

# HITACHI S-4500 cFEG SEM

## Operation Instructions

For additional assistance, please contact the facility manager.

Please contact in an emergency:

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S-4500 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 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.

### Sample Loading

1. Log in to FOM and log in to your reservation.
2. Assemble the sample holder according to the picture in the room. Be sure to verify the following critical items:
  - a) The total sample height is no taller than the sample height gauge.
  - b) The sample holder base is in the correct orientation (wide part on the bottom).
  - c) The screw does not protrude from the bottom of the sample holder.
  - d) All stubs are secured using a set screw – especially if your sample is magnetic or if you will be tilting the stage.
3. Turn on the **DISPLAY POWER** switch on the front of the column unit. This is done by pulling out slightly and flipping the switch into the ‘up’ or ‘on’ position.
4. Verify the following:
  - a) The **OBJ. APERTURE** switch is set to **HEAT**.
  - b) The green lights for **DP/TMP**, **WATER**, **AIR PRES.** and all three ion pumps (**IP**) are on.
  - c) The **S.C.** (Sample Chamber) and **S.E.C.** (Sample Exchange Chamber) vacuum gauges are at **HIGH** vacuum.
  - d) The **S.C. AIR LOCK VALVE** is in the **CLOSE** position.
  - e) The **S.C./S.E.C.** switch is set to **S.E.C.**
  - f) The **SPECIMEN STAGE** switch is set to the **FREE** position.
  - g) The **MV1** valve is in the closed position (turned fully clockwise).
  - h) The stage is in the exchange position: **X=25, Y=25, Z=15, T=0, R=0**.
5. Press the **AIR** button on the column control panel to vent the S.E.C.

6. Once the S.E.C. is vented, open the S.E.C. door by grasping the door itself (i.e. do not handle the exchange rod).
7. Screw the exchange rod into the sample holder by turning the black knob on the end of the rod.
8. Fully retract the exchange rod and verify the door o-ring is properly seated and free from dust/debris.
9. Close the exchange door and hold the door firmly when pressing the **EVAC** button on the front of the column unit.

(Note: If you hear hissing from the door, stop pumping immediately by pressing **Air**, check the door o-ring and evacuate the exchange chamber again.)

10. When the S.E.C. vacuum gauge is at **HIGH** vacuum open the **MV1** valve by turning the handle counterclockwise.
11. Turn on the chamberscope by pressing the white **CAMERA** button to the right of the monitors.
12. Transfer the sample from the S.E.C. into the S.C. by pushing the exchange rod into the sample chamber. Slide the sample holder onto the stage (the rod should be fully inserted) then unscrew the exchange rod using the black knob and fully retract the rod.
13. Close the **MV1** valve by turning the handle fully clockwise.

### **General Operation and Alignment**

1. Make sure the S.C. and S.E.C. vacuum gauges are at **HIGH** vacuum. Move the **S.C. AIRLOCK VALVE** switch to the **OPEN** position – the green **READY** light above the **HV OFF** should come on.
2. Check the logsheet or FOM to see if you are the first user of the day. The first user each day must flash the field emission tip prior to use. The buttons required for this procedure are located in the top left-hand corner of the console. Press the **FLASH** button once (the light above the button will blink). Press the **HV ON** button once (the emission current will rise, hold for two seconds and then fall again). The tip should not be flashed more than once per day.

(Note: The **PF** keys bring up the menus on the SEM CRT. Use the **UP/DOWN** arrows on the keypad to scroll from one item to another. Use the **LEFT** arrow to select a particular item. Once an item has been selected, enter the required parameter and press return. To clear the menu from the CRT, press the **PF16** (EXIT) button.)

3. Press **PF1** to set the accelerating voltage. Metallurgical samples benefit from higher accelerating voltages (15-25kV); non-conductive samples benefit from lower accelerating voltages (1-15kV). For EDS, the recommended accelerating voltage is 1.5 to 2 times the highest energy line in the spectrum. Example: the Cu K line appears at 8.041keV; the proper accelerating voltage for a sample in which copper is the highest energy line would be 12 – 16kV.
4. Press **PF2** to set the column conditions.

- a) The **Cond. Lens1** setting controls the strength of the condenser lens system – a higher value means a smaller probe (higher resolution) but less current (lower signal strength). Somewhere from **3 to 6** is usually a good starting point.
  - b) The **Cond. Lens2** should generally be **ON**.
  - c) The **Emission** current should nominally be set to 10  $\mu$ A for imaging, but a higher current may be necessary for EDS up to 20  $\mu$ A.
  - d) The **SE Detect** selects the secondary electron detector. The lower detector is generally better for working distances (WD) greater than 10mm and gives a better sense of surface topography. The upper detector provides higher resolution and should generally be operated with a WD less than 10mm.
5. Press the **HV ON** button in the top left-hand corner of the console. This will turn on the accelerating voltage. It is normal to hear the valves opening.

