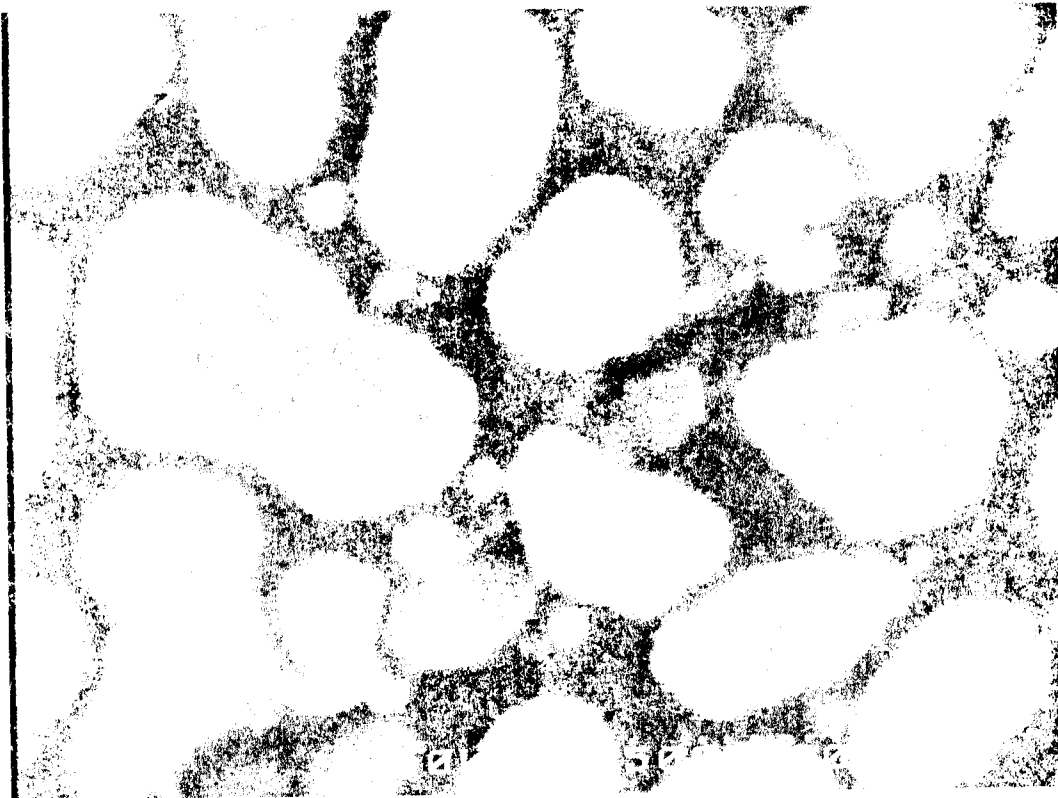


OPERATIONAL PROCEDURES MANUAL

*Dont
take out
of file*

FOR THE HITACHI S-4500 SEM

(revised 5/21/2007)



UNIVERSITY OF NOTRE DAME
DEPARTMENT OF ELECTRICAL ENGINEERING

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GENERAL ATTENTION

As of 5/21/2007, any SEM user who has not been trained to use the nitrogen purge system is no longer authorized to use the SEM. The nitrogen purging process has been added to the operational procedures in order to enhance image quality at high magnifications and reduce sample corrosion caused by the electron beam.

Heating the **OBJ. APERTURE** for 20 minutes before imaging is required to prevent the objective aperture from corroding prematurely. If the aperture is not heated properly before each SEM session, the aperture shape may alter, due to corrosion, leading to a misshapen electron beam and poor imaging quality. Due to this added waiting period, the time limit of SEM sessions between 8am and 8pm, Monday thru Friday, has been extended to two and a half hours.

Flashing the gun, as described in section VI of the EXPANDED SEM PROCEDURES, is required to prevent cathode corrosion—see **Figures 1A – 1C** or pages 2 – 4 of the Hitachi Manual for more detail.

All users must reserve the SEM at <http://localendar.com/> before each SEM session.

Gloves are required when touching any parts that may go into the SEM specimen chamber (**S.C.**). Use gloves when loading and unloading your sample. Do not use gloves that have been contaminated by the SEM keyboards, knobs, switches, or anything else that may have been touched with bare hands.

Some SEM switches have lock features that prevent accidental switching, such as the switch for **DISPLAY POWER**. To move these switches, pull the switch's spring-lock jacket outward along the switch's handle, move the switch to the desired position, and gently allow the spring-lock jacket to go back in place. Never force a switch to move.

Never adjust the **Z** or **ROTATION** knobs when the **SPECIMEN STAGE** is set to **LOCK**.

Unless authorized by Dr. Bernstein, do not store personal items in room B13 or remove any SEM equipment or paperwork from B13.

Downloading software on the SEM PC is prohibited. Only image files created by the SEMICAPS software may be saved on the SEM PC. However, these images may be purged during periodic maintenances with or without notice.

All SEM users are required to keep the SEM workstations clean and orderly.

If you experience technical difficulties while using the SEM, contact Dr. Bernstein (bernstein.1@nd.edu, 1-6269) for assistance. If the SEM malfunctions, and if it is safe to do so, return the SEM to STANDBY CONDITIONS and immediately contact Dr. Bernstein.

This manual and other SEM documentation can be found at <http://www.nd.edu/~FESEM>.

