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WHY COLD FIELD EMISSION?

There are several benefits to cold field emission. Cold Cathode Field Emission microscopy provides higher resolution, higher beam density (brightness), and longer tip life than Thermal Tungsten wire SEMs and thermally assisted “Schottky” field emitters. The following Table highlights those parameters responsible for the Cold Cathode Field Emission’s higher performance at lower accelerating voltages.

<table>
<thead>
<tr>
<th>Source Diameter</th>
<th>Energy Spread (eV)</th>
<th>Brightness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thermal Tungsten</td>
<td>50 – 100 kA’</td>
<td>&lt; 2.0</td>
</tr>
<tr>
<td>Cold Cathode Field Emission</td>
<td>30 – 50 A’</td>
<td>0.2</td>
</tr>
</tbody>
</table>

HOW DOES IT WORK?

The field emission tip is made up of a sharply etched piece of monocrystalline tungsten. A field is applied to the tip causing electrons to tunnel out of the tip and accelerate down the column. This is a basic diagram of the electron gun assembly in a field emission system. Notice that, in addition to the usual accelerating voltage anode ($V_0$), a second voltage anode has been placed in close proximity to the FE tip. The desired accelerating voltage is obtained by adjusting $V_0$ to accelerate or decelerate electrons emitted at the extraction voltage $V_1$. It is the ultimate combination of these two anode potentials which sets the final electron speed down the optical column. The computer automatically ratios these values for the operator.
APPEARANCE OF THE S-4700

Fig. 2-2  S-4700 Main Unit (type I)
SECTIONAL VIEW OF THE S-4700 COLUMN

Fig. 2-3   Sectional View of S-4700 Column
STARTING CONDITIONS

1. EVAC POWER:
   **ON** (-)
   DP/TMP, WATER, and AIR PRES lamps: **LIT** (green)

2. OBJ. APT.:
   Switch is set at **HEAT**

3. ION PUMP VACUUM:
   IP1, IP2, and IP3 **LIT** (green).
   * IP: Ion pump

   Gun and Column Minimum Vacuum Levels:
   IP-1: $1 \times 10^{-8}$ Pa;  IP-2: $1 \times 10^{-7} - 1 \times 10^{-7}$ Pa;  IP-3: $1 \times 10^{-7} - 2 \times 10^{-6}$ Pa (<3x10$^{-6}$ Pa)
   * Check gun chamber vacuum condition (IP1, IP2, and IP3) by turning the knob.

4. CHAMBER VACUUM:
   HIGH Lamps of S.C and S.E.C **LIT** (green)
   $P_e$: $L \times 10^{-3}$ Pa; $P_i$‘s: $L \times 10^{-1}$ Pa
   * S.C: Sample Chamber
   * S.E.C: Sample Exchange Chamber
   * $L$: Less than 1
   * $P_e/P_i$: Penning / Pirani vacuum gauge
   * Check S.C and S.E.C chamber vacuum condition by turning the knob.

5. VALVE:
   GUN: Toggle switch to AUTO (OPEN)
   EXCHANGE (MV1, manual valve 1): **FLASHING** (yellow)
   Both “CLOSED” are **LIT** (green)
   * When HV is on:
     GUN, AUTO (OPEN): **FLASHING** (yellow)
     EXCHANGE (MV1): **LIT** (green)
   * When MV1 is open (during sample exchange)
     GUN, AUTO (OPEN): **LIT** (green)
     EXCHANGE (MV1): **LIT** (yellow)
     CHAMBER EVAC: **FLASHING** (yellow)

6. CHAMBER:
   S.C./S.E.C toggle switch to **S.E.C position**. S.C EVAC CHAMBER button **LIT** (green).
   * Buzzer will go if the switch is toggled to S.C (due to OBJ. APT. HEAT)
7. Specimen Bias Voltage (labeled VSP) cable CONNECTED

