Knowledge Enterprise Development

Arizona State University

NanoFab

HITACHI S-4700 FESEM STANDARD OPERATION PROCEDURE

Rev I

Table of Contents

ERC-131 Hitachi S-4700 FESEM Laboratory Rules

1. Please follow all NanoFab laboratory safety and user regulations. Failure to do so will result in a safety violation according to the NanoFab Safety Violations Escalation policy.
2. Reservations can be made on-line through iLab NanoFab equipment scheduling: https://asu.corefacilities.org/sc/3688/nanofab?tab=equipment.
3. Normal staff working hours are 8 AM – 5 PM, Monday – Friday. ISAAC access to the ERC-131 Laboratory must be requested at the end of a successful training session. If there is an emergency, please contact one of the following people. Proceed down the list until you reach someone.

Primary contact:	Kevin Nordquist: ERC-144 kevin.nordquist@asu.edu Phone: 480-965-3410
Secondary contacts:	Kevin Hilgers: ERC-152 kevin.hilgers@asu.edu Phone: 480-965-5256
Local Facilities contact:	Art Handugan: ERC-151 Handugan@asu.edu Phone: 480-727-7143
Hitachi Service contact:	Phone: 800-253-3053
EDAX Service contact:	Phone: 800-535-3329

3. No food or drink is allowed in NanoFab laboratories, PLEASE.

4. Please sign the iPad log and verify the FESEM default conditions (see checklist) before proceeding with loading of any samples.

5. NEVER HANDLE ANY PARTS (SAMPLE HOLDER, EXHANGE ROD, ETC.) WHICH GO INTO THE SAMPLE CHAMBER WITHOUT PROTECTIVE GLOVES: GLOVES ARE PROVIDED FOR USE IN THE LAB.

6. Please keep the FESEM facility clean and organized.

7. Do not remove any tools, equipment, or manuals from the FESEM facility.

8. All specimen/samples must be clean and dry before placing into the FESEM specimen chamber. If necessary, the specimen should be cleaned with an approved solvent and dried thoroughly using dry nitrogen gas prior to introduction to SEM vacuum. Qualified personnel (rule #2) may suggest alternative cleaning procedures for particular and/or unusual specimen samples.

9. The following specimens should never be placed in the FESEM specimen chamber: The FESEM vacuum system is oil free, i.e. it is maintained by a magnetically levitated turbo-pump and two dry mechanical pumps. We wish to minimize or avoid oil-related (organic) contamination in the vacuum chamber.

Dirty, Greasy or Oily Samples (contamination problems)
Uncured Epoxies and Resins (outgassing problems).
Liquid/Liquid Laden Samples (outgassing problems).
High Vapor Pressure Materials (outgassing problems).
Salts (high moisture content, corrosive).

10. The samples listed below may be placed into the microscope upon approval of the FESEM Applications Committee.

Powders
Insulators
Minerals
Photoresists

11. **DO NOT FORCE ANYTHING!** Damaging equipment through ignorance or negligence will result in banishment from the lab.

12. Do not tamper with anything on the instrument that you have no prior experience with or training. **IN PARTICULAR, THE MECHANICAL ALIGNMENT OF THE APERTURES IS PRESET; DO NOT ADJUST THESE UNDER ANY CIRCUMSTANCE**. Such adjustments are primarily reserved for Hitachi Technical. Attempting to repair items associated with the instrument is strictly forbidden. Report any problems first to personnel as per rule #2.

13. No unauthorized person is allowed to use the FESEM.

14. Any malfunction or damage of the FESEM should be reported to personnel as per rule #2.

15. IF DAMAGE OCCURS THROUGH NEGLIGENCE OR INFRACTION OF THE RULES, THE PROJECT SPONSOR/USER WILL BE HELD FINANCIALLY RESPONSIBLE.
16. Show common courtesy; please do not interrupt FESEM users during their assigned time.

17. Laboratory and instrument access requires successful completion of a proficiency test. Users are expected to understand and comply with general safety and the laboratory rules and maintain the instrument's default conditions. The hands-on test will be given by one of the primary or secondary contacts or designee.

18. Each operator will be given a user ID and password to log onto the S-4700. It is recommended that newly trained FESEM operators use the tool between normally working hours (Monday – Friday, 8 AM – 5 PM) so that if they have an issue with the tool NanoFab staff may be on hand to assist.

19. The procedure to save/transfer files from the FESEM or EDX – which is secure and endorsed by RTS policy, is to the Gateway PC. Please save your image files to the FESEM desktop IMAGES folder. The path is C:\WINNT\PROFILES\ADMINISTRATOR\DESKTOP\IMAGES. The folder on the EDX desktop is called DATA – you can save directly to it.

Both these folders are for temporary use only – once you FTP the files to the Gateway PC – you should delete them from the IMAGES folder. These folders will be checking periodically. Old files found more than two months old will be deleted with no notification to the user. As the available free memory is used up by files remaining on the drives – the likelihood of software failure increases. Access to the Gateway PC is via FileZilla and you will need to use either your ASURITE or ASUAD. Hostname is fesem-ex.eas.asu.edu with Port 990. The trainer will send a request to RTS for new user access to the Gateway PC. You will need to download WinSCP onto your lap-top/PC to retrieve your files from the Gateway PC. Software window settings and/or user preferences must not be altered on either Hitachi or EDAX systems. The loading of any additional software requires NanoFab authorization.

20. The operator may not use any image processing or microanalysis software or hardware without prior training. There are some software/hardware conflicts with the system.

