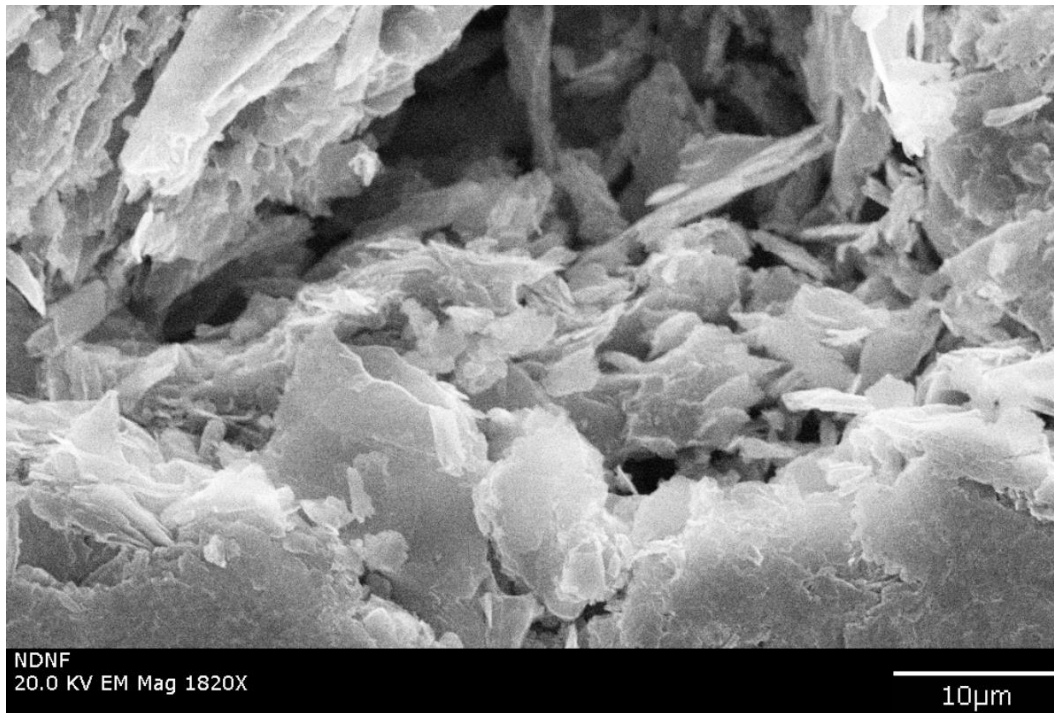


Operating Instructions

Hitachi S-4500 Field-emission SEM



University of Notre Dame

Department of Electrical Engineering

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General Precautions

Contacts

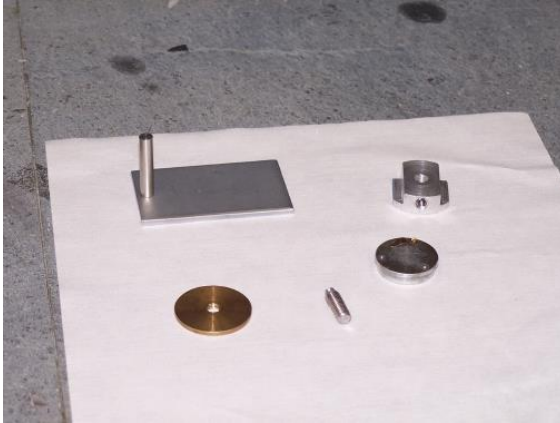
For problems, clarifications of procedures, or general information pertaining to this machine, please contact lab staff at staff-ndnf-list@nd.edu