8. Infrared Chamber scope and Sony monitor ON.

9. Ensure that the BSE Detector is in the fully retracted (out of the column) position!!

COLD FINGER (Optional)

The built-in anti-contamination “cold finger”, located on the right side of the specimen chamber, can be filled with liquid nitrogen to reduce visible contamination on the specimen by condensing any volatiles or impurities that can impede image observation, especially at high magnifications. Note, it will not decontaminate your specimen. The cold finger has a capacity of 0.9 L and is usable for nearly 5 hours once filled with LN₂. Pour in slowly at first and allow the trap to chill down for several minutes, then add liquid Nitrogen until overflow occurs. About 1.3 L of liquid nitrogen is required to cool Dewar from 24 °C to attain filled condition. It is important to maintain the trap fully cooled throughout your session because trapped gases will release if warmed—increasing specimen contamination.
SPECIMEN LOADING (standard Hitachi stage; Hitachi specimen rod)

A sample mounted at the appropriate height is shown on the right.

The Sled is attached to the Specimen Exchange Rod; the Post connects the Sled to the Sample Mount of choice; the Locking Ring is tightened to secure the sample at the appropriate height.

There are a variety of Sample Mounts that fall into two categories: those to which the samples are attached directly; those which accept the standard Pin-Type stubs.
SAMPLE INSERTION

1. Always check the sample height using the sample height gauge (if height exceeds the gauge, consult with staff).

2. The Stage must be in the **HOME position**. Move the stage manually to the appropriate Home Settings in the Table below. Current Home Setting can be obtained from the “Stage Z Axis Setting Today” posting on the front of the column.

<table>
<thead>
<tr>
<th>Stage</th>
<th>Rod</th>
<th>Z (mm)</th>
<th>Rotate (°)</th>
<th>Tilt (°)</th>
<th>X (mm)</th>
<th>Y (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EMITECH cryo-stage</td>
<td>EMITECH rod</td>
<td>15</td>
<td>0</td>
<td>0</td>
<td>12.5</td>
<td>12.5</td>
</tr>
<tr>
<td>EMITECH cryo-stage</td>
<td>HITACHI rod</td>
<td>28.5</td>
<td>0</td>
<td>0</td>
<td>12.5</td>
<td>12.5</td>
</tr>
<tr>
<td>HITACHI stage</td>
<td>HITACHI rod</td>
<td>13</td>
<td>0</td>
<td>0</td>
<td>12.5</td>
<td>12.5</td>
</tr>
</tbody>
</table>

3. **Make sure the High Voltage is OFF**; **S.C./S.E.C toggle switch to S.E.C position**; **and the MV1 Chamber Valve is in the closed position**.

*Consult “Computer Startup and Gun Flash Procedure” section if the computer/software is not on/open.

4. Ensure that the **STAGE LOCK** is **OFF**.

5. Hit the **AIR** button (on the front panel of the scope) to vent the sample exchange chamber.

6. Pull the door open by grabbing the S.E.C unit, not the rod. **NEVER TOUCH THE EXCHANGING ROD** because it goes into the vacuum.

7. Push the Specimen Exchange Rod to the left side slightly to unlock it and screw the sample holder onto the end of the rod.

8. Pull the rod back to the **locked position** (until you feel a bump) and push slightly the S.E.C unit against the chamber O-ring (you will hear a hissing of the chamber vent line).
9. Press the EVAC button to evacuate the exchange chamber.

10. Once the S.E.C CHAMBER VACUUM reading reaches **HIGH, OPEN** the MV1 Valve.

11. While viewing the specimen chamber, push the rod in completely, so that the specimen base is securely inserted into the mating receiver on the stage, and unscrew the sample holder to release the rod. Pull back slightly on rod to ensure disengagement. Use the chamber monitor to oversee this process.