21. The operator will be required to note, in the logbook, any defaults not properly set.

22. When you have finished using the FESEM, reset all the software defaults and cleaned up the work station, please be sure the HEPA filter fans are turned on and the lights are tuned off when you leave the room.

23. **Each operator must ensure that the S-4700 has the correct default settings prior to and upon conclusion of FESEM work.**

DEFAULT CHECK LIST AND BASIC OPERATIONS GUIDE

PURPOSE: The checklist provides the operator with all column, vacuum, and software defaults established by Hitachi and the NanoFab in a simple and understandable fashion. The basic operational guide will provide the operator with the correct procedures for obtaining high quality images.

Suggested further reading: http://www.geology.wisc.edu/~johnf/g777/JEOLguide.pdf

DEFAULT CHECK LIST

Sign in the iPad log and enter additional information as required:

_____**User Name:**

_____**Sample:**

_____**Flash:** Flash the gun if "PLEASE FLASH" is seen flashing in Scanning Image Screen.

_____**Flash Current:** Record the flash current displayed in the emission current window. A single flash of the filament should be all that is required to achieve an acceptable current around 30 microamperes

_____**V_{ext} (Start):** Record the gun extraction voltage after acceleration voltage is applied. This should be approximately 4.3 to 4.5 kV if the gun has just been flashed. High extraction voltage (5.3-5.5 kV) when acceleration voltage is applied gives an indication that flashing may be required during the course of the session.

_____**V_{ext} (End):** Record the gun extraction voltage at the end of the session. The absolute values and differences in the extraction voltages are used by Hitachi Technical to establish a history on gun performance (which will assist with instrument diagnostics)

_____**Comments:** Include here any pertinent information that may be used to assist with instrument diagnosis (keV used), comments regarding sample type, instrument performance (such as, difficulty with alignment, astigmatism, focusing, etc.), for whom the sample has been run for if a service, etc.

Column defaults

_____Robinson Chamber View camera and monitor must be turned off. **To view inside S.C.**

_____Pico-ammeter: Off

_____EDAX Detector: Retracted to 6 cm. LN2 will be topped off by Center staff.

_____Beam Monitor Aperture (upper aperture): In position 2 or 3. **Do not change**.

_____Objective Aperture (lower aperture): In position 2 or 3. **Do not change.**

_____Faraday Cup: Mechanical control setting is out and switch is set to meter.

_____BIAS Cable: Ensure cable is connected. * **Continuity Cable**

Ensure that MV-1, specimen chamber is closed (handle pointing down, just slightly past the six-o'clock position).

Evacuation control panel defaults

_____Emergency Off: Disabled. Large red push button, use only for emergency to completely shut down the instrument. Care must be taken not to accidentally hit this switch

Evacuation Power

_____EVAC Power: On
_____TMP: Green *Turbo Molecular Pump
_____Water: Green
_____Air Pressure: Green
_____Objective Aperture: Switch to Heat with green light.

Ion Pump Vacuum

Selector switches to note the vacuum on each pump.		Typical Conditions
IP1	Green - indicates a vacuum better than 2E-7Pa	1E-8Pa
IP2	Green - indicates a vacuum better than 2E-6Pa	1E-7Pa
IP3	Green - indicates a vacuum better than 7E-5Pa	2E-6Pa

Chamber Vacuum

Selector switch to indicate the vacuum degree on Penning and Pirani gauge.		Indicator	Typical Conditions
Specimen Chamber	high	Green	< 7E-3 Pa
Specimen Chamber	low	Off	7E-3 Pa < Low < 10 Pa
Specimen Chamber	air	Off	> 10 Pa
Specimen Exchange Chamber	high	Green	< 5 Pa (TC: 2 Pa)
Specimen Exchange Chamber	low	Off	5 Pa < Low < Pa
Specimen Exchange Chamber	air	Off	> 10 Pa

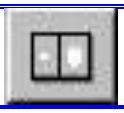
Gun

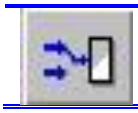
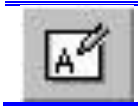
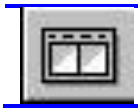
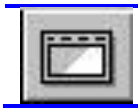
_____V-1 switch (High Voltage Interlock) in the closed position.
_____Gun: Green - On; Yellow - Flashing - indicates that the proper conditions have been satisfied for the auto opening of the V-1 value to be enabled.
_____Exchange: Green - On; Yellow - Flashing - indicates that the proper conditions have been satisfied for the exchange valve and can be opened when necessary.

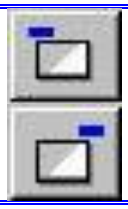
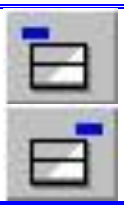
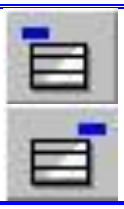
Chamber

_____Evac: Green – indicates that the exchange chamber is under vacuum.
_____Air: Off
_____S.C./S.E.C. – The toggle switch should always be in the S.E.C. position.