STANDBY CONDITIONS

- **DISPLAY POWER** is off (switched downward)
- **OBJ. APERTURE** is set to **HEAT**.
- **EVAC POWER** is on (switched upward)
- **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
- Stage-positioning knobs (**X**, **Y**, **Z**, **ROTATION** and **TILT**) are at default loading positions
- **MV-1** valve is completely closed (rotated clockwise and set to **C**)
- Camera is off (**CAMERA** button not lit)

BASIC SEM PROCEDURES

- I. Reserve the SEM at <http://localendar.com/>.
- II. Sign in on the log sheet.
- III. Stop the nitrogen purging system (returning the SEM to STANDBY CONDITIONS).
- IV. Mount your sample onto a sample mount.
- V. Load your mounted sample onto the **S.C.** specimen stage.
- VI. Initialize the electron gun.
- VII. Maximize the imaging conditions.
- VIII. Image your sample.
- IX. Shut off the electron gun.
- X. Sign out on the log sheet.
- XI. Unload your sample.
- XII. Return the SEM to STANDBY CONDITIONS.
- XIII. Start the nitrogen purge.
- XIV. Retrieve your sample from the sample mount STAGE.
- XV. Clean and organize the SEM workstations.

EXPANDED SEM PROCEDURES

I. RESERVE THE SEM

- a) Go to <http://localendar.com/>.
- b) Log in using the username ***ghbsem1*** and the password ***hitachi***.
- c) Under the ***Standard*** tab, select ***Add Event***.
- d) Type your name in the ***Title*** bar.
- e) Select the appropriate date, start time and duration.
- f) Click the ***Save*** button.
- g) Verify that your reservation is correct on the calendar.
- h) Click ***Sign off***.

II. SIGN IN ON THE LOG SHEET.

Fill in your name, the date and start time on the log sheet.

III. STOP THE NITROGEN PURGING SYSTEM.

- a) Push in both the ***TURBO POWER ON/OFF*** and the ***TURBO MOTOR START/STOP*** buttons. (The red LEDs will remain lit.)
-
- b) When the ***S.C. VACUUM*** reaches ***LOW*** (in approximately 10 minutes):
 - i) Close the ***MV-1*** valve.
 - ii) Move the ***S.C./S.E.C.*** switch to ***S.E.C.***
 - iii) Turn the ***OBJ. APERTURE*** switch from ***OFF*** to ***HEAT***.
 - iv) **IMPORTANT**: Wait 20 minutes for the ***OBJ. APERTURE*** to heat before turning on the electron gun.

IV. MOUNT YOUR SAMPLE.

- a) Put on latex gloves.

- b) If the lab wipe in front of the blue toolbox is dirty or missing, replace it with a new, clean lab wipe.
- c) Retrieve the following sample mount parts from the blue toolbox—see **Figures 2A** and **2B**:
 - i) **MOUNT BASE**
 - ii) **SCREW**
 - iii) **BRASS TIGHTENER**
 - iv) **STAGE**
- d) Place the parts on the clean lab wipe.
- e) Assemble the sample mount parts on the lab wipe in the following order:
 - i) Screw the **BRASS TIGHTENER** onto the bottom of the **SCREW** until about 1/8" of **SCREW** sticks out.
 - ii) Screw the bottom of the **SCREW** into the **MOUNT BASE**.
 - iii) Tighten **BRASS TIGHTENER** over the **MOUNT BASE**.
 - iv) Screw the **STAGE** onto the top of the **SCREW**.
- f) Attach the sample you wish to image to the **STAGE** using carbon tape or a stage clip.
- g) Use the **HEIGHT REFERENCE STANDARD** to make the sample height working distance correspond with the SEM **Z** knob, if desired.
- h) Place the mounted sample on another clean lab wipe in front of the **S.E.C.**

V. LOAD YOUR SAMPLE.

- a) Verify that the SEM is in STANDBY CONDITIONS.
- b) Turn on **DISPLAY POWER** (switch upward) and allow the monitors to warm up.
- c) Turn the camera on. (The **CAMERA** button will light up, and the camera view screen will appear on SEM screen **A**.)
- d) Making sure the **MV-I** valve is closed, vent the specimen exchange chamber (**S.E.C.**) by pressing the **AIR** button.
- e) While still wearing gloves, screw your mounted sample onto the **LOADING ROD**.
- f) Completely extend the **LOADING ROD** outward. While holding the **S.E.C.** closed, evacuate the **S.E.C.** by pressing the **EVAC** button. (Be aware that the **LOADING ROD** may be pushed in by atmospheric pressure as the **S.E.C.** reaches vacuum.)

- g) When the **S.E.C. VACUUM** returns to **HIGH**, verify that the **LOADING ROD** is completely extended from the **S.E.C.** Then open the **MV-1** valve by turning the handle counter-clockwise to the **O** position.
- h) Transfer your sample to the specimen chamber (**S.C.**) stage using the **LOADING ROD**.
- i) While pushing gently on the rod, unscrew the **LOADING ROD** from your mounted sample.
- j) Withdraw the **LOADING ROD** from the **S.C.** and close the **MV-1** valve.
- k) Remove and discard your gloves.

→ VI. INITIALIZE THE ELECTRON GUN.