Standby Conditions

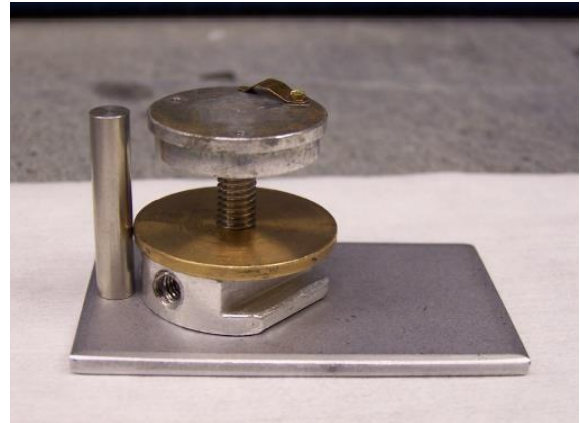
- DISPLAY POWER is off.
- OBJ. APERTURE is set to HEAT.
- EVAC POWER is on.
- S.C. VACUUM and S.E.C. VACUUM are both HIGH (green lights on the SEM panel).
- S.C. AIR LOCK VALVE is switched to CLOSE (two green lights blinking).
- S.E.C. EVAC button is lit.
- S.C. / S.E.C. switch is set to S.E.C.
- SPECIMEN STAGE is set to FREE position.
- MV-1 valve is completely closed (rotated clockwise and set to C).
- Camera is off (CAMERA button is “out” and is not lit).
- X-ray detector is backed out of the chamber to the “6” position.
- X-ray detector thermoelectric cooler reads room temperature.
- Backscatter detector is fully backed out of the chamber.
- X, Y, rotation & tilt axis are set to 0, Z set to ~18 mm (comply with the marks made on the nobs)

Sample preparation

1. Retrieve the following sample mount parts from the green toolbox which is set in the EBL resist prep hood. Place the parts on a clan wipe.
 1. HEIGHT REFERENCE STANDARD
 2. MOUNT BASE
 3. SCREW
 4. BRASS TIGHTENER
 5. STAGE



Height standard, tightener, screw, base, stage



How it looks like after assembly and compares to the height reference standard

2. Assemble the sample mount parts in the following order:
 1. Screw the BRASS TIGHTENER onto the bottom of the SCREW until about 1/8" of SCREW sticks out.
 2. Screw the bottom of the SCREW into the MOUNT BASE.
 3. Tighten BRASS TIGHTENER over the MOUNT BASE.
 4. Screw the STAGE onto the top of the SCREW.
3. Attach your sample to the STAGE using a double-side carbon tape or a stage clip.
4. Use the HEIGHT REFERENCE STANDARD to make the sample height working distance correspond with the SEM Z stage position.
 1. Make sure that the sample is not too tall for the entry chamber!!
 2. To adjust the sample height, loosen the BRASS TIGHTENER away from the MOUNT BASE, then **turn the STAGE and the SCREW together as needed** to correct the sample height, then retighten the BRASS TIGHTENER against the MOUNT BASE.

Sample loading

1. Verify that the SEM is in STANDBY CONDITIONS – see page 4.
2. Make sure the MV-1 valve is **truly closed**, and then vent the specimen exchange chamber (S.E.C.) by pressing the AIR button.
3. Open the S.E.C. by sliding it to the right, then carefully screw your mounted sample onto the threaded end of the LOADING ROD.
4. Completely extend the LOADING ROD outward (to the right). While holding the S.E.C. closed, evacuate the S.E.C. by pressing the EVAC button.
5. Wait until the S.E.C. VACUUM returns to HIGH (green HIGH LED will be lit).
6. Verify that the LOADING ROD is **completely extended** from the S.E.C.
7. Open the MV-1 valve by turning the handle counter-clockwise to the “O” position, and **slowly** approach the sample holder to the stage by pushing the LOADING ROD.
8. Watch through the view port of S.E.C. to slide the sample holder into the slot of stage. Verify that the holder is **truly engaged** on the stage!
9. While pushing **gently** on the rod, unscrew the LOADING ROD from the sample holder.
10. Withdraw the LOADING ROD from the S. C. and close the MV-1 valve.