12. Pull the rod back into the **locked position** and **CLOSE** the MV1 Valve.

**SAMPLE WITHDRAWAL**

1. Make sure the Stage Lock is **RELEASED** and that **High Voltage is OFF**.

2. Move the stage to the appropriate Home Settings in the Table below.

<table>
<thead>
<tr>
<th>STAGE HOME POSITIONS</th>
<th>Stage</th>
<th>Rod</th>
<th>Z (mm)</th>
<th>Rotate (°)</th>
<th>Tilt (°)</th>
<th>X (mm)</th>
<th>Y (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>EMITECH cryo-stage</td>
<td>EMITECH rod</td>
<td>15</td>
<td>0</td>
<td>0</td>
<td>12.5</td>
<td>12.5</td>
</tr>
<tr>
<td></td>
<td>EMITECH cryo-stage</td>
<td>HITACHI rod</td>
<td>28.5</td>
<td>0</td>
<td>0</td>
<td>12.5</td>
<td>12.5</td>
</tr>
<tr>
<td></td>
<td>HITACHI stage</td>
<td>HITACHI rod</td>
<td>13</td>
<td>0</td>
<td>0</td>
<td>12.5</td>
<td>12.5</td>
</tr>
</tbody>
</table>

3. **OPEN** the MV1 Valve.

4. While viewing the Specimen Chamber, insert the Specimen Exchange Rod and screw it into the Specimen Holder.

5. Pull the Specimen Exchange Rod out and into the **locked position**.

6. **CLOSE** the MV1 Valve.

7. Make sure the SEC S.C/S.E.C toggle switch is set to **S.E.C** and press the AIR button to vent the Sample Exchange Chamber.

8. Unlock the Specimen Exchange Rod and unscrew the specimen from the exchange rod.

9. **Lock** the Specimen Exchange Rod and press the EVAC button to evacuate the exchange chamber.
SET IMAGING PARAMETERS

* [From OLD version] Switch GUN VALVE to OPEN/AUTO. The valve can be kept on OPEN/AUTO all the time, and the gun valve will automatically open upon turning on the High Voltage.

1. Open the HV Control window by left clicking anywhere in the gray HV Control area (top right corner of the viewing screen). If you are prompted to flash, click on “Flashing” --- “Flashing Execute OK?” --- “Execute” (see “Computer Startup and Gun Flash” section).

2. Select the Accelerating Voltage in the HV Control window, by scrolling through the Vacc selections.

3. An additional parameter that can be adjusted in this window is the Emission Current (range 1 – 20 uA). The default setting is 10 uA. A higher emission current will provide a greater signal but may also induce faster contamination or charging of the sample. The advantage to altering the emission current is that a specific voltage can be maintained, yet neither the alignments nor system resolution are significantly affected.

4. Click the ON button in the HV Control window to apply the Accelerating Voltage. Once HV has turned on, the ON button turns into the SET button. If Emission Current subsequently decreases from your chosen setting, click the SET button to restore it.

5. Open the Column Setup window by clicking on the icon.

    - Select the Operation Mode (see Table below). Note: changing modes alters the path of the beam down the column, changing cross-over positions or the strengths of the stigmator coils. Thus, realignment may be necessary. Since the S-4700 has been made with automated Operation Modes, it is not necessary to change the Objective Lens Aperture on the column.
<table>
<thead>
<tr>
<th>Mode</th>
<th>W.D. Range</th>
<th>Benefit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultra High Resolution</td>
<td>2.5 mm – 10 mm</td>
<td>Provides for short working distances and high resolution capabilities</td>
</tr>
<tr>
<td>Normal</td>
<td>6 mm – 15 mm</td>
<td>Provides a good compromise between high resolution and increased depth of field</td>
</tr>
<tr>
<td>Long Working Distance</td>
<td>More than 15 mm</td>
<td>Provides increased depth of field when high resolution is not the major concern</td>
</tr>
<tr>
<td>Magnetic</td>
<td>6 mm – 30 mm</td>
<td>The beam cross over is positioned in the area of the stigmator coils -- thus extending the strength of the stigmators to adjust for the field from the sample.</td>
</tr>
</tbody>
</table>

