

Hitachi S-3400N VP-SEM Operation Instructions

For additional assistance, please contact the facility manager.

Please contact in emergency:

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S-3400 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 3400, 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 locally on the D:\Users directory or directly to the SEM Server (U:\). EDS data must be stored locally first on the H:\Users directory and then can be transferred to the SEM Server via the desktop shortcut. You may log into the EDS system with username “imix” and password “imix1”. If needed, the username and password for the network are both “semuser” – this is the same for both the SEM and EDS computers.

System Startup

1. Log in to FOM and log in to your reservation.
2. Prepare the sample as necessary and mount on a Hitachi sample holder.
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. Vent the chamber by pressing the **AIR** button through the PC-SEM interface.
6. The stage will automatically be sent to the Home Z65 position and the chamber will vent. You can tell the chamber is vented by listening for 3 beeps, or when the light by the AIR button stops blinking.
7. **Use the sample gauge to measure both the sample Height and Size (width).** Enter these values into the dialog box, rounding up to the next value if it is between two choices. This is critical so you don't crash your sample into fragile components around the objective lens.
8. Load your sample holder onto the stage

9. Hold the chamber door shut and press the **EVAC** button in the PC-SEM interface.
10. The chamber will take about 90 seconds to pump down and you can tell it is at high vacuum when the light by the EVAC button stops blinking.
11. Select **Optics Setup** from the **Setup** menu.
12. Set the high voltage (HV) level, which is variable from 300V-30kV and turn the beam on by pressing the **On** button.
13. As soon as the high voltage is on, select **Mid** filament saturation and press **AFS** to engage the automatic filament saturation. Whenever the HV is changed, always re-saturate the filament by pressing AFS.

General Operation and Alignment

1. Under the **Image** tab, select the SE detector and set the SE bias to the default value (30).
2. Lower the magnification all the way and adjust the brightness and contrast (or hit ABCC) to see the sample holder.
3. Use the track ball to control the stage X/Y movement and locate the sample you want to image.
4. From the **Stage** tab, hit the analysis button to send the stage to a ~10mm working distance (Note: Make sure your sample dimensions were correct or you may crash the stage. 10mm is the analytical working distance for quantitative EDS, but for imaging, use a shorter working distance for improved resolution and a longer working distance for better depth of focus).
5. Under the **Cond.** tab, set the desired **probe current** level – this controls the strength of the condenser lenses in the column. Higher values of probe current will result in greater signal strength, but poor resolution – 50 is a good starting point.
6. Insert the desired aperture. Align the aperture for the brightest image using the X and Y knobs on the aperture,
7. Zoom into a relatively high magnification (>10,000X) and find a feature on your sample to use for alignment.
8. Select **Alignment** under the **Operate** menu.
9. Select **Aperture Alignment** - the focus wobble will turn on automatically. Adjust the X and Y **Stigma/Alignment** knobs to minimize translation of the image.
10. Select **Beam Align. Tilt** and hit the **Auto** button. This will align both the Beam Shift and Tilt – you can do these manually by selecting each alignment and adjusting the X/Y Stigma/Alignment knobs for the brightest image.
11. Select **Stigma Align. X** and use the X and Y stigmator/alignment knobs on the control surface to minimize translation of the image. Select **Stigma Align. Y** and repeat.
12. Close the alignment menu and use the X/Y **Stigma/Alignment** knobs on the control surface to correct for astigmatism (Hint: First get good focus and then adjust the stigmators one at a time for the sharpest image).

BSE Detector

1. The BSE detector is useful for obtaining backscattered electron images, which provide 'Z' contrast, i.e. contrast based on average atomic number.
2. You may select the BSE detector by choosing **BSE** on the **Image** tab.
3. The BSE detector has 5 segments, which can be turned on and off independently for different modes of operation. Comp mode uses all 4 axially mounted segments for good compositional contrast, while Topo and 3D modes have different configurations to enhance topographic contrast. If you press the **Detail** button, you can fully configure the detector.
4. The **BSE Gain** setting is used to control the preamplifier gain on the detector, with the default setting in the third position. For low signal applications (ie, low kV, low Probe Current, etc.) you may need to increase the gain to get a good image. If you find the contrast is saturated, you should decrease the gain.

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 automatically named *Project01*, *Project02*, etc.
4. From the PC-SEM software, adjust the image capture options by clicking on the capture setting button (black triangle above the capture button).
5. Click on the Image **Capture** button to acquire the image.
6. All images will appear in the Captured Images window and you may transfer them to PCI by click the **PCI** button.
7. From PCI, you can save all images at once by selecting **Export All** under the File menu.

X-ray Microanalysis

1. Energy Dispersive X-ray analysis (EDS) is available through the attached PGT system.
2. To capture an x-ray spectrum, select **X-ray Collection** under the **X-ray** menu. Set the desired parameters and hit start.
3. Make sure the chamber scope is turned off, or you will get 100% dead time and no spectrum.
4. 20-25 kV is a good starting voltage for EDS. You can lower this number to detect lighter elements.
5. When capturing a spectrum the counts per second (cps) should be between 3,000 and 5,000. Adjusting the probe current higher will give you more cps and lower will give you fewer cps. Remember to click "AFS" when you change the probe current to resaturate the filament.
6. Image capture and x-ray mapping are available through this system as well by select **Image Collection** under the **Image** menu.
7. For more details on the PGT system, see the operation manual or contact the facility manager.

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 **AIR** button to automatically send the stage to the Home Z65 position and vent the chamber.
3. After the chamber vents (you will hear 3 beeps), gently open the door and remove your sample.
7. While holding the door firmly shut, press the **EVAC** button to evacuate the sample chamber.
8. Wait for the chamber to reach high vacuum. If you used the VP-SEM mode, make sure you pump down to high vacuum at the end of your session.
9. Log in to FOM and log out of your reservation.
10. Make sure to clean up any mess you have made, any tape on the sample holders, etc.