

Operating Procedure for Hitachi S-4800 Scanning Electron Microscope

CFN

Laboratory 1L-32

C. T. Black

Operation of the Hitachi S-4800 Scanning Electron Microscope (SEM) requires specific user training and authorization. This operating procedure is meant as a general overview of tool operation and is NOT A SUBSTITUTE for obtaining proper training and authorization.

A list of authorized tool users is included with the ESR. If your name is not on this list then you are NOT AUTHORIZED to use the Hitachi S-4800 SEM.

Users should have reserved tool access via the appropriate SEM signup calendar.

NITRILE GLOVES ARE WORN AT ALL TIMES when handling sample specimens, sample stages, and any parts to be introduced into the tool vacuum chamber. Suitable gloves are located in the drawer of the workbench at the rear of the laboratory.

Sample Preparation

Suitable samples for imaging will be mounted on a solid flat substrates such as silicon or glass.

Sample specimens must be mounted on a sample holder for insertion into the tool. Several different sample holder sizes are available. Sample holders are located in the workbench at the rear of the laboratory.

Mount sample on sample holder using the workbench at the rear of the laboratory. Specimens should be mounted using either double-sided sticky tape or clips. Silver paint is to be used only if absolutely necessary.

Samples may be blown free of dust and debris using the nitrogen spray gun located at the laboratory workbench.

Clean and organize sample preparation area after finishing sample mounting.

Microscope Preparation

Observe that microscope is energized and ready for operation. If any warning lights indicate a problem then STOP and contact one of the laboratory administrators using the contact sheet posted on the laboratory door.

(optional) Fill the small dewar attached to the microscope with liquid nitrogen following the SOP for this procedure. See attached Liquid Nitrogen Fill Procedure. Wear all required Personal Protective Equipment when performing this operation – i.e., (1) safety glasses with side shields or safety goggles, (2) face shield, and (3) cryogenic gloves.

Loading a sample

After mounting sample on a sample holder, adjust the specimen height using the *specimen height gauge* located on the workbench. To adjust the height, loosen the lock screw and adjust the specimen height so that the height of the highest point of the specimen is the same as the bottom of the height gauge. Then tighten the lock screw by holding the fixed upper part of the holder..

Move to the SEM console. Verify that there is no sample stage already loaded in the chamber by looking at the small monitor showing a view inside the chamber.

Click the **EXC** button on the control panel. The stage inside the SEM will move to the *specimen exchange position*. Once this occurs the indicator beside the button will turn green.

Press the **AIR** button on the *specimen exchange chamber*. After 15sec. a buzzer will sound indicating that the chamber has been vented.

Open the exchange chamber door. Be **SURE** to use the handle to open the chamber door and **DO NOT USE THE SAMPLE LOAD ARM**. Pulling on the arm in this way can damage the sample lock vacuum. The exchange chamber door is interlocked and will not open unless all vacuum conditions are satisfied.

Confirm that the *specimen exchange rod* is in the **UNLOCKED** position by turning clockwise until the **UNLOCK** mark faces upwards

Insert the two spring pins at the end of the rod into holes of the specimen holder.

Turn the knob of the specimen exchange rod counterclockwise until the **LOCK** mark comes upwards. The sample holder is now locked onto the load arm.

Pull out the *sample exchange rod* completely and close the *exchange chamber door*.

Press the **OPEN** button on the *specimen exchange chamber*. This will pump out the loadlock and automatically open the gate valve to the chamber when a suitable low pressure has been achieved.

After the gate valve opens, slide the *specimen exchange rod* into the chamber **WHILE LOOKING INTO THE CHAMBER** on the small microscope screen to ensure that the specimen holder inserts into the guide rails.

Turn the specimen rod knob to the **UNLOCKED** position.

Pull out the *specimen exchange rod* completely while **VISUALLY WATCHING** that the rod disengages from the sample holder.

Press the **CLOSE** button on the specimen exchange chamber to close the gate valve. The sample is now loaded into the chamber.

Applying the SEM high voltage

WARNING: Operation of the microscope is authorized only after completion of user training. If you are not included on a list of authorized SEM users then **STOP** and obtain access authorization prior to use.

Set the size of the sample holder you are using in the **STAGE** menu. If you are using the cross-section stage then you should select a stage size of 4 inches.

High voltage can be applied only if the **HV DISPLAY** area on the control panel is blinking in yellow and blue. If the indicator is solid blue then the SEM conditions are not appropriate for high voltage application. Once the high voltage has been applied the indicator will display (nonblinking) yellow.

Use the **HV CONTROL** dialog window to set SEM accelerating voltage and emission current.
To set the accelerating voltage, select a voltage from the **Vacc** list box.
To set an emission current, set a current in the **SET Ie to** list box. Hitachi recommends 10uA emission current for normal SEM operation.

Click the **ON** button on the control panel at the left of the **HV DISPLAY** area. The SEM will automatically turn on the accelerating voltage, the extracting voltage, and the emission current. These values are displayed in the **HV DISPLAY** area. The *electron gun airlock valve* will automatically open upon completion of gun turn-on.