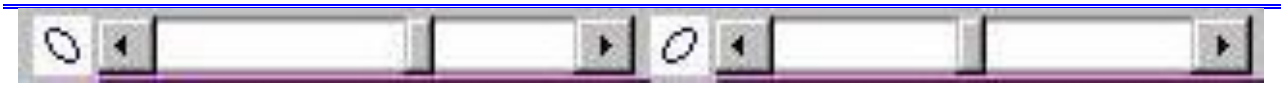
SOFTWARE INDICATORS AND CONTROLS

	Image Manager	Opens the Image Manager Window, identical to menu selection, File/Open Image Manger. Operations available in the Image Manager range from, create new user, delete existing user, the processing of images, storage of image files, printing of images, and some data/database entry.
	Save Image	Opens the dialog box to specify the name and location to save an image. Identical to the menu selection File/Save Image.
	Column	Opens the Column SetUp control window, identical to the menu selection Setup/Column.
	Alignment	Opens the Alignment control window, identical to the menu selection Operate/Alignment.
	Image	Opens the Image SetUp control window, identical to the menu selection Setup/Image.
	Data Display	Opens the Data Display control window, identical to the menu selection Setup/Data Display.
	Split DM	Opens the Split/Dual Mag control window, identical to the menu selection Scan/Split DM Mode.
	CD Measure	Starts the CD measurement tool, identical to the menu selection Analysis/Measure.
	Raster Rotation	Opens the Raster Rotation control window, identical to the menu selection Operate/ Raster Rotation.
	Stage Control	Open the Stage Control indicator and control window, identical to the menu selection Operate/Stage Control.

	Signal Select	Opens the Signal Select control window, identical to the menu selection Setup/Signal Select.
	Signal Processing	Opens the Signal Process control window, identical to the menu selection Image/Signal Process.
	Pseudo Color	Opens the Pseudo Color control window, identical to the menu selection Image/Pseudo Color.
	Data Entry	Opens the Data Entry control window, identical to the menu selection Image/Data Entry.
	Standard Screen	Displays images at 640 × 480 pixels, in addition to aligning the standard screen left, right or center.
	Dual Screen	Displays two images at 512 × 480 pixels in the dual screen mode.
	Full Screen	Displays a single image at 1024 × 768 pixels in a full screen mode.
	Analysis Mode	Enables the different scan modes for analysis.
	PCI	Starts the PCI system.
	HV Control	Vacc : Acceleration voltage indicator. Ie : Emission current indicator. Vext : Extraction voltage indicator.
	Freeze Run	Selection will Freeze current scan of image, while Run will resume the scan. Identical to the menu selection Scan/Run, Freeze.

	Capture	Captures the image currently displayed.
	Fast1 Fast2	Selection alternates between the Fast1 and Fast2 scan speeds.
	Slow1 Slow2	Selection alternates between the Slow1 and Slow2 scan speeds.
	Slow3 Slow4	Selection alternates between the Slow3 and Slow4 scan speeds.
	Reduced1 Reduced2	Produces a reduced image in the center of the screen. Produces a reduced bar scan.
	ABCC	Auto Brightness Contrast Control, established a baseline brightness and contrast.
	AFC	Auto Focus Control, not recommended works poorly
	AST	Auto Stigmation Control, centers stigmates.
	BC Monitor	Wave Function Monitor for brightness and contrast.

	Focus Monitor	Wave Function Monitor for focus.
	Wobbler	Automatically starts Aperture Alignment.
	Magnification Preset	Goes to the next highest preset magnification.
	Magnification	Current magnification
	H/L Magnification Mode	Changes between the High and Low Magnification Mode.



