

Hitachi S-3500N VP SEM

Operation Instructions

For additional assistance, please contact the facility manager.

Please contact in emergency:

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S-3500 reservations are made using the EPIC FOM online reservation system. Please follow all EPIC facility rules for using this system.

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

You are asked to make a copy of your data on your own disk IMMEDIATELY after your session is finished. You may save your data on a 100MB Zip disk, CDR/CDRW or transfer your data by FTP. The data may be deleted at any time without notice. EPIC is not responsible for any data loss.

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. Check the height of the sample using the sample height gauge. The total height of the sample holder and sample should not exceed the top of the height gauge.
3. Verify that the Hitachi PC_SEM software is running.

(Note: The software will occasionally crash and the PC will need to be rebooted. It is sometimes sufficient to simply restart the PC, but it is often necessary to re-sync the PC and SEM. To do this, simply shutdown windows, turn off the display power on the left side of the column control panel, wait ~10 seconds and turn the display power back on. The PC should automatically boot up – hit ‘cancel’ at the network prompt - and the PC_SEM software should load automatically. If the software still doesn’t load, try it again or contact the facility manager.)

4. Check to make sure the SEM stage is in the sample exchange position: **X=30, Y=20, Z=EX, Tilt = 0.**
5. Make sure the backscattered electron (BSE) detector is withdrawn and the accelerating voltage (HV) is off.
6. Depress the **AIR/EVAC** button on the column control panel to vent the sample chamber. Do not try to force the door open by pulling on it – wait for the chamber to vent and the door to release on its own.
7. Place the sample holder in the chamber. Using the sample chamber height gauge, double-check the height of the sample to insure the holder is fully inserted.

8. Select **Vacuum Mode** under the **Setup** menu and set the vacuum level. For standard, high vacuum operation, choose SEM and for variable pressure operation, choose the VP-SEM mode.

(Note: For VP-SEM mode the standard lower SE detector cannot be used and the system will automatically select the BSE detector. To insert the BSE detector, lift gently on the bottom rod to release the detector and slowly insert the detector by pushing it toward the column.)

8. While holding the door firmly shut, press the **AIR/EVAC** button to evacuate the sample chamber.
9. Watch the vacuum indicator lights on the screen as they change from RED to GREEN to BLUE. The **BLUE** light indicates that the chamber has reached the desired vacuum level.
10. Once the chamber reaches vacuum, select **HV Control** under the **Setup** menu.
11. Select the desired accelerating voltage and press the **HV ON** button.
12. As soon as the high voltage is on, select Low, Med or High filament saturation and hit **AFS** to engage the automatic filament saturation.

(Note: Never select high filament saturation if the sample chamber has just been at atmospheric pressure since the filament will likely become over-saturated as the vacuum improves. Choose Med or Low for normal operation to extend the filament life. When changing the accelerating voltage, never just switch it on the fly. First, select HV OFF, then change the accelerating voltage, select HV ON and then re-saturate the filament to the desired level.)

General Operation and Alignment

1. Locate your sample using the X, Y and rotate stage controls.
2. Select **Column Setup** under the **Setup** menu.
3. Set the **working distance (WD)** to the desired level and then adjust the Z-axis control to raise/lower your sample until it comes into focus.

(Note: For higher resolution, use a shorter working distance. For better depth of focus, use a longer working distance. For EDS analysis, the system is optimized at a 15mm working distance – this is of particular importance for any quantitative analysis.)

4. Set the desired **beam current** level – this controls the strength of the condenser lenses in the column. Higher values of beam current will result in greater signal strength, but poor resolution.
5. Insert the desired aperture. Align the aperture for the brightest image using the X and Y knobs on the aperture.
6. Zoom into a relatively high magnification (>10,000X) and find a feature on your sample to use for alignment.
7. Select **Alignment** under the **Operate** menu.

8. Select **Aperture Alignment** - the focus wobble will turn on automatically. Adjust the X and Y knobs on the aperture to minimize translation of the image.
9. Select **Gun Shift** and hit the AGA button.
10. Select **Gun Tilt** and hit the AGA button.
11. Select **Stig. X** and use the X and Y stigmator/alignment knobs on the control surface to minimize translation of the image. Select **Stig. Y** and repeat.
12. Close the alignment menu and use the X/Y stigmator/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).

Image Capture

1. The preferred method for digital image capture is through the PC_SEM software.
2. Select **H.R. Capture** under the **Scan** menu.
3. Select the desired scan speed and resolution then hit the H.R. Capture button to capture the image.
4. To save the image, select **Save Image As** 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. Image capture and x-ray mapping are available through this system as well by select **Image Collection** under the **Image** menu.
4. For more details on the PGT system, see the operation manual or contact the facility manager.

Shut Down and Sample Removal

1. Click the HV button on the screen to shut off the accelerating voltage.
2. Retract the BSE detector if it was used.
3. Move the stage to the sample exchange position: **X=30, Y=20, Z=EX, Tilt = 0**.
4. Depress the **AIR/EVAC** button on the column control panel to vent the chamber.
5. After the chamber vents, remove your sample. Do not try to force the door open by pulling on it – wait for the chamber to vent and the door to release on its own.
6. Select **Vacuum Mode** under the **Setup** menu and set the vacuum level to SEM mode (high vacuum).
7. While holding the door firmly shut, press the **AIR/EVAC** button to evacuate the sample chamber.
8. Watch the vacuum indicator lights on the screen as they change from RED to GREEN to BLUE. The **BLUE** light indicates that the chamber has reached the desired vacuum level. Do not leave until the system is under high vacuum.
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.