- Select the **Working Distance (W.D.)**. Select an initial working distance in the Column Setup window which corresponds to the Z value of your Home Setting. This will put you in the vicinity of focus for your specimen. Subsequent changes in working distance are made by mechanically changing Z and subsequently focusing. **You can’t adjust W.D. at LM mode.**

- Select the **Cond Lens 1** setting (range: 1 – 16 with higher numbers representing a smaller spot size; the default setting is 5). The condenser lens controls the final spot size on the specimen. The smaller the spot size, the better the resolution; however, beam current will be reduced. The Signal-to-Noise (S/N) ratio will decrease with less beam current; therefore, the operator must optimize the SEM for either a high signal image or a highly resolved image. **Cond Lens 2** also must be selected, but is not adjustable.

- Select the **SE detector** that is to be used: Upper, Lower, or Mixed.

<table>
<thead>
<tr>
<th>Detector</th>
<th>General Working Distance Ranges (VERY Sample Dependent)</th>
<th>Benefit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Upper (through-the-lens) SE Detector</td>
<td>6 mm or less</td>
<td>Only high resolution electrons (SEI, SEII) collected. Advantageous for high magnification/resolution applications; however, less topography is observed. Fully capable of signal collection through the entire range of working distances, even at low accelerating voltages; however, low S/N ratios occur at longer working distances</td>
</tr>
<tr>
<td>Lower SE Detector</td>
<td>15 mm or greater</td>
<td>The detector collects all types of secondary electrons, as well as an amount of backscattered electrons, from an angled point of view. Image includes topographical detail and appears to be less prone to charging due to the backscatter electron component of the signal.</td>
</tr>
<tr>
<td>Mixed</td>
<td>6 – 15 mm</td>
<td>Some combination of the above</td>
</tr>
</tbody>
</table>
- Select flashing intensity if directed to flash. (It is usually “2”.) It is very unlikely you will need to flash unless you are the first user of the day in “after-hours” (weekend, etc.)

- The Column Setup window also contains a DeGauss button which can be used to reduce hysteresis in the objective lens. DeGaussing should be used more often when large changes in magnification occur. DeGaussing is automatically performed after change of: Accelerating Voltage; Working Distance; and Operation Mode.

- Ensure that the ABCC Link box is checked. DO NOT adjust the Flash Intensity setting. Use “2” for usual operation.
GENERAL OPERATION

Tilting toward the Lower SE Detector will increase signal. See Table below for ranges.

<table>
<thead>
<tr>
<th>Stage Ranges</th>
<th>X (mm)</th>
<th>Y (mm)</th>
<th>Z (mm)</th>
<th>T (°) *</th>
<th>R (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HITACHI stage</td>
<td>0 – 25</td>
<td>0 – 25</td>
<td>2.5 – 27.5</td>
<td>-5 – 45</td>
<td>0 – 360</td>
</tr>
</tbody>
</table>
| EMITECH cryo-stage| 0 – 25 | 0 – 25 | 2.5 – 27.5 | 0 – 45  | 0 – 1.5 CW     
|                   |        |        |         |         | 20 – 10 CCW   |

* Care should be taken when applying Tilt at short Working Distances. The possibility of the specimen hitting the objective lens or backscatter detector increases under these conditions. Use the Chamber View monitor to ensure safe manipulation.

* Engaging the Stage Lock is advisable at 50,000 magnification and above to reduce specimen movement.

* NOTE: The Stage Lock should be turned OFF before a Z or Tilt motion is made on the stage. Failure to unlock the stage will result in damage to the stage locking plate and stage instability which may become evident in the SEM image.

Focusing and astigmatism compensation should be conducted at higher magnifications than those at which you will be acquiring images. Use the Reduced Area View screen to aid with these processes.