Stigma X adjustment slider | Stigma Y adjustment slider

Brightness adjustment slider | Contrast adjustment slider

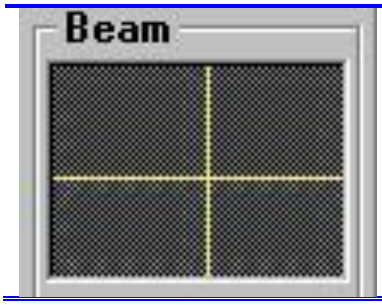
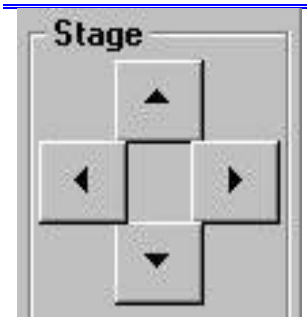
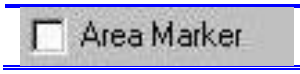
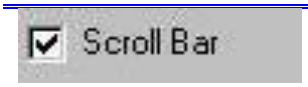
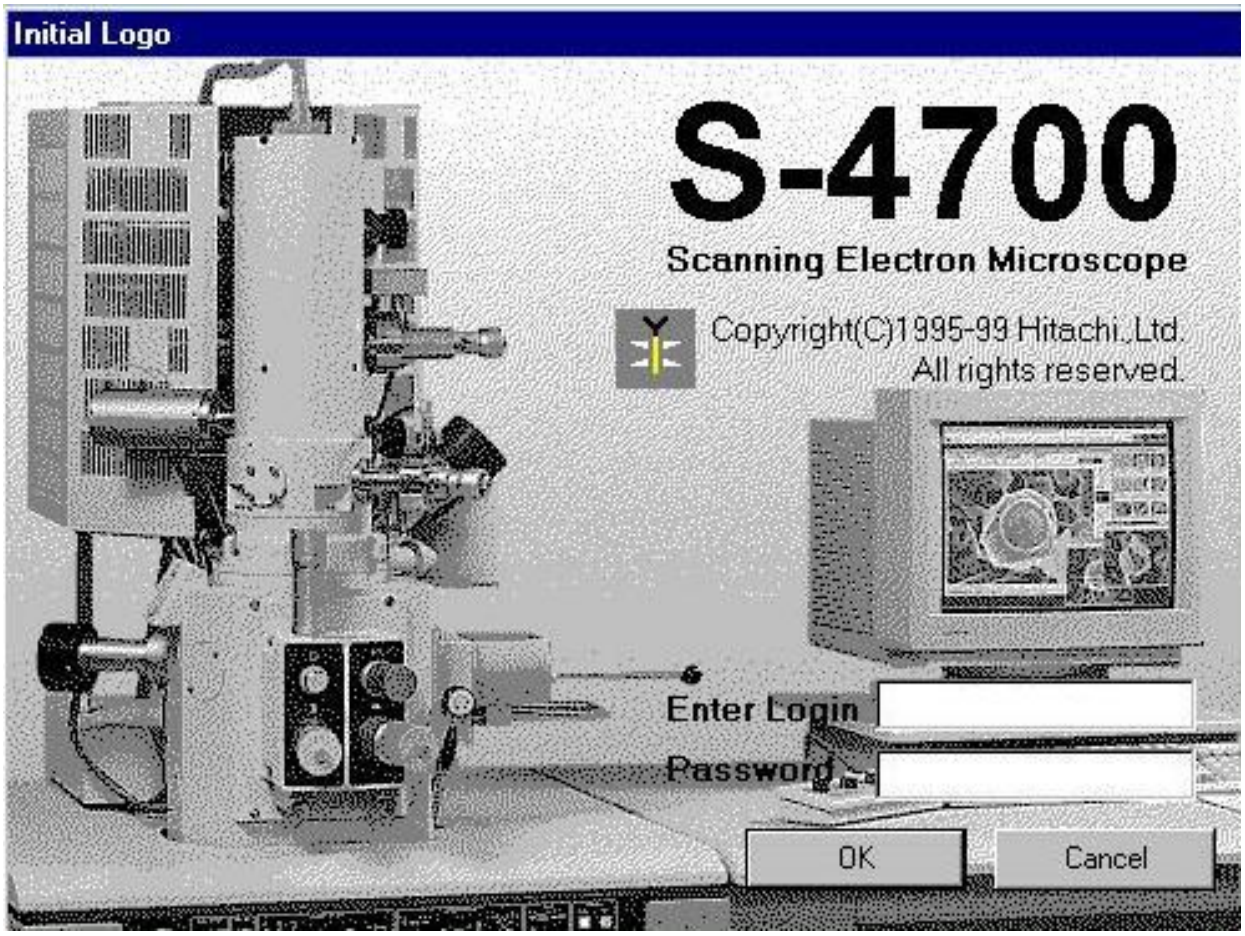
	Beam Monitor	Monitors beam current. *** If box is blinking red, click on adjust.**
	Image Shift	Beam raster indicator.

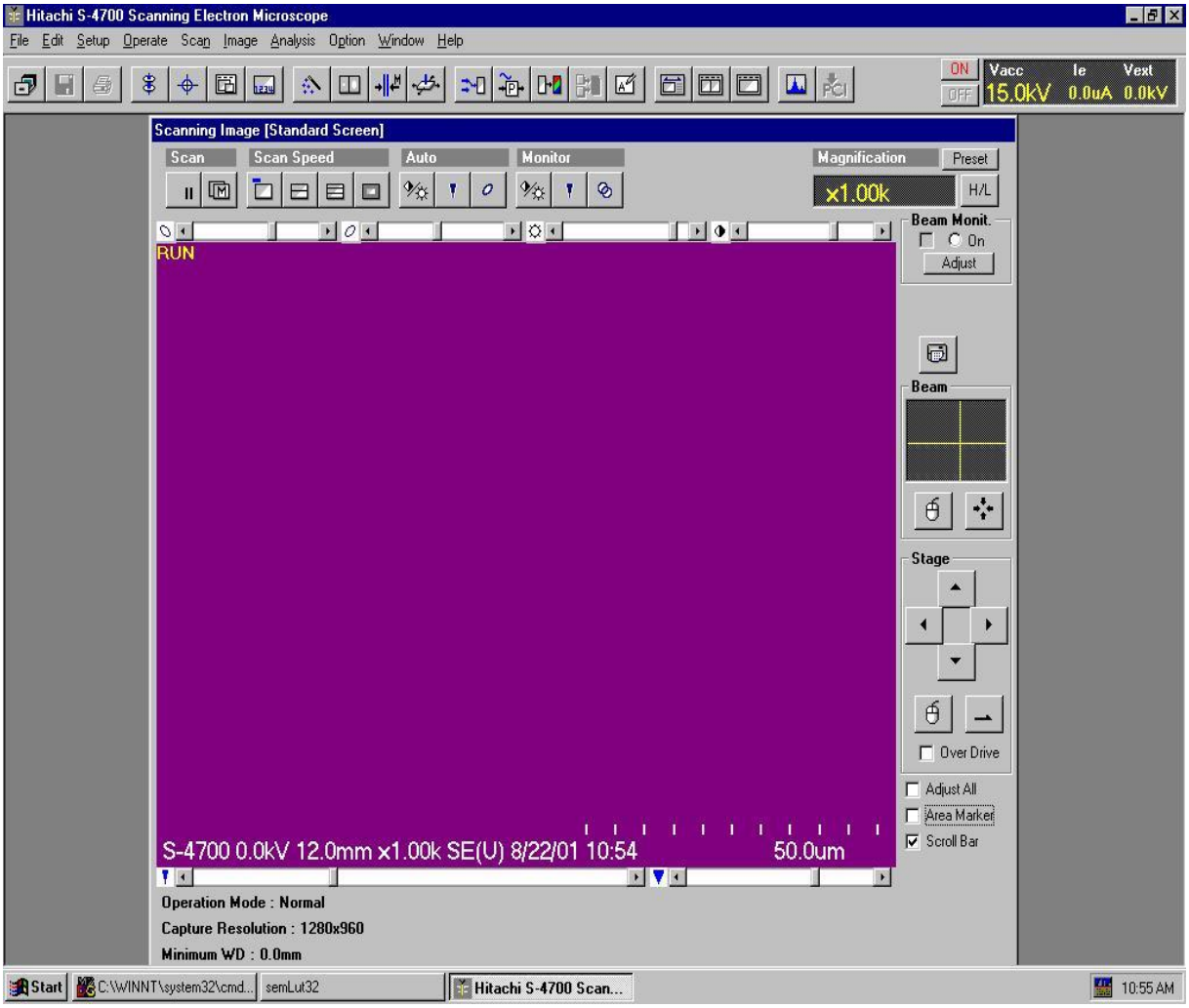
	Image Shift Mouse	Opens beam control cursor for click and drag image control.
	Image Shift Reset	Centers beam raster.
	Stage Step Control	Stage movement control.
	Stage Mouse	Opens stage control cursor for click and drag stage control.
	Stage Window	Opens Stage Control window.
	Stage Over Drive	The set moving distance will increase versus the mouse movement.
	Area Marker	Places cross hairs on image screen.
	Scroll Bar Display	Open digital slider control for brightness, contrast, course focus, fine focus, X-stigmation, and Y-stigmation.
 Digital control for Course Focus.	Course Focus	
 Digital control for Fine Focus.	Fine Focus	