- a) Verifying that both the **S.C. VACUUM** and **S.E.C. VACUUM** are at **HIGH**, move the **S.C. AIR LOCK VALVE** to **OPEN**.
- b) Record the **ION PUMP VACUUM** level on the log sheet.
 - Make sure the **SELECT** knob is set to **IP3** and the **RANGE (PA)** knob is set to 10^{-6} .
 - Important: If the IP3 vacuum level is ever above $7 \cdot 10^{-4}$ Pa, close the **S.C. AIR LOCK VALVE**, return the SEM to STANDBY CONDITIONS, and notify ~~Dr. Mike Young~~ *Dr. Mike Young* ~~Bernstein~~ immediately.
- c) Check the log sheet for the last flash time. If the gun has been flashed within the last eight hours, go to the next step. Otherwise, flash the gun by pushing the **FLASH STANDBY** button. When the red light blinks, push the **ON** button. The flashing current will show for about one second on the **EMISSION** display. Record its value.
- d) To select the accelerating voltage, press **PF1**. Input the desired voltage and press **PF16** to exit the screen.
- e) To select a condenser lens and set the emission current, press **PF2**. Select **1 COND.LENS1**, input a value (0-15), and press **RETURN**. Next, select **4 EMISSION**, input a value (5-20 μ A), and press **RETURN**. Press **PF16** to exit the screen.
- f) Press the **ON** button again to turn on the electron beam. The SEM will take about a minute to initialize. Once it has initialized, log the **Vacc** and **Vext** values.

VII. MAXIMIZE THE IMAGING CONDITIONS.

- a) Adjust the working distance by doing the following:

- i) Turn on the camera by pressing the **CAMERA** button. The **CAMERA** button will light and the camera's image will appear on screen **A**.
 - ii) Verify that the **SPECIMEN STAGE** switch is in the **FREE** position.
 - iii) Adjust the **Z** knob while simultaneously watching screen **A**, taking care not to let your sample hit the **POLE PIECE**. (The **POLE PIECE** is the metal housing of the objective lens located toward the top of camera image on screen **A**—see **Figure 3**.)
- b) Align/adjust the electron beam in order to obtain a clear image of your sample. This is an iterative procedure involving the following processes:
 - i) Move to the area you wish to focus on by using the **X** and **Y** knobs. (At high magnifications, use the **X** and **Y IMAGE SHIFT** knobs for more precise image movement.)
 - ii) Increase the magnification by rotating the **MAGNIFICATION** knob clockwise.
 - iii) Focus the beam using the **COARSE** and/or **FINE FOCUS** knobs.
 - iv) Stigmatize the beam using the **STIGMA/ALIGNMENT X** and **Y** knobs.
 - v) Align the beam:
 - Press **PF3**.
 - Select **1 BEAM ALIGN** (using the up and down arrow keys and pressing **RETURN**).
 - Center the beam in the screen using the **STIGMA/ALIGNMENT X** and **Y** knobs.
 - Press **RETURN** to return to the **PF3** menu or **PF16** to exit.
 - vi) Align the aperture:
 - Press **PF3**.
 - Select **2 APERT.ALIGN**.
 - Minimize imaging wobbling using the **STIGMA/ALIGNMENT X** and **Y** knobs.
 - Press **RETURN** to return to the **PF3** menu or **PF16** to exit.

(See section II of the APPENDIX for more imaging tips.)

VIII. IMAGE YOUR SAMPLE.

- a) Locate your sample using the **X** and **Y** knobs. (At high magnifications, use the **X** and **Y IMAGE SHIFT** knobs for more precise image movement.)
- b) Capture the image using SEMICAPS software—see sections IIIA and IIIB of the APPENDIX for software instructions.

IX. SHUT OFF THE ELECTRON GUN.

- a) Press the **OFF/READY** button to turn off the beam when you have finished imaging your sample.
- b) IMPORTANT: Close the **S.C. AIR LOCK VALVE**.

X. SIGN OUT ON THE LOG SHEET.

- a) Log your stop time and hours.
- b) Place the log sheet in the front folder of the 3-ring binder containing this operational procedures manual.

XI. UNLOAD YOUR SAMPLE.

- a) Turn the camera on.
- b) Verify that the *SPECIMEN STAGE* switch is in the *FREE* position.
- c) Turn the *X, Y, Z, ROTATION* and *TILT* knobs back to their default loading positions.
- d) Make sure the *LOADING ROD* is completely extended, and open the *MV-1* valve.
- e) Completely screw the *LOADING ROD* into the sample mount.
- f) Fully retract your mounted sample into the *S.E.C.*
- g) Close the *MV-1* valve.
- h) Press the *AIR* button to vent the *S.E.C.*
- i) Put on a new pair of gloves.
- j) When the *S.E.C.* reaches atmosphere, open the *S.E.C.* and unscrew your mounted sample from the *LOADING ROD*.
- k) 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.

XII. RETURN THE SEM TO STANDBY CONDITIONS.

- a) Turn off *DISPLAY POWER*. *Camera off*
- b) Verify all STANDBY CONDITIONS.

XIII. BEGIN THE NITROGEN PURGE.

- a) Verify that the nitrogen tank reads ~4 psi.

- b) Turn the **OBJ. APERTURE** switch from **HEAT** to **OFF**. (Warning: Do not switch to **DEGAS**.)
- c) Verify that the **S.C. VACUUM** and **S.E.C. VACUUM** are at **HIGH**.
- d) Open the **MV-I** valve.
- e) Move the **S.C./S.E.C.** switch to **S.C.**
- f) Push the **TURBO POWER ON/OFF** and the **TURBO MOTOR START/STOP** buttons. (The buttons will spring outward, but the red LEDs will remain lit.)

