS or BSE signal with the camera on and in general this should be left **Off** when you aren’t using the chamber scope and at the end of your session.)
5. Verify that the stage is at the exchange position (green light next to EXC) and if it is not, press the **EXC** button to move the stage to this position.
6. Press the **AIR** button on the load lock and wait for the beep.
7. Open the load lock door using the metal handle (do not use the exchange rod as a handle).

8. With the exchange rod in the **UNLOCK** position, load the sample holder by sliding it onto the banana clips and turning the exchange rod to the **LOCK** position.
9. Close the chamber door and press the **OPEN** button. The load lock will automatically pump down and the valve separating it from the main chamber will open. When the operation is complete, you will hear another beep.
10. Slide the exchange rod all the way into the chamber to load the sample holder onto the stage.
11. Turn the exchange rod to the **UNLOCK** position and pull the rod all the way back until it snaps into place.
12. Press the **CLOSE** button.
13. Press the **HOME** button in the PC-SEM interface to center the stage under the objective lens.

General Operation and Alignment

1. Under the **Setup** menu, click on **HV** to open the HV setup menu. There are several important items in this menu:
 - a) **Vacc**: Accelerating Voltage – variable from 500V-30kV
 - b) **Ie**: Emission Current – typically set to 10 μ A for imaging. May be increased for applications where somewhat higher probe current is needed, such as EDS.
 - c) Emission Adjust (checked by default)
 - d) Deceleration Mode (unchecked by default) – Deceleration mode allows the user to apply up to 1.5kV negative bias to the sample stage. This effectively decelerates the primary electron beam, allowing for landing energies as low as 100V. By activating this mode, the Vacc control changes to Vlanding and is adjustable from 0.1-1kV with a variable amount of deceleration.
 - e) Flashing – The tip needs to be flashed about once a day and the software is programmed to prompt you to flash (Please Flash in red/blinking) when necessary. **Do not flash the tip unless prompted.** To flash the tip, click on Flashing and press Execute.
2. Under the **Setup** menu, click on Column setup menu. There are several more important items in this menu:
 - a) **Probe Current**: Norm by default – set to High for high current mode for EDS mapping, etc.
 - b) **Focus Mode**: Use UHR mode for short WD (<5mm) and HR mode for long WD (>5mm).
 - c) **Cond Lens 1** (Checked): 5 is the default setting and not usually necessary to change. Higher values may provide somewhat better resolution, but with a noisier image.
 - d) **Cond Lens 2** (Checked)
 - e) Focus Depth: 1.0 by default – increasing will improve depth of focus.
 - f) Magnetic Sample (Unchecked)
 - g) Degauss: Use to periodically clear lens hysteresis (shortcut F2)

- h) Specimen Bias Voltage (Unchecked): Useful improve even brightness at low magnifications.
3. Press the **ON** button in the upper-left corner of the screen to apply the high voltage.
 4. You will see a prompt that displays the sample dimensions. If these are not correct, click on the **Stage** tab and press the **Set** button to enter the correct Size (width) and Height of your sample (Again, this should always be the Standard height)
 5. Click on the **SEM** tab and select the detector you wish to use:
 - a) Upper SE: High resolution SE imaging. Best at short WD (<10mm)
 - b) Upper BSE: Low kV BSE detector in HA mode and LA mode collects low-angle BSE with variable energy filter (1-100eV).
 6. Lower the Magnification (Hit the H/L button to toggle between High and Low Mag modes) and adjust the brightness and contrast to visualize the center of the sample holder.
 7. Use the X/Y trackball to control the sample stage and move a sample into view.
 8. Focus on the surface of the sample to determine the current WD.
 9. Use the focus and Z-control on the stage to set the appropriate WD. Choose a very short WD (<3mm) for the highest resolution imaging at low kV. 10mm is the analytical working distance.
 10. After setting the WD, you can switch the **Camera On/Off** button to turn off the camera until you need it again.
 11. Zoom into a relatively high magnification (~20,000X) and focus on a small feature.
 12. Under the **Operate** menu, select **Alignment**.
 13. Select **Beam Align** and center the bright spot using the X/Y Stigma/Align knobs on the control surface.
 14. Select **Aperture Align** to turn on the focus wobble and use the X/Y Stigma/Align knobs to minimize any image translation.
 15. Use the same method to minimize translation for both **Stigma Align X** and **Stigma Align Y**.
 16. Exit the alignment menu and focus on a small feature.
 17. Adjust the **X/Y Stigma/Align** knobs to correct for any astigmatism – the result will be a sharp image that doesn't stretch during focusing.
 18. If you are using a very low V_{acc} or $V_{landing}$, you may find the ULV Align under the Alignment menu helps to optimize the image.

Image Capture

1. The preferred method for digital imaging is to capture the images with the PC-SEM software and transfer into the Quartz PCI software.
2. Open the **Quartz PCI** software from the Windows Start Menu.
3. Under the **File** menu, select **Series Name** and enter a project name. All images transferred to PCI will be named series01, series02, etc.
4. From the PC-SEM software, adjust the image resolution by clicking on the black triangle above the capture button.

5. The capture scan speed is adjustable from the **Setup** menu, under **Image Display** setup. For frame integration, use a fast scan mode and select the number of frames to average. For a slow scan image capture, use a slow scan mode and select the scan time.
6. Click on the Image **Capture** button to acquire the image.
7. All images will appear in the Captured Images window and you may transfer them to PCI by click the **PCI** button.
8. From PCI, you can save all images at once by selecting **Export All** under the File menu.

Digital Imaging and EDS with Inca

1. Open the INCA software by double-clicking on the desktop icon.
2. 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 allow 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)
3. 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.
4. Basic instructions are presented below, but more detailed information can be readily obtained from the bubble help (upper right corner of the screen).
5. 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
6. 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.
7. 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.
- 8. To save any images, maps or EDS spectra, right click and select Export.

Shut Down and Sample Removal

1. Click the Acceleration Voltage **OFF** button on the control panel to shut off the high voltage.
2. Click the **EXC** button to send the stage to the exchange position.
3. Switch the **Camera On/Off** button to turn on the chamber scope.
4. Press the **OPEN** button to pump down the load lock and open the separation valve.
5. When the valve opens and the buzzer sounds, push the exchange rod (in the **UNLOCK** position) into the chamber and slide it into the sample holder.
6. Turn the knob on the exchange rod to **LOCK** and pull the rod all the way back.
7. Press the **AIR** button to close the separation valve and vent the load lock.
8. When the chamber vents and the buzzer sounds, open the load lock door with the handle.
9. Remove the sample by turning the rod to the **UNLOCK** position.
10. Close the chamber door and press the **EVAC** button to evacuate the load lock.
11. Switch the **Camera On/Off** button to turn off the chamber scope.
12. Clean up any mess you made in the SEM room.
13. Transfer any data to the SEM Server if you have not already done so.
14. Log into FOM and log out of your reservation.