Initialize the E-GUN

1. Verify that the S.C. VACUUM and S.E.C. VACUUM are both at HIGH (green LEDs), move the S.C. AIR LOCK VALVE switch to **OPEN**. You will hear the isolation valve open.
2. Verify that the IP3 vacuum level is better than **7.0×10^{-5} Pa**.
 - SELECT knob is set to IP3 and the RANGE (PA) knob is set to 10^{-6} Pa.
 - Read the pressure of IP3: if the vacuum level is above 7.0×10^{-5} Pa, set the S.C.AIRLOCK switch to CLOSE position, return the tool to STANDBY CONDITIONS, and contact Fei Cheng to report the problem.
3. Flash the tip only when it was not flashed within the last 8 hours.
 - Check the iLab SEM calendar, do the following if the tool is not used within the last 8 hours.
 - Press the **FLASH STANDBY** button. When the red light blinks, push the **ON** button. The flashing current (normally $\sim 30 \mu\text{A}$) will appear for about 1 second on the EMISSION display.
4. To select the accelerating voltage, press PF1. Input the desired voltage (usually 5 to 10 kV) and press PF16 to exit the screen.
 - For non-conducting samples, use 5 kV or lower.
 - For dose check with resists on samples, use 2 kV or lower.
5. To select a condenser lens and set the emission current, press PF2. Select 1 COND.LENSI, input a value (1-15, usually use 10), and press RETURN. Next, select 4 EMISSION, input a value (5-20 μA , usually use 10 μA), and press RETURN. Press PF16 to exit the screen.
6. Press the **ON** button to turn on the electron beam. The SEM will take ~ 1 minute to initialize. Once it has initialized, the V_{acc} , V_{ext} , and I_{EM} values are displayed on the keyboard.
7. Find your sample under low magnification:
 - Raise the Z axis from 20 mm (STANDBY CONDITION) to ~ 10 mm
 - Press the LOW MAG button on the right panel
 - Move X-Y axis to find easy identifiable features on your sample such as the edge or corners
 - Once the sample is located, depress LOW MAG button to go to high-mag mode

Maximize the imaging condition

1. Once you located the sample and switched back to high-mag mode, you need to set up the beam correctly in order to obtain a clear image of the sample. This is an iterative procedure involving the following processes.
2. Press the ABC button, and then wait a few seconds. The SEM will attempt to automatically adjust brightness and contrast to usable values.
 - a. This may be repeated as needed, as you continue to adjust and align the SEM.
 - b. You can also adjust manually with the CONTRAST and BRIGHTNESS up and down buttons, but pressing ABC button is recommended for users.
3. Using the COARSE FOCUS control, bring an image of your sample into focus.
4. Move the X-Y axis to find the area you are interested in.
5. To zoom in / zoom out on a feature, rotate the MAGNIFICATION knob clockwise or counter-clockwise.
6. Re-focus the beam using the COARSE and/or FINE FOCUS knobs.
7. Stigmatize the beam using the STIGMA/ALIGNMENT X and Y knobs.
8. Align the beam:
 - a. Press the PF3 key.
 - b. Select 1 BEAM ALIGN (using ↑ and ↓ keys and pressing ENTER)
 - c. A round white circle appears on screen A. **Center the circle** using the STIGMA/ALIGNMENT X and Y knobs. You may alter the size of the circle by adjusting the MAGNIFICATION control.
 - d. Press RETURN to return to the PF3 menu.
9. Align the aperture:
 - a. Select 2 APERT.ALIGN.
 - b. Minimize imaging wobbling using the STIGMA/ALIGNMENT X and Y knobs
 - c. Press RETURN to return to the PF3 menu or PF16 to exit.
10. Align the stigmators:
 - a. Select 3 STIG ALIGN X.
 - b. Minimize imaging wobbling using the STIGMA/ALIGNMENT X and Y knobs.

- c. Press RETURN to the PF3, and do the same thing for STIG ALIGN Y.
Note: in this case, the X control adjusts motion in Y, and the Y control adjusts motion in X.
 - d. Press RETURN to return to the PF3 menu.
 - e. Press PF16 to exit.
11. Re-focus on the interested area using the COARSE and/or FINE FOCUS knobs.
 12. You may need to repeat the step 7 – 11 when you zoom in to higher mag. to obtain good images. Please follow APPENDIX I for more imaging tips.