“Specimen movement” at higher magnifications (15,000 and above) is conducted on screen with electrical image shift controls: At lower magnification, center the yellow crosshair by clicking on the Crosshair button below it. Center the object of interest on the screen manually with the Stage Controls and then raise the magnification. Click on the Mouse icon under “Beam” to activate the “Blue hand/Beam” cursor. Move object to the screen center by left-clicking and dragging. Right-click to turn off “Blue hand”

If the Beam Monitor flashes red, click Adjust

Charging and beam-sensitive samples: Low accelerating voltages are required for these specimens. 1 – 3 kV is a good starting point. A lower emission current can also significantly reduce charging/contamination effects, but at the expense of signal. The advantage to altering the emission current is that a specific voltage can be maintained, yet neither the alignments nor system resolution are affected. Specifically with biological/polymeric and semi-conductor samples, lowering the emission current has proven to be very helpful. Imaging can be accomplished at emission currents as low as 1 µA.
OBTAINING AN IMAGE

1. Select Low Magnification mode. Click on the H/L button to toggle between High and Low Magnification modes.

2. Click the Automatic Brightness and Contrast Compensation button.

3. Locate and center an area of interest with the manual Stage Controls (Magnification may also need to be adjusted at this point). Rough focus the area of interest with the Focus Knob on the Control Panel.

4. Select High Magnification mode. To adjust the magnification continuously, place the mouse in the gray Magnification area, click and hold the left mouse button, then drag the mouse to the left to reduce the magnification, or to the right to increase the magnification. Clicking the right mouse button and dragging will enable faster magnification adjustment. Use the manual stage controls to locate the region of interest.

5. Adjust Brightness and Contrast:

   Click the Automatic Brightness and Contrast Compensation button; or

   Click in the upper right quadrant of the screen. Depress the appropriate mouse button (left – fine; right – course) and drag the mouse in the X-axis direction to adjust; or

   Use the scroll bars directly above the image to adjust.

6. Adjust Focus:

   Use the Focus knobs on the control panel; or

   Click in the bottom half of the screen. Depress the appropriate mouse button (left – fine; right – course) and drag the mouse in the X-axis direction to adjust; or

   Use the scroll bars directly above the image to adjust.

7. Astigmatism compensation (X and then Y):

   Use the Stig knobs on the control panel; or

   Click in the upper left quadrant of the screen. Depress the appropriate mouse button (left – fine; right – course) and drag the mouse in the X-axis direction to adjust; or

   Use the scroll bars directly above the image to adjust.
ALIGNMENT

Once the image is well focused and initial astigmatism compensation has been performed, the alignment procedure should be performed. In fact, any time a detector, operating condition, or voltage is changed, the alignment sequence should be completed.

The alignment procedure is an iterative process in that you generally need to start at lower magnification and repeat Aperture align and Stigma Align X and Y at successively higher magnifications for greater precision. Also, at times, you should click the Off button in the Alignment window, focus and compensate for astigmatism, and then continue with the alignment process.

1. Click on the Alignment icon and the Alignment window will appear. For all alignments procedures, use the Stigma/Alignment knobs on the Control Panel.

2. Select Beam Align. A target will appear. Using the Control Panel knobs, move the circular beam to the center of the “bulls-eye” target. If the circular beam does not appear, increase Contrast.

3. Click “OFF”, then center and focus a unique point on the specimen. Select Aperture Align. Eliminate any lateral shift in the specimen image with the alignment knobs.


5. Aligning High Mag to Low Mag Mode:
   - Select Off in the Alignment window.
   - Set the magnification in High Mag Mode to the minimum and center a unique point on the specimen. Centering can be facilitated with the screen cross-hair by checking the Area Marker box in the lower right portion of the screen.
   - Switch to Low Mag Mode by clicking the H/L button. Focus and stigmatate.
   - Click Low Mag Position in the Alignment window.
   - Turn the Stig/Alignment knobs to move the point of interest on the specimen to the center point

6. Click Off and then Close the Alignment Window.
7. Raster Rotation

RASTER ROTATION: The scanning of the electron beam can be rotated within a range of -200° to +200°, using the RASTER ROTATION slider. Image rotation caused by the objective lens magnetic field is compensated automatically even when the above rotation angle is set at 0 or the RASTER ROTATION box is not checked.