(Note: The nitrogen purge will begin when the turbo pump shuts off in 15 to 20 minutes.)

XIV. RETRIEVE YOUR SAMPLE FROM THE SAMPLE MOUNT.

Remove your sample from the sample mount STAGE using tweezers and/or a razor.

XV. CLEAN AND ORGANIZE THE SEM WORKSTATIONS.

- a) Disassemble the sample mount.
- b) Place the sample mount components back into the appropriate bins of the blue toolbox.
- c) Place any tools used to retrieve your sample into the appropriate bins of the blue toolbox.
- d) Close the toolbox.
- e) Dispose of any superfluous lab wipes, aluminum foil pieces, loose papers, etc.
- f) Verify that the 3-ring binder containing the log sheet and the operational procedures manual is placed to the right of the SEM control panel.
- g) Remove and discard your gloves.

II. IMAGING THEORY AND RECOMMENDATIONS

- a) 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.
- b) 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.
- c) Working Distance – The greater your working distance is, the greater your depth of focus is. However, smaller working distances generally give you clearer images in the focus plane. Adjust the working distance of your sample using the **Z** knob. For fairly flat surfaces, choose a relatively small working distance. For samples with more topography, choose a larger working distance. See **Figure 4** for the SEM focusing limits with respect to working distance and accelerating voltage, **V_{acc}**.
- d) 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 astigmatized, 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. As with defocus, astigmatism is more noticeable at higher magnifications.
- e) Electron Beam Power – The power of the beam is the emission 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.
 - i) Restoring Emission Current – Emission current varies over time. This is due to molecules collecting on the cathode tip, changing its resistance—see **Figures 1A – 1C**. 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, press the **ON** button again.
 - ii) Sample Charging – Samples that have not been grounded or are not conductive may experience charging. Mild charging may appear as fairly drastic brightness changes 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 chrome, over your sample will make your sample conductive. If these techniques either do not work or cannot be used, lowering **V_{acc}** and/or the emission current may help reduce charging as well.
- f) Cond.Lens1 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, allowing for sharper images. The larger lenses allow more primary

electrons from the electron beam to hit your sample, thus increasing the signal-to-noise ratio of your image.

- g) Stage Lock – Locking the stage reduces image noise due to vibrations in the room. Move the **SPECIMEN STAGE** switch from **FREE** to **LOCK** to lock the stage. You will need to refocus and readjust the **X** and **Y** knobs after doing this. **WARNING:** Adjusting the **Z** or **TILT** knobs while the stage is locked may damage the SEM!
- h) 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** arrows.
- i) Scan Speeds – The slower your scan speed is, the less noisy your image will be. The SEMICAPS software automatically sets the scan speed when capturing an 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 concentrates the beam on a smaller area, making the image less noisy.
- j) Image Rotation – If your image is rotated at an undesired angle on the screen, you can adjust it either physically or electronically. Physically rotate your sample using the **ROTATE** knob—see **Figure 5A**. Electronically rotate the screen image by setting the **RASTER ROTATION** switch to **ON** and setting the **RASTER ROTATION** knob to the desired angle—see **Figure 5B**.
- k) Sample Tilt – Tilting your sample to one side may allow you to obtain a cross-sectional image of your sample. Due to the **TILT** knob's limited range, you may need to mount your sample to the 45-degree or 90-degree sample mounts to obtain a cross-sectional image. Take extra precautions when using the **TILT** knob:
 - i) Make sure the **SPECIMEN STAGE** is not in the **LOCK** position.
 - ii) Watch the camera image on screen **A** in order to prevent your mounted sample from hitting the **POLE PIECE**.
 - iii) Make sure your sample is well attached to the sample mount with carbon tape or a stage clip.
 - iv) Don't forget to return the **TILT** knob back to 0 degrees before retrieving your sample from the **S.C.**
- l) Image Shifts Knobs – The **X** and **Y IMAGE SHIFT** knobs allow for easy image-shifting at high magnifications when the manual **X** and **Y** knobs 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.

IIIA. SEMICAPS 2000P INSTRUCTIONS

- a) Double-click the *shortcut to S2kP.exe* icon on the PC desktop.
- b) Verify that *PhotoSpeed* is set to **40**, *Resolution* is set to **Full**, and *Continuous Capture* is not checked.
- c) Using the mouse, press the **Snap** button in the right of the SEMICAPS windows or select **Snap** in the Capture menu.
- d) Move your sample to the desired position on the SEM monitor and press **DIRECT** in the **PHOTO** area on the SEM keyboard. An image on screen **A** and in the SEMICAPS window should immediately appear, starting from the top and finishing in about 10 to 15 seconds.
- e) When the **Image Data Edit** window pops up over your image, fill in the **Mag:** and **Acc Voltage** values, and press the **OK** button. (If necessary, you can change these values by selecting **Edit** in the Data menu.)
- f) Save your image WITHOUT measurements, micron marker or annotation by selecting **Export** under the File menu. Verify that **Micron Bar**, **Measurement**, and **Annotation** are not checked, and save your image to your personal user folder.
- g) Optional image modifications:
 - i) Process the image by selecting various options in the Process menu, such as **Noise Reduction**, **Sharpen...**, **Auto Brightness Contrast...**, etc.
 - ii) Take measurements of your image by selecting the **Measure** function in the toolbar or under the Tools menu. (Measurement properties, such as line width, color and font size, can be modified by selecting **Measure Setting...** in the Tools menu.)
 - iii) Add a micron marker by checking **Micron Marker** in the **Image Data** section to the right of your image or by selecting **Micron** in the Data menu.
 - iv) Add annotation by selecting **Annotate...** in the Tools menu.
- h) Save your image WITH measurements, micron marker and/or annotation by selecting **Export** under the File menu. Place a check next to the modifications you wish to include on your image, and save your image in your user folder. (Note: Once you have exported an image with measurements, micron marker and/or annotation, they cannot be erased from your image. Also, annotation will not be saved if not checked when first exported.)
- i) Repeat the process from step (c) to capture another image.
- j) Exit out of the software when you are finished.
- k) Use a thumb-drive or <https://webfile.nd.edu> to backup any images you wish to permanently save.