Obtain the images of your sample

1. Locate the area of interest of your sample using the stage control X and Y on the left column.
 - a. Always check if the readings are at the standby condition (X, Y, Tilt and rotation are all set to 0, and Z set to 20 mm) before you start.
 - b. Always make small adjustments along X / Y axis or you may lose your target easily especially at high-mag.
2. Capture the image using the Quartz-PCI software on the PC to the right of the SEM console. See Appendix II for details.

Shut off the E-gun

1. Press the **OFF/READY** button to turn off the beam when you have finished imaging your sample.
2. IMPORTANT: Move the S.C. AIR LOCK VALVE switch to the **CLOSE** position. You will hear the column isolation valve close.
3. Switch off the DISPLAY POWER.

Unload your sample

1. Verify that the SPECIMEN STAGE switch is in the FREE position.
2. Make sure the LOADING ROD is completely extended, and open the MV-1 valve.
3. Extend the rod into the chamber while watching through the view port. **Completely and gently** screw the LOADING ROD into the sample mount.
4. **Slowly, carefully, and fully** retract your mounted sample into the S.E.C.
5. Securely close the MV-1 valve.
6. Press the AIR button to vent the S.E.C.
7. When the S.E.C. reaches atmosphere, open the S.E.C. and unscrew your mounted sample from the LOADING ROD.
8. While holding the S.E.C. closed and making sure the LOADING ROD is completely extended, evacuate the S.E.C. by pressing the EVAC button.
9. Verify all STANDBY CONDITIONS (see page 4).

Retrieve your sample from the sample mount

1. **Carefully** remove your sample from the sample mount STAGE using tweezers and/or a razor blade if needed.
2. Disassemble the sample mount.
3. Place the sample mount components back into the appropriate bins of the green toolbox.
4. Clean and organize the SEM workstation. Collect your belongings before you leave.
5. Logout from iLab.

APPENDIX I

Imaging theory & tips

Focus – Just as light is focused to a point by a series of lenses, an electron beam is focused to a point by a series of magnetic fields. When your sample is in the same plane as the convergence point in the focal plane, your image has the greatest focus. Adjust the COARSE and FINE FOCUS knobs to vary the final magnetic field through which the electron beam travels, thus aligning the focal plane to the plane of your sample.

Depth of Focus – Planes just above and below the focal plane can also be in relative focus, depending on how narrow the electron beam is. The distance above and below the focal plane in which your sample is in focus is called the depth of focus. A smaller final aperture will give a better depth of focus, for a fixed working distance.

Astigmatism – In order to achieve a clear sample image, the electron beam hitting your sample needs to be as circular as possible. When the beam is astigmatic, the beam is more elliptical than circular, which creates a skewed or blurred image on the screen. Stigmatize the electron beam by adjusting the STIGMA/ALIGNMENT X and Y knobs. These adjustments will affect focus to some degree, so going back and forth between the stigmators and the fine focus might be a good idea. As with defocus, astigmatism is more noticeable at higher magnifications.

Electron Beam Power – The power of the beam is the probe current multiplied by the accelerating voltage, V_{acc} . As beam power increases, the signal-to-noise ratio (SNR) of your image generally increases, making your image less noisy. A larger final aperture will result in a larger probe current, for a fixed accelerating voltage and condenser lens setting. High beam power combined with high magnification can result in sample heating effects in some samples, which can modify (destroy?) delicate structures.

Restoring Emission Current – Emission current varies over time. This is due to the emission characteristics of the cold-cathode field emitter. Shortly after flashing the cathode, the current may drop substantially over an imaging session, dimming the screen image. To restore the emission current to the original setting, simply press the ON button again.

Sample Charging – Materials or structures that have not been grounded or are not conductive may exist on your sample. Heavy charging may either make your sample extremely bright or extremely dark, depending on whether it is positively or negatively charged. There are a few techniques to reduce charging. Carbon tape or paste may be used to ground your sample. Depositing a thin layer of metal, such as chromium or gold, over your sample will make your sample conductive. If these techniques either do not work or cannot be used, lowering V_{acc} , the probe current, and/or the emission current may help reduce charging as well.