DYNAMIC FOCUS: When a specimen is tilted large angle, the field of view that can be focused is small especially under the condition of low magnification. DYNAMIC FOCUS function allows you to focus the beam for the entire field of view by correcting the deviation in focus for the tilt direction (particularly at low magnifications and at a short working distance). Move slider to the midpoint (0). Focus on the center of the image, and then adjust the slider so that the entire image is in focus.

TILT COMPENSATION: When a specimen is tilted, magnification along the tilting direction is lower than that of a non-tilting direction. As a result, the image appears to be contracted in the tilting direction. The image can be corrected at the magnification in all directions by using TILT COMPENSATION function. Y direction: sample insertion direction. Use DYNAMIC FOCUS, if necessary.

8. If “Beam Monit.” (Beam current monito) box is blinking red, click on adjust.
IMAGE CAPTURE

There are a few options for capturing images:

a. Integration of a TV (Fast Scan) rate scan
b. Slow scan capture
c. “Instant freeze”, in which case the image can automatically be transferred to PCI. (Screen capture, 640 X 480 pixels only)

1. To set up the capturing methods, click on the “Image Setup” icon. The standard settings are: Fast Scan 1: 16 frames; Fast Scan 2: 64 frames; All four Slow Scan Modes: 80 sec.

2. Once the conditions for capturing images have been established, simply click on the “Capture Image” icon in one of the two Fast Scan rates, or any of the four slow scan rates to activate the capturing sequence. Alternatively, the live image can be frozen at any time by depressing the “Run/Stop” icon. In this case, whether in TV rate or a slow scan, the image will be available for saving or transferring to PCI once the scan has finished. The image will be a 640 x 480 digital image, and will not be entered into the “Captured Image” window.

3. The “Captured Image” window will automatically appear after an image has been captured. Once in the “Captured Image” window, an image can be highlighted and:
   - Displayed in the Scanning Image window by selecting the icon;
   - Transferred to PCI by selecting the icon; (It only activates when scanning STOPS.)
   - Opened and displayed in a Window Viewer by selecting the icon;
   - Saved by selecting the icon.
BACKSCATTER ELECTRON IMAGING

1. Establish a 10 mm or greater distance on the Chamber Scope between the top of your specimen and the bottom of the Objective Lens. This is a SAFE distance; ensures detector will not strike specimen. Actual BSE working distance can be as short as 5 mm.

2. Screw the Backscatter Detector fully into the column (see orange indicator guides on the detector).

3. Go to Low Mag Mode. Retract the detector slightly to center its outline on the viewing screen. Return to High Mag Mode if desired.

4. Select the YAGBSE (Yttrium-Aluminum-Garnet) detector in the Signal Select window.

5. There are several ways to increase the signal when BSE imaging:
   - Increase the Accelerating Voltage. 5 kV is a good starting point. (BSE signal is low at < 4 kV, depends on specimen.)
   - Selecting Analysis Mode in the Column Set-up window will increase the beam current by a factor of approximately ten over the Ultra High Resolution Mode (remember that switching modes will require column re-alignment). However, this might charge up or damage specimen.
   - While BSE imaging in any Mode, you can also raise the emission current in the HV Control window, up to 20 uA. This is advantageous in that neither the resolution, nor the alignment settings, are affected. The current density in the beam will simply increase and there will be a better Signal-to-Noise ratio. However, this might charge up or damage specimen.
   - High accelerating voltage could also lead to increased charging and/or beam damage. You can also increase the Cond Lens 1 (Spot Size) setting in the Column Set-up window while in any Mode. The default is 5 (1 represents the largest spot size and 16 the smallest). Remember, however, that as the spot size increases, the resolution will decrease.