(Note: Due to the nature of the field emission tip, it is not uncommon to have some beam instability at the beginning of the workday. Continued use will stabilize the beam. If the current drops at any time during a session, indicated by the flashing of the **HV ON/ADJ** light, press the **HV ON** button to bring it up again.)

6. Adjust the contrast and/or brightness using the up/down buttons until you can see some signal on the CRT. Auto Contrast and Brightness is operated by pressing the **ABC** button.

(Note: The CRT brightness and contrast is adjusted using the knobs located just under monitor B.)

7. Decrease the magnification using the magnification knob. For the lowest magnification, press the **LOW MAG** button to the right of the CRTs. Note: You will not be able to change any options in the PF menus while in the Low Mag mode.
8. The raster rates are adjusted using the buttons at the top of the console. The F (fast) TV rate is generally the best for navigation and alignment.
9. Locate your sample by first using the X/Y stage controls to move the edge of the sample holder then rotating the holder until you locate a sample stub.
10. Focus on the surface of the sample using the coarse and fine **FOCUS** knobs.
11. Set the **WD** by turning the focus knob and adjusting the stage Z-axis control. A shorter WD provides better resolution, but lower depth of focus. For EDS analysis, the system is calibrated for a 15mm WD – this is critical for quantification.
12. Increase the magnification (~15,000X) and find a small particle/feature to use for alignment.
13. Insert the desired objective aperture (the lower aperture set on the column) by turning the assembly clockwise. A smaller aperture will provide better resolution and depth of focus at the expense of signal strength.
14. Adjust the X/Y alignment knobs on the objective aperture to achieve the brightest image.

15. Press **PF3** and perform the following alignments (use the arrows to navigate and press return to activate/deactivate):
  - a) **#1 > BEAM ALIGN** – Align the bright spot (filament image) in the center of the screen using the **X/Y STIGMA/ALIGNMENT** knobs. You can use the magnification to increase the size of the spot.
  - b) **#2 > APERTURE ALIGN** - This will turn on the focus wobble. Use the **X/Y STIGMA/ALIGNMENT** knobs to adjust the alignment and eliminate translation of the image. It is easiest to adjust the X/Y knobs independently and eliminate translation in one direction at a time.
  - c) **#3 > STIGMA ALIGN X** - Use the **X/Y STIGMA/ALIGNMENT** knobs to eliminate translation of the image to align the X stigmator (similar to the aperture alignment).
  - d) **#4 > STIGMA ALIGN Y** – Repeat for the Y stigmator - the x and y knobs are reversed for this alignment.
16. Exit the alignment screen by pressing **PF16** and correct for astigmatism by adjusting the **X/Y STIGMA/ALIGNMENT** knobs. The objective here is to eliminate any stretching of the image you go through focus. The easiest way to achieve this is by focusing, adjust X stigmator, adjust Y stigmator and then repeat to achieve the sharpest image.
17. If high resolution imaging is required and vibration is present, flip the **SAMPLE** switch on the column control panel to the **LOCK** position. **CAUTION: DO NOT ATTEMPT TO MOVE THE Z AXIS OR THE TILT WHILE THE STAGE IS IN THE LOCKED POSITION!** Be sure to transfer to the **FREE** position before relocating the stage to the exchange positions at the end of the session.

### **Digital Imaging and EDS**

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. Press the **HV OFF** button in the top left-hand corner of the console.
2. Set the **SC AIR LOCK** switch on the column control panel to the **CLOSE** position.
3. If the stage lock was used, return it to the **FREE** position.
4. Move the stage to the exchange positions: **X=25, Y=25, Z=15, T=0, R=0**
5. Turn the chamberscope on by pressing the **CAMERA** button beside the CRT.
6. Open the **MV1** valve by turning the handle fully counterclockwise.
7. Insert the exchange rod and screw it into the sample holder.
8. Fully retract the exchange rod with sample holder.
9. Close the **MV1** valve.
10. Press the **AIR** button on the column control panel.
11. When the chamber vents, remove the sample, completely retract the exchange rod and close the door.
12. Hold the door firmly shut and press the **EVAC** button on the column control panel.
13. Turn off the chamberscope by pressing the **CAMERA** button.
14. Turn the **DISPLAY** switch on the column control panel to the **OFF** position.
15. Wait for the **S.C.** lights to blink green and for both the **S.C.** and **S.E.C.** vacuum lights to read **HIGH** before leaving the laboratory.
16. Clean up any mess you have made on the sample prep table and remove any carbon tape residue from the sample holder.
17. Log out in FOM and on the SEM log sheet.