

HITACHI 4700 FE-SEM

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WHY COLD FIELD EMISSION?

There are several benefits to cold field emission and few detractors. 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.

	Source Diameter	Energy Spread (eV)	Brightness
Thermal Tungsten	50-100 kA°	< 2.0	1 x
Cold Cathode Field Emission	$30 - 50 \text{ A}^{\circ}$	0.2	1,000 x

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 Vo 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.



STARTING CONDITIONS

- 1. EVAC POWER is **ON** (-)
- 2. DP/TMP, WATER, and AIR PRES lamps are **LIT (green)**
- 3. IP-1, IP-2, and IP-3 lamps **LIT** (green).
- 4. OBJ. APT. switch is set at **HEAT**
- 5. Gun and Column MinimumVacuum Levels: <u>IP-1</u> -- 1x10⁻⁸ Pa; <u>IP-2</u> -- 1x10⁻⁷ Pa; IP-3 -- 5x10⁻⁶ Pa
- 6. Chamber Vacuums: <u>Pe</u> -- $1x10^{-3}$ Pa; <u>Pi</u>'s: -- $1x10^{-1}$ Pa
- 7. HIGH Lamps of S.C Vacuum and S.E.C Vacuum are LIT (green)
- 8. AUTO (OPEN) of GUN VALVE and OPEN of MV1 (EXCHANGE) VALVE FLASHING YELLOW
- **9.** S.C./S.E.C toggle switch to **S.E.C position**. S.C. EVAC CHAMBER button **LIT**.
- **10.** Specimen Bias Voltage (labeled VSP) cable **CONNECTED**

OBJ. APT. HEAT OFF DEGAS OFF







- 11. Infrared Chamber scope and Sony monitor ON.
- 12. 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 collecting any grease, dirt, or impurities that can impede image observation, especially at high magnifications. The cold finger has a capacity of 0.9 L and is usable for nearly 5 hours once liquid Nitrogen is injected. Fill the Cold Finger with liquid Nitrogen. Pour in slowly at first and allow the trap to chill down for several minutes. Then add liquid Nitrogen until overflow occurs. 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 (<u>if height exceeds the gauge</u>, <u>consult with staff</u>).
- 2. The Stage must be in the **HOME position**. Move the stage manually to the appropriate Home Settings in the Table below. <u>Current Home Setting can be obtained from the</u> <u>"Stage Z Axis Setting Today" posting on the front of the column.</u>

STAGE HOME POSITIONS							
StageRodZ (mm)Rotate (°)Tilt (°)X (mm)Y (mm)							
EMITECH cryo-stage	EMITECH rod	15	0	0	12.5	12.5	
EMITECH cryo-stage	HITACHI rod	28.5	0	0	12.5	12.5	
HITACHI stage	HITACHI rod	12	0	0	12.5	12.5	

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 to vent the exchange chamber.
- 6. Pull the door open by grabbing the SEC unit, not the rod.
- 7. Push the Specimen Exchange Rod slightly to unlock it and screw the sample holder onto the end of the rod.



- 8. Pull the rod back to the **locked position** and push the SEC 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 Chamber 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 Chamber 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.

STAGE HOME POSITIONS						
Stage	Rod	Z (mm)	Rotate (°)	Tilt (°)	X (mm)	Y (mm)
EMITECH cryo-stage	EMITECH rod	15	0	0	12.5	12.5
EMITECH cryo-stage	HITACHI rod	28.5	0	0	12.5	12.5
HITACHI stage	HITACHI rod	12	0	0	12.5	12.5

- 3. **OPEN** the MV1 Chamber 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 Chamber 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

- 1. Switch GUN VALVE to **OPEN/AUTO**. <u>The valve can be kept on OPEN/AUTO all the time, and the gun valve will automatically open upon turning on the High Voltage</u>.
- 2. Open the HV Control window by left clicking anywhere in the gray HV display 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).
- 3. Select the Accelerating Voltage in the HV Control window, by scrolling through the Vacc selections.
- 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

HV Control		×
ON	OFF	Flashing
Vacc	S	et le to
10.0 kV	•÷	10 uA 💌
	Close	

altering the emission current is that a specific voltage can be maintained, yet neither the alignments nor system resolution are significantly affected.

5. 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.



- 6. Open the Column Setup window by clicking on the
- icon.
- Select the Operation Mode (see Table below). <u>Note</u>: 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.