6. There are two ways to compare images using the SE or the BSE detector – Dual Mag mode or Split Screen mode. After selecting one of these modes, open the “Signal Select” window and choose SE for the right side, and BSE for the left side (or vice versa).

7. Some final notes about using the BSE detector
   - BSE imaging is less affected by charging; therefore, it may be advantageous to use the BSE detector on samples that tend to charge excessively, or that will not provide suitable SE images.
• The greatest signal collection for BSE detection occurs when the sample is at 0 degrees tilt. The working distance varies for each specific model: **5 mm should be considered a minimum working distance.**

8. **Ensure that you retract the BSE detector fully from the column when you are done imaging!**

CHARING: FINDING THE E2 VALUE

Charging is the condition when a material cannot effectively conduct the beam energy imparted to it. There are two types of charging: Negative (the most common) and Positive.

Negative charging occurs when impinging electrons are trapped within the sample and an electrostatic charge builds up. The image will glow or show distortion as electron production is artificially enhanced and the beam is unintentionally deflected.

Positive charging occurs when more electrons are emitted from the sample than the primary electron beam provided. Rather than glowing, a dark box will appear as secondary electrons are emitted.

For each material, E1 and E2 are constants – voltages at which the sample achieves charge balance. Usually there is a range of voltages for which a sample can be exposed, up to and including the E2 value. E2 is the value to seek for uncoated observation since it is found at a higher accelerating voltage, thus utilizing a higher resolution condition of the SEM. However, the voltage to use is dependent on the required sample information. Beams at high voltages will image layers deeper into the sample, while low voltages will provide more information from the sample surface.

**Trick of the trade: Scan-square method**

1. A practical and easy way to test the sample’s conductivity is as follows:
2. Focus on an area at a high magnification.
3. Wait a few seconds, letting the beam irradiate the selected area.
4. Reduce the magnification and observe the sample.
5. If a bright square appears (which may disappear upon going to the lower magnification), negative charging is probable. Therefore, lower the voltage.
6. If a dark square appears, and then quickly disappears, positive charging is probable. Therefore, raise the voltage. Note: If the dark square remains, contamination is most likely the problem. See the section on Contamination Control.

7. The effect of charging is exacerbated at higher magnifications, so be sure to check these conditions at levels near to or at the highest magnification you intend to use.
COMPUTER (SOFTWARE) STARTUP

1. Turn on DISPLAY POWER (- / 0) switch in computer cabinet.

2. Turn on PC Power. (Button on Compaq CPU in cabinet) Green “POWER” light lights.
   System starts and prepares to boot. *(Note: Monitor is normally left ON (small green LED lighted and flashing). Check it.)*

3. Compaq / MS Windows 95 screen opens for ~ 30-40 seconds, then closes.

4. “Enter Network Password” window appears:
   System asks; “Enter network Password”
   “User name: S-4700”
   “Password: ______________”
   *(Type FESEM2 for password (NOT case sensitive), then Enter or OK.)*

5. Windows Desktop opens and “Initial Logo” window appears:
   “S-4700 Scanning Electron Microscope”
   “Enter Login: S-4700”
   “Password: ______________”
   *(Hit Enter key or click OK)*

   Set VALVE: GUN, on column panel, to AUTO, if it’s not already there. (flip toggle switch up)

GUN FLASH PROCEDURE

1. Left click on “Vacc”, in upper right corner of screen. “HV Control” window appears.
   *(Note: Vacc can be any value at this point, but Emission Current (“Ie”) should be set at 10 uA.)*

2. Left click “Flashing”.

3. “Flashing Execute OK?” Click “Execute”. “Flashing Executing” message appears under Ie and write down Vext. The flashing is finished when “FLASHING” disappears.