Once the high voltage is applied the **ON** button will change to **SET**, allowing the user to set the emission current during high voltage operation. The **OFF** button turns off the high voltage. Note that during SEM operation the current will drop gradually over time. You can reset to the specified emission current (typically 10uA) by pressing **SET**.

After high voltage turn-on a message will appear saying: “*Stage is at specimen exchange position. Move to Home position?*” Selecting **YES** will move the stage to the *Home position* such that the center of the sample holder will be directly underneath the electron beam.

Obtaining a sample image

There are many possible imaging variations for using the Hitachi S-4800 tool and these depend on the type of sample being imaged. We include here a summarized list of many commonly-used possibilities but do not provide detailed descriptions or an exhaustive list. The user is directed to the *tool operating manual* (located on the workbench at the rear of the laboratory) for a complete discussion.

We repeat here that use of this tool requires completion of specific training and proper authorization.

Imaging mode:

Selecting a Magnification Mode: The SEM has two magnification modes, **HIGH MAG** and **LOW MAG**. Most common applications will utilize the **HIGH MAG** mode.

Within **HIGH MAG** magnification mode, user specifies:

Probe current mode: **NORMAL** or **HIGH**. Most applications will utilize **NORMAL**

Focus mode: **UHR** or **HR**. **UHR** mode allows full range of working distances. Working distance (**WD**). Shorter **WD** means higher resolution. Longer **WD** allows greater depth of focus and more sample tilting.

Focus depth: Best resolution attainable with this value set to 1.0

Specimen bias voltage: Specimen can be voltage-biased during imaging to minimize sample charging effects.

Magnetic sample: Use this mode for observation of ferromagnetic samples

Degauss operation:

Setting **ABCC** link: Automatically adjusts brightness and contrast during sample imaging.

Column Alignment Operation: Obtaining the highest resolution image will involve aligning the *electron optical column*. This operation is done entirely through an *electromagnetic alignment* (i.e., using the computer interface) **without need for touching any knobs on the SEM column**. Alignment conditions are saved for each accelerating voltage and probe current setting such that only minimal alignment adjustment should be necessary.

Press **TV1** to obtain an image on the screen.

Typically the beam can be tuned up using only an **APERTURE ALIGNMENT**. The procedure is as follows:

Set the magnification to about 5,000 and position a point of interest in the center of the display

Focus the image and correct astigmatism

Click **APERTURE ALIGN** in the **ALIGNMENT** dialog box and make adjustments such that the wobbling motion of the image is minimized

Turn off the **APERTURE ALIGN** mode by clicking the **OFF** button

Sample imaging

Detector selection: The SEM has two secondary electron detectors, the **UPPER** and the **LOWER** detectors. User can select either detector or alternatively image using a mixture of the two signals. Select a detector on the **SIGNAL SELECT** block of the **OPERATION** panel.

Image Magnification: Select *image magnification* by dragging the mouse in the **MAGNIFICATION INDICATION** area on the **CONTROL PANEL**. The magnification can also be set by rotating the **MAGNIFICATION** knob on the control box.

Focus and astigmatism correction: Iteratively adjust *focus* and *astigmatism* using the knobs on the *SEM control box*.

Brightness and Contrast: Adjust *brightness* and *contrast* either manually by using **BRIGHTNESS** and **CONTRAST** knobs on SEM control panel or by pressing **ABCC** button for automatic control.

The recorded images are stored in a memory buffer and displayed across the screen bottom. At the end of the session you will need to save them in the appropriate folder.

Unloading sample

When finished imaging, turn off the high voltage by clicking **OFF** on the **HV DISPLAY** area.

Move the sample stage to the *exchange position* by clicking the **EXC** button on the *control panel*. Once this occurs the indicator beside the button will turn green.

Press the **OPEN** button on the *specimen exchange chamber* to open the gate valve

Turn the knob on the *specimen exchange rod* clockwise so that the **UNLOCK** mark is facing upwards

Insert the *specimen exchange rod* straight into the chamber and fit the two spring pins into the holes at the end of the specimen holder

Turn the knob counterclockwise so the **LOCK** mark comes upwards to hook on the sample holder.

VISUALLY INSPECT that the sample holder comes out with the rod as you pull the rod out of the chamber

Press the **AIR** button on the *specimen exchange chamber*. This closes the gate valve and vents the loadlock.

Open the *exchange door* and turn knob clockwise to **UNLOCK** position. The sample holder can now be disengaged from the sample rod.

Close specimen exchange chamber door and press **EVAC** to pump the loadlock back down.

Cleanup

Unmount your samples from the sample holder making sure to remove all sticky tape and residue from the sample sample mount. Tape may be removed using tweezers and residue using a small amount of isopropanol from the squeeze bottle.

Return sample stage to appropriate stage storage box located near the workbench at the rear of the laboratory

Clean workbench area and properly dispose of any materials used during sample preparation

If any chemical waste is generated during sample mounting and unmounting, dispose of it properly in the satellite area located in laboratory 1L-10.

TURN OFF laboratory lights when leaving the room