IIIB. SEMICAPS 2000A INSTRUCTIONS

- a) Double-click the **SEMICAPS 2000A** icon on the PC desktop.
- b) Select the desired image size (512x512, 1024x1024, etc.) under **Resolution**.
- c) Select the desired average/integration method under **Method**.
- d) Select the number of samples per averaging step (1, 2, 4... 4096) under **Sample**. (Use large values to reduce image noise.)
- e) Using the mouse, press the **Snap** button in the right of the SEMICAPS windows or select **Snap** in the **Capture** menu. An image should immediately appear in the SEMICAPS window, starting from the top. (Note: Capture speed depends highly on **Resolution** and **Sample** sizes.)
- f) When the **Image Data Edit** window pops up over your image, fill in the **Mag:** and **Acc Voltage** values, and press the **OK** button. (If necessary, you can change these values by selecting **Edit** in the **Data** menu.)
- g) Save your image WITHOUT measurements, micron marker or annotation by selecting **Export** under the **File** menu. Verify that **Micron Bar**, **Measurement**, and **Annotation** are not checked, and save your image to your personal user folder.
- h) Optional image modifications:
 - i) Process the image by selecting various options in the **Process** menu, such as **Noise Reduction**, **Sharpen...**, **Auto Brightness Contrast...**, etc.
 - ii) Take measurements of your image by selecting the **Measure** function in the toolbar or under the **Tools** menu. (Measurement properties, such as line width, color and font size, can be modified by selecting **Measure Setting...** in the **Tools** menu.)
 - iii) Add a micron marker by checking **Micron Marker** in the **Image Data** section to the right of your image or by selecting **Micron** in the **Data** menu.
 - iv) Add annotation by selecting **Annotate...** in the **Tools** menu.
- i) Save your image WITH measurements, micron marker and/or annotation by selecting **Export** under the **File** menu. Place a check next to the modifications you wish to include on your image, and save your image in your user folder. (Note: Once you have exported an image with measurements, micron marker and/or annotation, they cannot be erased from your image. Also, annotation will not be saved if not checked when first exported.)
- j) Repeat the process from step (b) to capture another image.
- k) Exit out of the software when you are finished.
- l) Use a thumb-drive or <https://webfile.nd.edu> to backup any images you wish to permanently save.