SOFTWARE DEFAULTS

The Hitachi imaging software must be closed out at the end of each session, therefore, you must click the Hitachi icon,  , on the desktop to begin the software program by entering login and password. A login and password will be assigned to you after you demonstrate tool competency.



Default screen, this should be the view on the monitor after the program has started.



Stage Control [5-axis] [Operate → Stage Control]

_____Go to Home: Green indicator denotes the stage is home
_____Lock: Gray or off

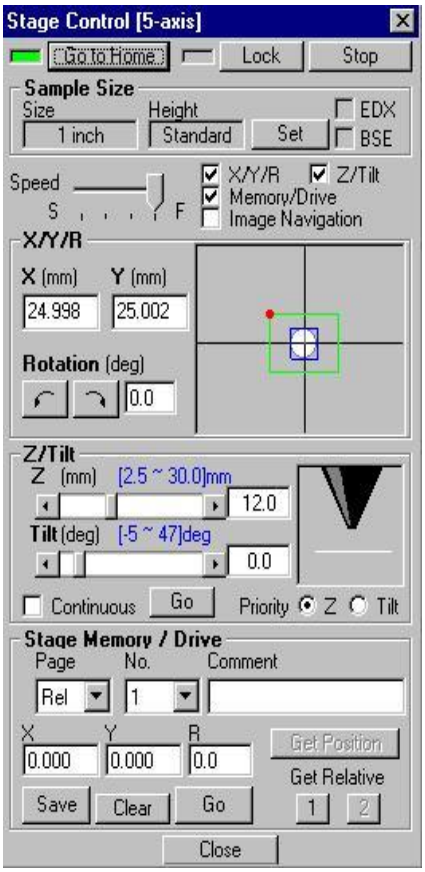
Sample Size
_____Size: 15 mm or 1 inch.
_____Height: Standard.
_____EDX: Not checked. (Energy Dispersive X-ray Analysis)
_____BSE: Not checked. (Back-Scattered Electron Imaging)

X/Y/R
_____Speed: Maximum.
_____X (mm): ≈ 25.000.
_____Y (mm): ≈ 25.000.
_____Rotation (deg): 360 or 0.

Z/Tilt.
_____Z (mm): 12.0.
_____Tilt (deg): 0.0.
_____Continuous: Not checked.
_____Priority: Z dotted - Tilt not dotted.

Stage Memory/Drive.
_____Page: Rel.
_____No.: 1.
_____Comments: None.
_____X: 0.000.
_____Y: 0.000.
_____R: 0.0.

Image Navigation. (We don't use this function)
_____Need refresh: gray.
_____Navigation box: gray.

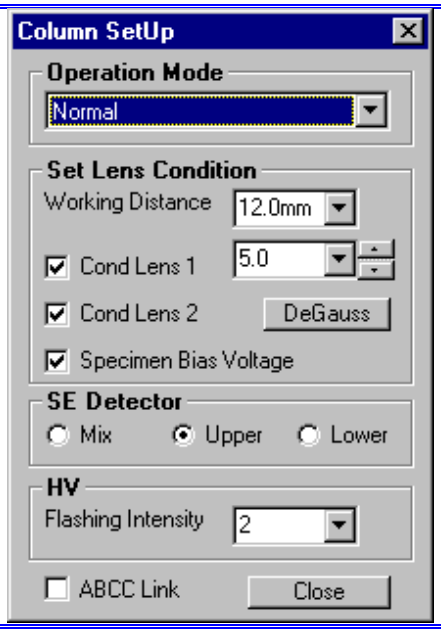


HV Control (Right Top) [Setup → HV]

_____ON: Enables the acceleration voltage. (V_{acc})
_____OFF: Disables the acceleration voltage.
_____V_{acc}: 15.0 kV - Allows operator to select desired acceleration voltage. (kV = kilovolts)
_____Set I_e to: 10µA - Allows operator to select desired emission current. (µA = micro-amps)
_____Flashing: To open Flashing Execute (see flashing procedures, p 24).

*I_e = Emission current

Column SetUp [Setup → Column]



Operation Mode:

_____Normal selected.

Set Lens Condition - **Note**: Ensure that the magnification is at x250 in the high magnification setting otherwise working conditions cannot be adjusted.