Condenser Lens 1 Size – The first condenser lens has sixteen sizes to choose from: 0 to 15, 0 being the largest and 15 being the smallest. The smaller lenses produce a narrower electron beam and therefore a smaller spot size, allowing for sharper images. The larger lenses cause more primary electrons from the electron beam to hit your sample, thus increasing the SNR ratio of your image (see “electron beam power” above).

Brightness/Contrast Adjustment – If your screen image is too bright or dark, press the ABC button located above the BRIGHTNESS and CONTRAST buttons on the SEM control panel to automatically readjust the image brightness/contrast. To manually adjust the image brightness/contrast, use the up/down BRIGHTNESS and CONTRAST buttons.

Scan Speeds – The slower your scan speed, the less noisy will be your image. However, you may find it useful to use the slower scan speeds when viewing an image or adjusting the beam conditions. Experiment with the four SCAN SPEED buttons to determine their usefulness. You may find the fourth SCAN SPEED button (represented by a small rectangle) useful when stigmating and focusing the beam, since it scans the beam on a reduced area, making the image appear less noisy.

Sample Tilt – Tilting your sample to one side may allow you to obtain a cross-sectional image of your sample. Due to the limited range of the tilt stage, you may need to mount your sample to the 45-degree or 90-degree sample mounts to obtain a cross-sectional (“on-edge”, or 90 degree) image. Take extra precautions when using the TILT up/down buttons:

- Ensure the SPECIMEN STAGE is **NOT** in the LOCK position.

- Watch the camera image on the monitor above the console, in order to prevent your mounted sample from hitting the POLE PIECE or any other object (detector, etc.) in the chamber.
- Make sure your sample is well attached to the sample mount so that it does not fall off inside the specimen chamber.

Image Shifts Knobs – The X and Y IMAGE SHIFT knobs allow for easy image shifting at high magnifications when the manual X and Y stage drives are too coarse for practical use. The image shift is created by magnetic deflection of the beam rather than by movement of the sample. Therefore, heavy use of the IMAGE SHIFT knobs may skew and/or defocus the electron beam. Press the X/Y RESET button before aligning/adjusting the electron beam for best image results.

Appendix II:

Abbreviated Instructions for Image Capture using Quartz-PCI Passive Capture System

1. Image the area you wish to photograph on the SEM display (as per usual: align beam, focus, stigmat, etc.)
2. On the PC: launch “Quartz PCI Version 8” (icon on desktop)
3. Select “Acquire → Image Size...”, and choose small, medium, large, extra-large, or custom.
4. Select “Acquire → Single”. A blank image window will open.
5. On the SEM console: press the “PHOTO MEMORY” button. The red LED next to the button should begin to flash.
6. Now, wait for the SEM to capture an image and transfer it to the PC. You should eventually see the image window fill with the desired image, from the top down.
7. Once the capture finishes, the SEM image channel A has stopped, as indicated by the unlit LED next to the “A” button. Now restart channel A by clicking on the “A” button; its LED will illuminate. The SEM is now back to its normal operating mode.
8. On the PC: to save the image, go to “File → save as...”.
 - a. At Storage Volume, if your netid is not already selected, click on “Browse” and select your netid. If your netid is not listed:
 - i. Click on “New”
 - ii. Enter your netid as the Volume ID
 - iii. Use Browse button to direct to the following path:
C:\Documents and Settings\All Users\Documents\Users\<your netid> (note: you might have to create this folder under Users, using the new folder button)
 - iv. Click OK, probably twice.
 - v. **Uncheck** the “Make Volume Network Accessible” button.
 - vi. Click OK, then click OK again.
 - b. Enter the File Name you want
 - c. Select the image format (jpg, tiff, bmp) you desire
 - d. Click OK.
9. Once you have saved the first image, you should only have to fill in the file name for subsequent images.
10. Please be aware that we DO NOT back up the computer’s hard drive. If the image file is important to you, COPY IT SOMEWHERE ELSE.