APPENDICES

I. FIGURES

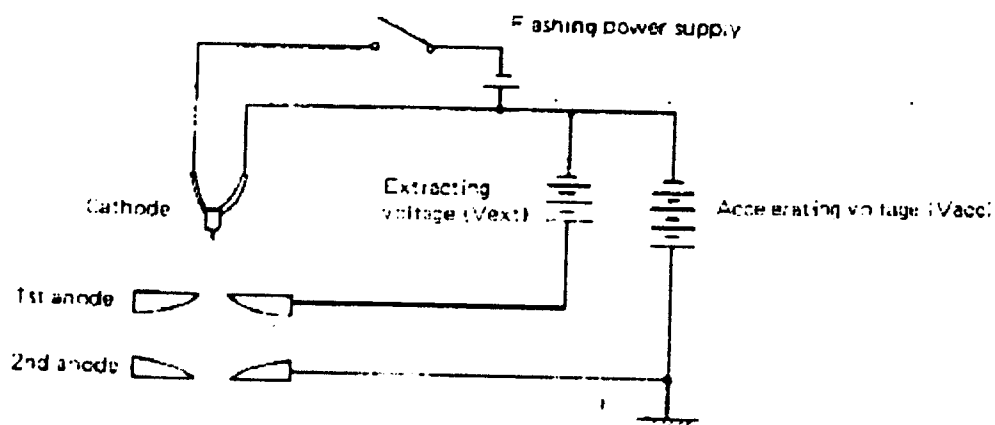


Figure 1A: Flash Schematic

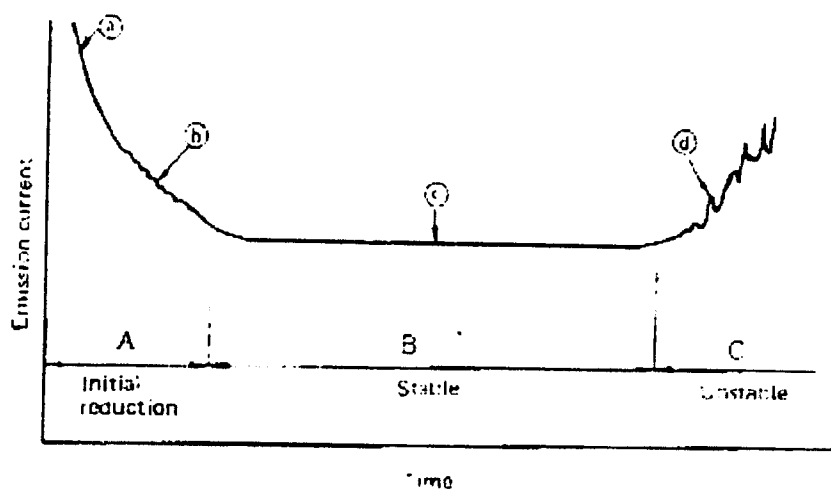


Figure 1B: Emission Current After Flashing

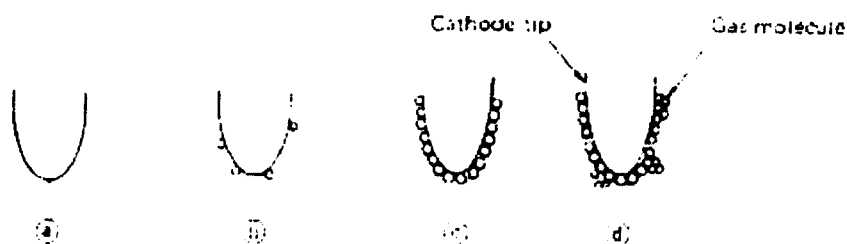


Figure 1C: Particle Collection on Cathode Tip