_____Working Distance: 12.0mm.

_____Condenser Lens 1: Checked and set at 5.0

_____Condenser Lens 2: Checked

_____Specimen Bias Voltage: Checked

SE Detector

_____Mixed: Not dotted.

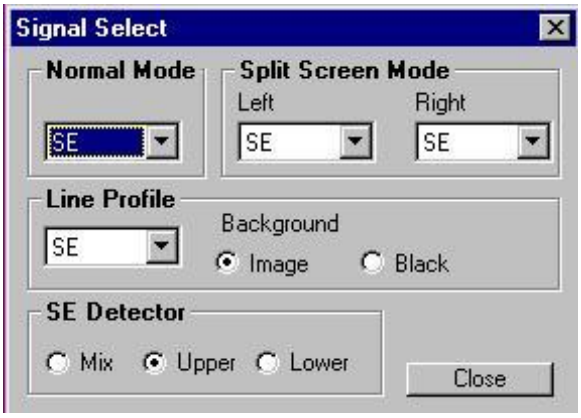
_____Upper: Dotted.

_____Lower: Not dotted.

HV - High Voltage

_____Flashing Intensity: 2

Signal Select [Setup → Signal Select]



Normal Mode

_____SE. (SE = Secondary Electron)

Split Screen Mode

_____Left: SE.

_____Right: SE.

Line Profile

_____SE.

_____Background: Image – Dotted.

SE Detector

_____Mix: Not dotted.

_____Upper: Dotted.

_____Lower: Not Dotted.

Signal Processing [Image→Signal Processing]

Frame Averaging.

_____Fast1: Set to 16.

_____Fast2: Set to 32.

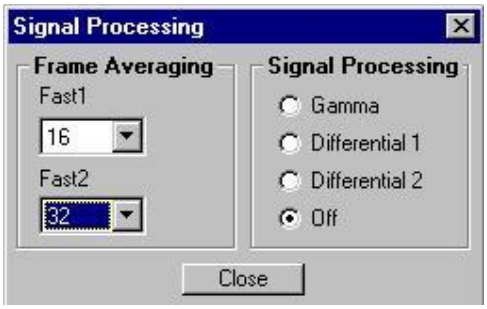
Signal Processing.

_____Gamma: Not Dotted.

_____Differential 1: Not Dotted.

_____Differential 2: Not Dotted.

_____Off: Dotted.



Raster Rotation [Operate → Raster Rotation]

_____**Raster Rotation:** Checked and set to zero.

_____**Dynamic Focus:** Not Checked.

_____**Tilt Compensation:** Not Checked

* Raster rotation will uncheck itself when user logs off

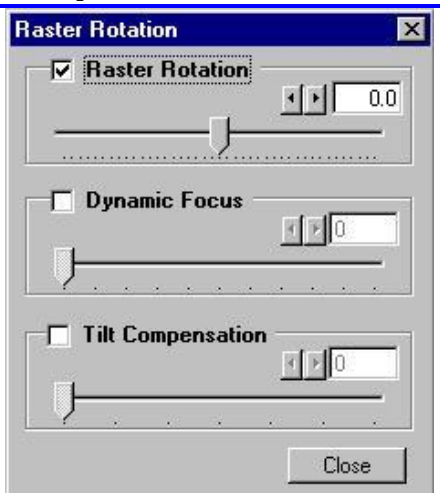


Image SetUp [Setup → Image]

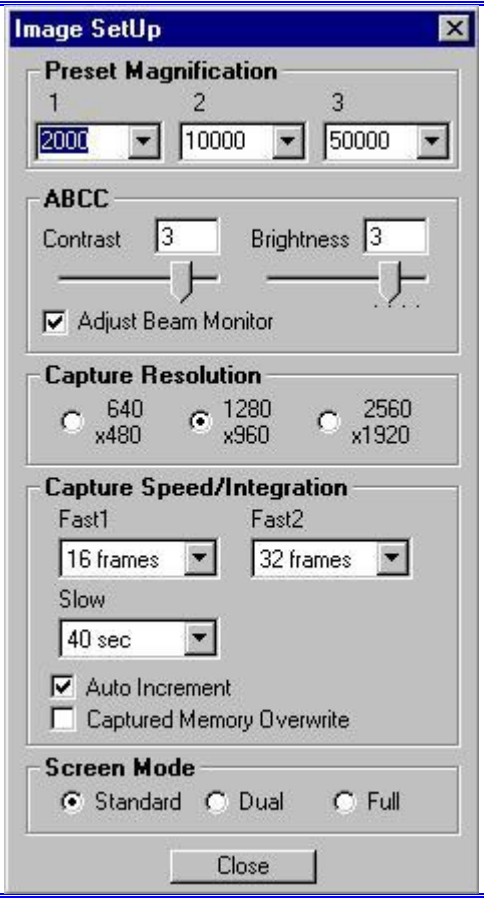
Preset Magnification
_____1: 2,000
_____2: 10,000
_____3: 50,000

ABCC (Auto Brightness Contrast Control)
_____Contrast: 3
_____Brightness: 3
_____Adjust Beam Monitor: Checked

Capture Resolution
_____640x480: Not dotted
_____1280x960: Dotted
_____2560x1920: Not dotted

Capture Speed/Integration
_____Fast1: 16 frames
_____Fast2: 32 frames
_____Slow: 40 sec.
_____Auto Increment: Checked
_____Captured Memory Overwrite: Not checked.