4. When “HV Control” window reappears, click “ON”. The FE-PCSEM / “HV On” window appears and displays the rise in Vext and Ie.
5. When Step 4 is finished, note the “Ie”, “Vext”, and time of flash and enter data in the log book. *(Note: Occasionally the flash will fail. Check the log book… the Vext value at this point should be consistent with previous days’ post-flash values. If not (usually will be much higher if flash failed) just repeat steps “3” and “4”, above. The system may ask you to wait 20 seconds or so until flash occurs.)*

6. Monitor system every 15-20 minutes for next hour or so if possible…click “SET” to restore Ie to selected value (normally 10 uA) when it decreases below 10 uA. Vext will begin to rise in increments of 100 volts or so, usually each time “SET” is clicked. Both Ie and Vext will eventually stabilize…usually takes 1-2 hours. *If you cannot be around to do this, you can also just turn the HV off.* The microscope can be used at any time after flash, but users need to be aware that the Ie will be unstable for a while. Clicking “SET” when Ie decreases, especially just before recording an image, will help to ensure good images, and image quality will improve as Vext returns to 3.7 kV and above.

**GUN FLASH**

The “flash” procedure helps to establish stable operation of the cold field-emission electron gun by driving excess adsorbed gas molecules from the gun’s cathode, or electron emitter. This is done by briefly heating the cathode to high temperature. Remember that the S-4700’s field-emission gun normally operates with an unheated cathode.

Flashing is usually done in the morning prior to the start of the day’s observations, and it will usually not be necessary to repeat the procedures until the next morning, or even several mornings later. From our experience, it’s safe to say that it needn’t be done every day, but routine flashing each day that the microscope may be used is an acceptable practice to ensure predictable performance. It’s also possible that instability in the gun might require an additional flash, but this microscope has not had this problem.

Be aware that there is a sequence of events that begins with the flash which might affect the quality of your images, and is why it’s preferable to flash the gun early in the morning. First, for about five or ten minutes after flash, the cathode is “clean”, or free of adsorbed gas molecules, and the emission is stable. But gas molecules soon begin to be adsorbed, desorbed, and transferred. This activity can result in emission current fluctuation and is seen as changes in image brightness and quality as the emission current decreases.

The next period, usually beginning about one hour after flash (can be 30 minutes to 3 hours afterward) is the most stable period, and results from a monolayer of gas molecules having been adsorbed onto the cathode. It can last anywhere from hours too many days, depending on the quality of the vacuum in the gun area.

The last period begins when a sufficient quantity of gas has been adsorbed onto the cathode to end the stable monolayer phase and once again cause emission instability. If allowed to continue too long there could be a high voltage discharge in the gun and the cathode could be damaged.
TROUBLESHOOTING

If GUN does not TURN ON…

1. Check S.C. vacuum. If the vacuum is high, check your sample (high vapor pressure) or check anti-contamination cold finger dewar.

2. Check DP/TMP, WATER, and AIR PRES on the microscope panel.

3. Or restart the computer to re-establish communication between computer and microscope.

If this not solve, check with staff or contact Hitachi Engineer (contact is on the white board)

If you think you are lost during alignment…

1. Hit AFC (Auto Focus Control) and AST (Auto Stigmation Control)

2. Go to Alignment and Reset All.

3. If 1) and 2) does not work, try a long W.D. and find your sample and gradually change W.D. while you observe your sample at HM mode.
Hitachi S-4700 FEGSEM
End-of-Day Shutdown Procedure

Follow this simple shutdown procedure when you use the instrument after normal hours (08:00 am ~ 4:30 pm) or on weekends, or if instructed to when staff is away.

1. Left click X in upper right corner to close the “PC-SEM” operating system program.
2. Window [“FE-PC SEM”] appears: [“Exit SEM Manager. OK?”]
3. Click “OK”
4. Left click START → Shutdown → “Shut down the computer?”
5. Click “Yes”
6. Monitor display goes to blue, then to black. **Wait ~ 30 seconds as CPU turns off.**
7. Open the tall narrow cabinet door; below, left.
8. Just above the **PC-Power** button, set “Display” [- / 0] switch to 0.
9. You can leave the Monitor /display power on. (small green light blinking).