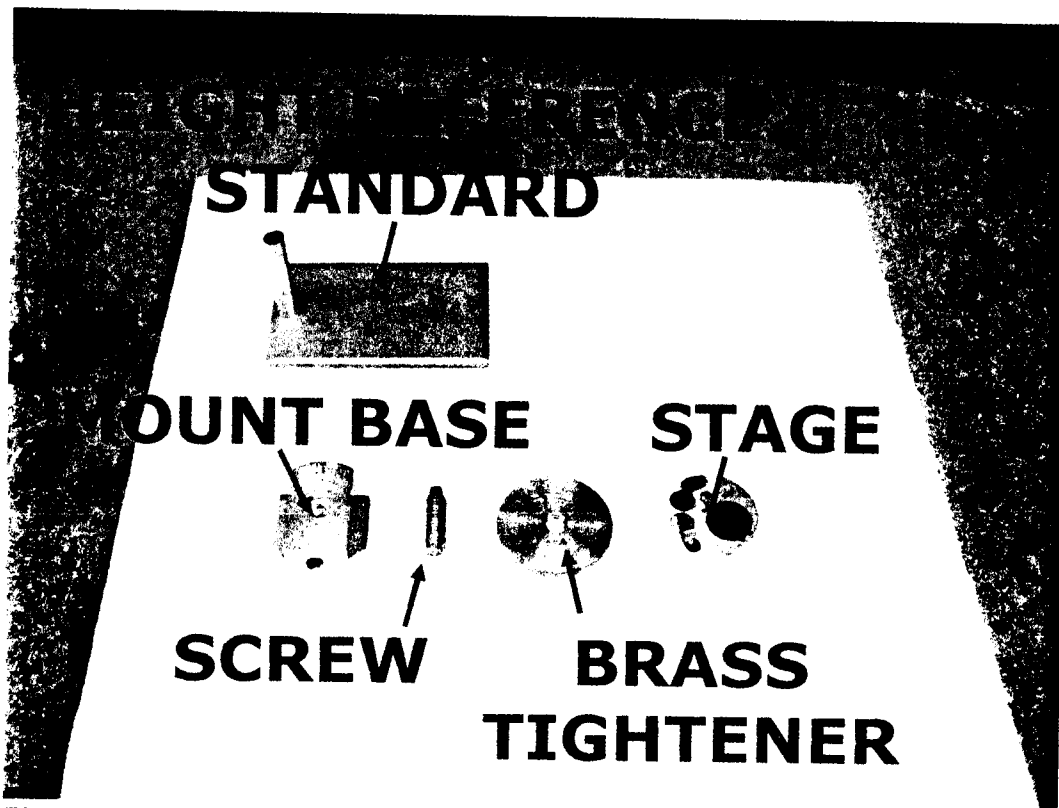


Figure 2A: Sample Mount Parts

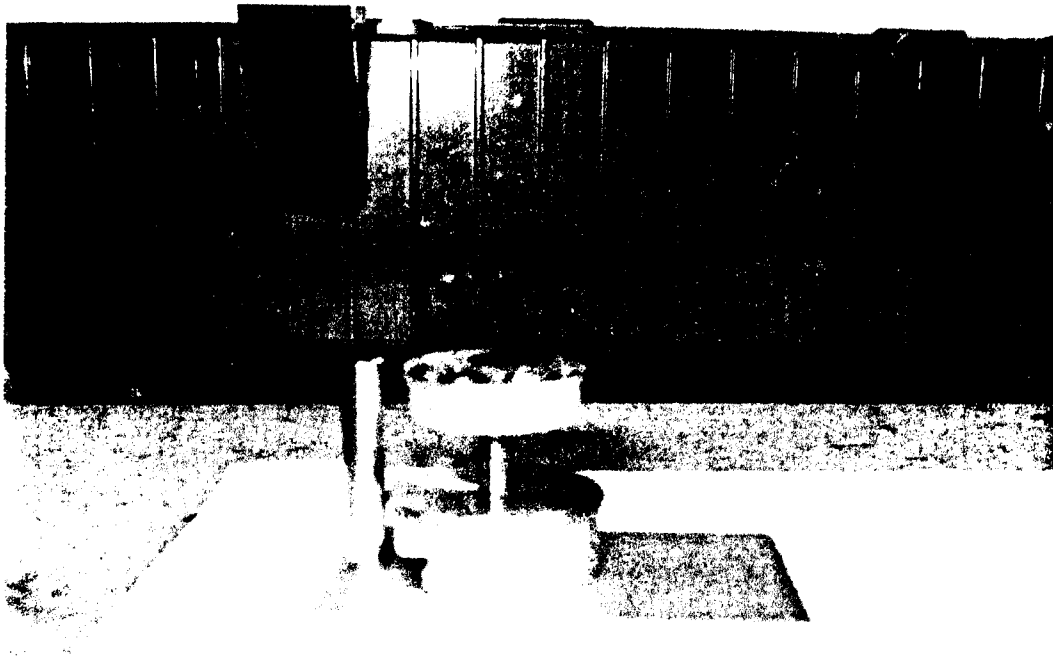


Figure 2B: Assembled Sample Mount

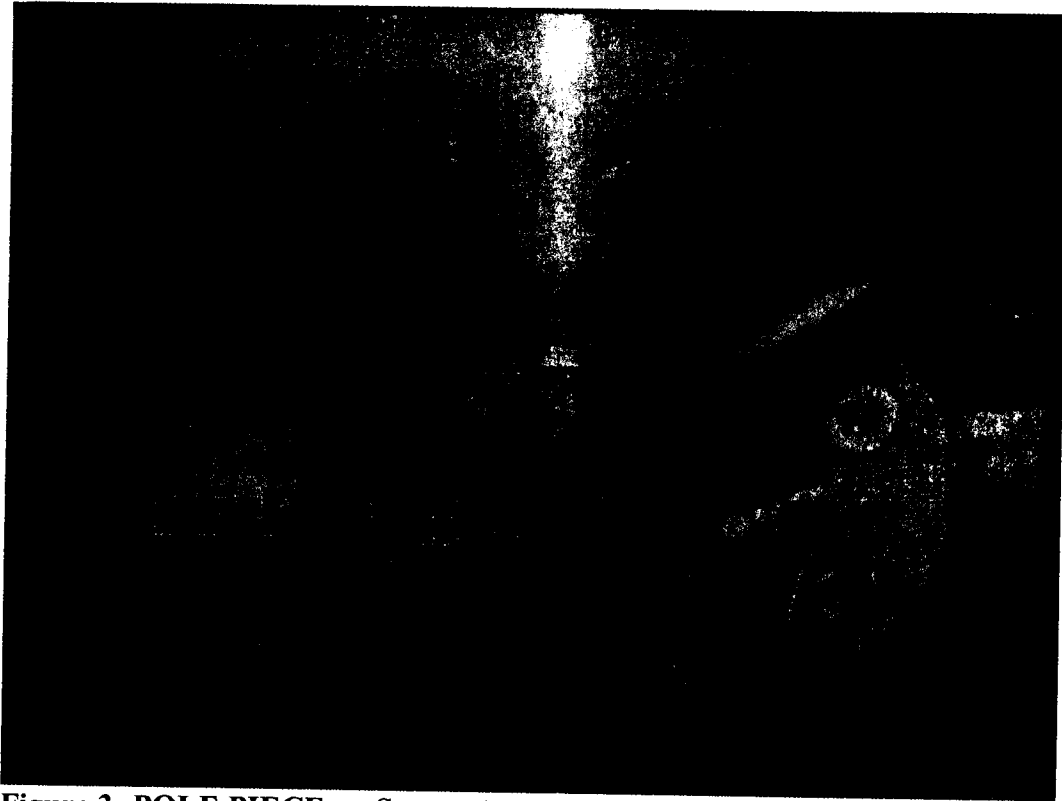


Figure 3: POLE PIECE on Screen A