Column SetUp 🛛 🔀
Operation Mode
Ultra High Resolution
- Set Lens Condition
Working Distance 12.0 mm
Cond Lens 1 5.0
Cond Lens 2 DeGauss
📕 Specimen Bias Voltage
SE Detector
HV
Flashing Intensity 2
ABCC Link Close

Mode	W.D. Range	Benefit
Ultra High	2.5 mm. – 10 mm.	Provides for short working distances and high
Resolution		resolution capabilities
Normal	6 mm. – 15 mm.	Provides a good compromise between high resolution
		and increased depth of field
Long	More than 15 mm.	Provides increased depth of field when high resolution
Working		is not the major concern
Distance		
Magnetic	6 mm. – 30 mm.	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.

• Select the Working Distance. 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.



- 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.
- The Column Setup window also contains a <u>DeGauss</u> 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. <u>DO NOT adjust the Flash Intensity</u> <u>setting.</u>

Detector	General Working Distance Ranges (VERY Sample Dependent)	Benefit
Upper (through- the-lens) SE Detector	6 mm or less	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
Lower SE Detector	15 mm or greater	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.
Mixed	6 – 15 mm	Some combination of the above

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



OBTAINING AN IMAGE

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

Auto

- 2. Click the Automatic Brightness and Contrast 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 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**. 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.

Aperture Align and Stig Align should be carried out at successively higher magnifications.

- 1. Click on the Alignment icon and the Alignment window will appear. For all alignments procedures, use the Stig/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. Center and focus a unique point on the specimen. Select Aperture Align. Eliminate any shift in the specimen image with the alignment knobs.

Aperture Align	Reset	Reset All
C Beam Align		
C Aperture Align		
🔿 Stigma Align, X		
🔿 Stigma Align, Y		
C Low Mag Position	1.0000000000000	
C 09		

- 4. Perform the same procedure for both Stig Align X and Stig Align Y.
- 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 Marked box in the lower right portion of the screen.
 - Switch to Low Mag Mode by clicking the H/L button. Focus and stigmate.
 - Click Low Mag Position in the Alignment window.
 - Turn the Stig/Alignment knobs until the point of interest on the specimen comes to the center point
- 6. Click Off and then Close in the Alignment Window.



GENERAL OPERATION

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

Stage Ranges						
	X (mm)	Y (mm)	$Z (mm) \bullet$	T (°) * •	$R(^{\circ})$	
HITACHI stage	0 25	0 25	2.5 27.5	-5 45	0 360	
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.

• <u>NOTE</u>: 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.

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

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. 2-3 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 1μ A.



Charging: 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.



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.



IMAGE ACQUISITION

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.
- 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;
 - opened and displayed in a Window Viewer by selecting the _____ icon;
 - saved by selecting the **saved** 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.
- 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 detector in the Signal Select window.
- 5. There are several ways to increase the signal when BSE imaging:
 - Increase the Accelerating Voltage (but this may also lead to increased charging).
 5 kV is a good staring point.
 - 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).
 - While BSE imaging in any Mode, you can also raise the emission current in the HV Control window, up to 20uA. 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:Noise ratio.
 - 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, with 1 representing 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. <u>Ensure that you retract the BSE detector fully from the column when you are done imaging!</u>



Hitachi S-4700 FEGSEM Computer Startup and Gun Flash Procedure

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 fieldemission 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.

Computer/Software Startup

- 1. Turn on DISPLAY POWER (- / 0) switch in computer cabinet.
- Turn on PC Power. (Button is on Compaq CPU in computer cabinet) System starts and prepares to boot. (*Note: Monitor is normally left ON (green LED lighted). Check it.*)
- 3. When "F1: Boot" (lower left corner of screen) appears, **hit F1 key**.
- 4. Compaq / MS Windows 95 screen opens, then closes.
- 5. "Enter Network Password" window appears: System asks; "Enter network Password" "User name: S-4700" "Password: " (Type FESEM2 for password, then Enter or OK.)
- 6. Windows Desktop opens and "Initial Logo" window appears: "S-4700 Scanning Electron Microscope" "Enter Login: S-4700" "Password: (Hit Enter key or click OK)
- "Hitachi S-4700 Scanning Electron Microscope" window appears. Set VALVE : GUN, on column panel, to AUTO, if it's not already there. (flip toggle switch up)

Gun Flash Procedure

- 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, and then disappears when flashing has been completed.



- 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 e 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* <u>much higher if flash failed</u>) just repeat steps c and d, 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 5.3 kV and above.