Screen Mode
_____Standard: Dotted

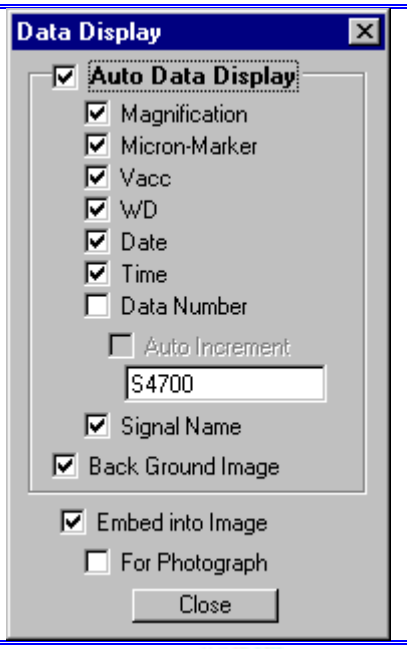


Data Display **[Setup → Data Display]**

Auto Data Display: Checked.
_____Magnification: Checked.
_____Micron-Marker: Checked.
_____Vacc: Checked.
_____WD: Checked.
_____Date: Checked.
_____Time: Checked.
_____Data Number: Not Checked.
_____Auto Increment: Not Checked and S-4700 displayed.
_____Signal Name: Checked.
_____Back Ground Image: Checked.

_____Embed into Image: Checked.
_____For Photograph: Not Checked.

*Boxes checked here will be displayed on default screen



Pseudo Color **[Image→ Pseudo Color]**

Pseudo Color.
_____Color Mode: Off.

FLASHING

Flashing is a procedure for removing excess gas molecules that have been adsorbed on the surface of the filament tip. By applying a burst of heating current these molecules are blown off the tip. However, this effect causes an initial period of instability in the emission. The emission current will drift lower for periods lasting for 60 to 75 minutes as the tip stabilizes viz. re-establishing a uniform initial coating of gas molecules. The emission current must therefore be reset periodically to maintain the correct emission current – otherwise the current can drift to 'zero' and signal/noise is very poor. Please be attentive. The instrument determines when flashing is required - when "PLEASE FLASH" is seen blinking in the upper left of the Scanning Image window. Flashing is typically required when the extraction voltage is maxed out for a given operating voltage. The operator must flash with 20 minutes or so – otherwise the tool automatically shuts off the HV.

NOTE: The flash current is displayed under the emission current (Ie), This occurs quickly, so be prepared to note it and record in logbook.

1. Click on the HV indicator, this is identical to Setup/HV Control for the menu bar. This will open the HV Control window.
2. Click Flashing to open Flashing Execute.
3. Click Execute.

The goal is to have a flash current between 20.0 - 40.0 μA.

NOTE: After flashing, the emission current will drop for a period of time as explained above. This will last until a mono-layer of molecules forms on the surface of the filament. It will be necessary to reset the emission current when it drops 2 μA – 3 μA below the set emission current.

SAMPLE PREPARATION GUIDELINES

Preparation of non-standard samples/specimens for imaging and/or microanalysis in the S-4700 must first have been reviewed by either Kevin Nordquist, Stefan Myhajlenko or any NanoFab staff.

At no time should sample, sample stub, and dove tail base be handled without using protective gloves. This procedure is a requirement to minimize sample/chamber contamination.

Samples must be cleaned in a manner compatible with its composition, additionally all parts of the sample stub assembly must be cleaned. NanoFab provides three general-purpose solvents for sample cleaning, isopropanol, methanol, and acetone in the Cleanroom. Use N_2 to dry samples and all components as may be required.

The Center provides carbon tape for mounting samples upon the stub. Alternatively, colloidal graphite can be used for mounting samples – this requires thorough drying before loading into the load lock. The operator should have a good understanding of the mechanical properties of their sample – colloidal graphite may be preferable to carbon tape.. Furthermore, if additional experiments are required of the sample to be imaged, the operator should establish a plan. This plan details the sequence of imaging and experiments to be done with a particular sample.

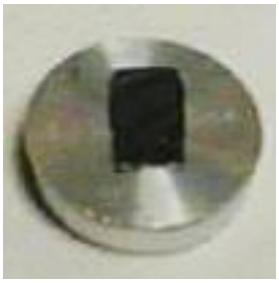
This is important considering that repeated mounting and removal of the sample increases the probability that the sample could be destroyed. It is therefore imperative that the operator be proficient in the mounting and removal of samples. The section below details some important points for both methods of mounting samples.

COLLOIDAL GRAPHITE

Colloidal graphite is a paste consisting of graphite suspended in isopropanol. Placing a small drop in the center of the stub, as seen here, is enough to adhere at most 1 cm by 1 cm samples to the stub. Naturally this will vary depending on the size of the sample.

Using tweezers apply slight pressure to the top of the sample, if the right amount of graphite is used, a small bead will appear around the interface of the stub and sample. If this bead exceeds the height of the sample, too much graphite was applied. Carefully remove excess graphite with a swab or cleanroom wipe; this will reduce the dry time you must wait.

To ensure the sample is secure to the stub, place a small drop of graphite at each corner of the sample. Specifically covering the top and side of the sample, in addition to the stub. This process will aid in the dissipation of current from the sample surface of insulating materials.