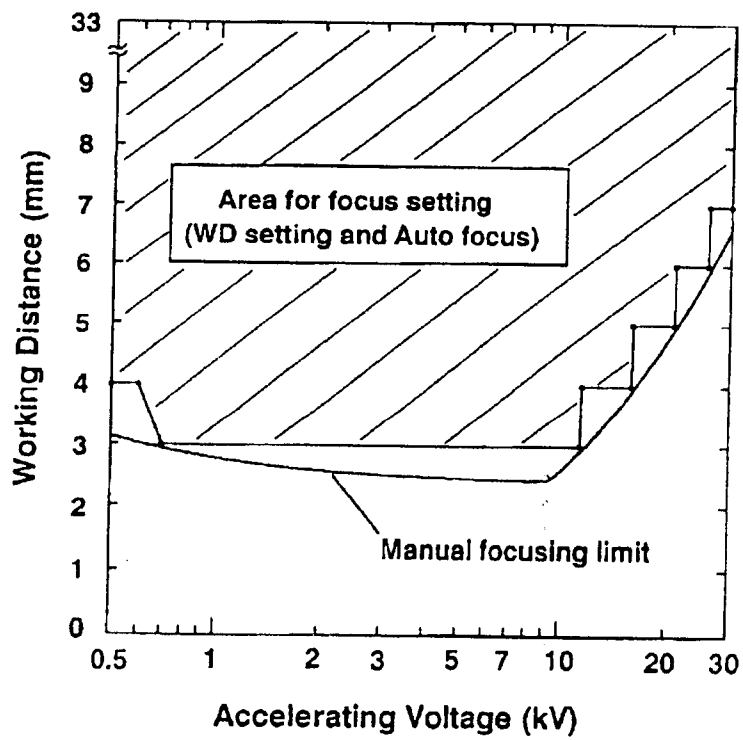


Figure 4: Focusing Limits of the SEM

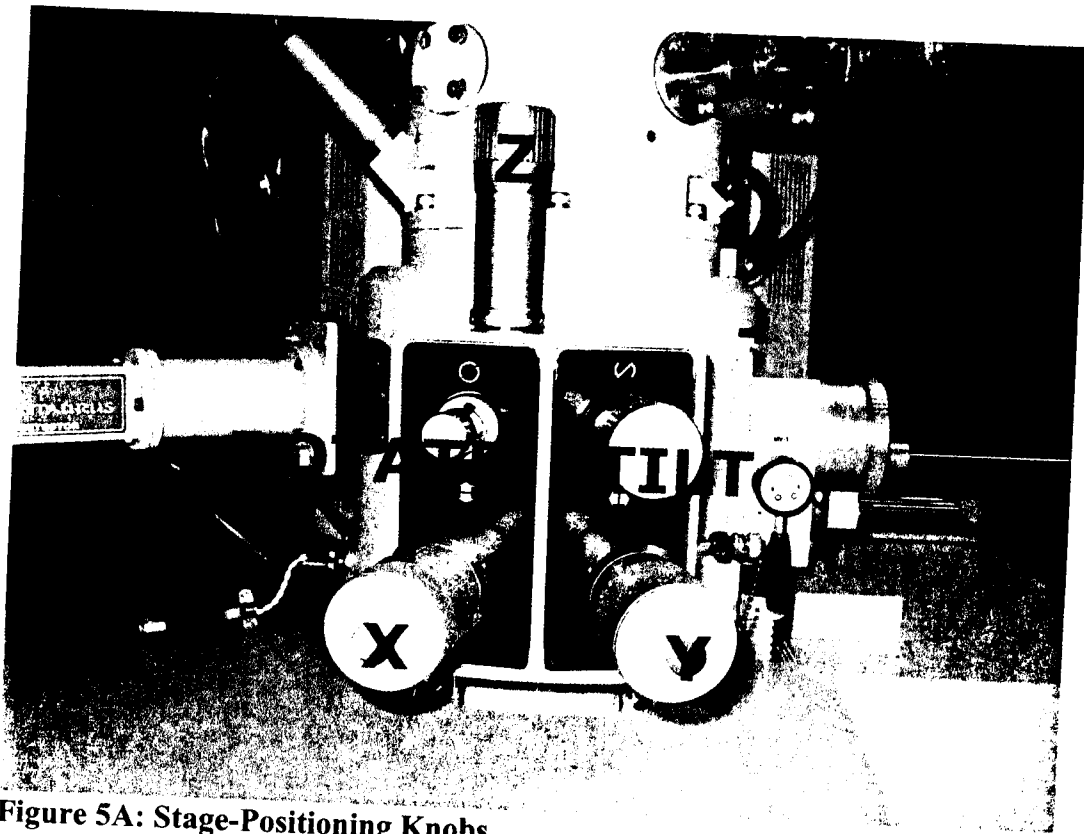


Figure 5A: Stage-Positioning Knobs

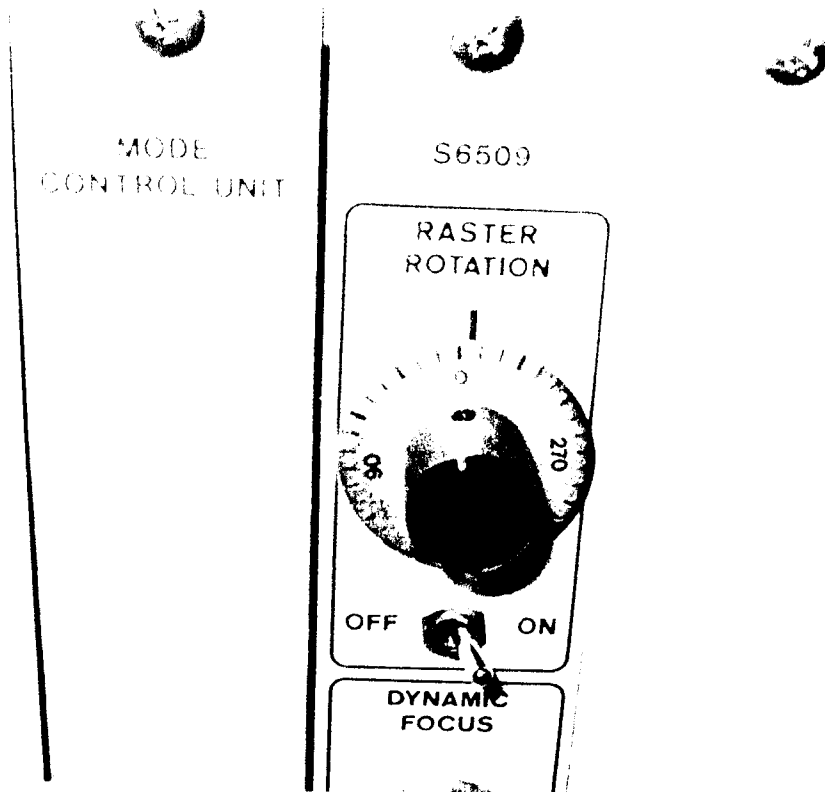


Figure 5B: RASTER ROTATION Knob and Switch