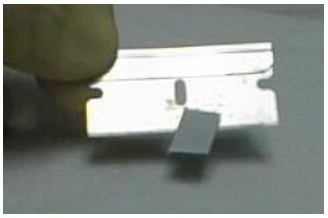


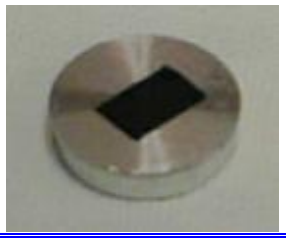
At this point the sample should be allowed to dry for a minimum of 20 minutes. This will help avoid possible problems with out-gassing that can degrade the vacuum. The exact time to dry will depend on the amount of graphite used. The graphite under the sample will take the longest to dry completely. To accelerate the drying process, you can (if safe) place the sample directly under an incandescent light source (it provides some heat) or within a desiccate jar.

Advantages	Disadvantages
Will hold sample for long duration	Removing sample from stub is often messy.
Aids in dissipating charge	20 – 30 minute wait time for sample to dry.
Compatible with most samples	

CARBON TAPE

The second method uses carbon tape to secure the sample to the stub. A roll of 8 mm or 12 mm wide tape is kept in the dry box with the colloidal graphite. Using the razor provided cut a piece of tape that is just slightly larger than the sample. Center the tape on the stub, with tweezers apply slight pressure to both ends. Please note, it does not require significant pressure to adhere the tape to the sample. The tape is rather sticky and can be difficult to work with. Excessive pressure upon the tape will result in stub cleaning to be more problematic.
Remove the white backing from the tape and discard into the trash. For larger size samples, simply cut two pieces of approximately the same size and place side by side.





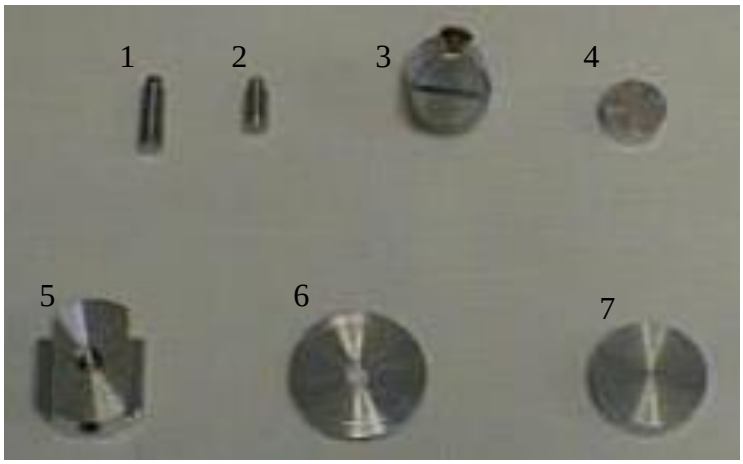
Using tweezers align the sample with the center and gently place on the carbon tape. Open tweezers and simultaneously apply a slight pressure to both ends of the sample. Again it is important not to apply excessive pressure -this will prevent difficulty in removing the sample from the stub.

Advantages	Disadvantages
Can hold sample indefinitely.	Removing sample from stub can be very difficult.
Quick sample prep time.	Not recommended for brittle samples.
	Does not aid in dissipating charge from surface.

SAMPLE STUB ASSEMBLY

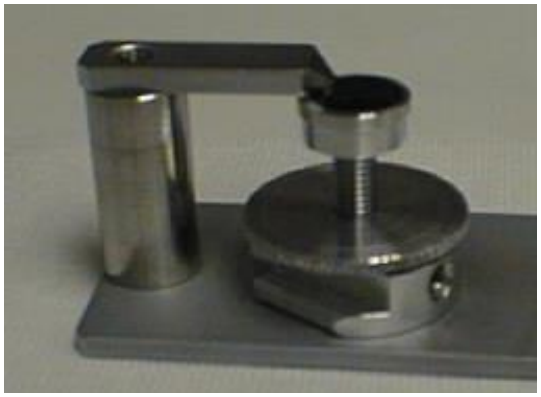
In the figure to the right are the three basic sample stubs and stub assembly components used in the majority of SEM work done on the S-4700:

1) Long threaded post.
2) Short threaded post.
3) Cross-sectional sample stub.
4) 15 mm sample stub.
5) Dove tail assembly base.
6) Locking washer.
7) 32 mm sample stub.



These components are within the dry-box located on the sample prep-bench. Only a limited number of stubs will be kept there at any given time, additional stubs can be made available upon request. Users will be responsible for cleaning and returning sample stubs/holders to dry-box.

Specimen height should be adjusted carefully. The top surface of the specimen must not extend above the bottom of the level gauge on the sample jig. The entire assembly of specimen, specimen stub, and stub holder must fit within the gap of the sample jig. Stage control is based partly on the assembly height: this is to prevent a collision with the specimen and any component in the chamber. Otherwise, when the stage is tilted or working distance is decreased, the specimen may collide with the objective lens and cause damage to both. Correct use of the jig aids in repeatability of obtaining high-resolution images by ensuring sample height is at 12 mm working distance default.



1. Screw the post into the bottom of the sample stub, ensure it is tight.
2. Take the assembly stub base and ensure the larger surface area is down, place locking washer on top.
3. Align base and locking washer, screw post through locking washer into base a short distance only, do not tighten locking washer.
4. Check height with sample gauge and align the highest point of the specimen with the jig. Then tighten the lock screw.

The height of specimen can be adjusted. Loosen the lock washer.

1. If the height is greater than the gauge, rotate the sample stub between an eighth and a quarter of a turn clock-wise. Check height after each turn. Once the gauge height is obtained, lock washer.
2. If the height is less than the gauge, pinch the post with thumb and index finger and rotate slightly counter clock-wise. Check height after each turn. Once the gauge height is obtained, lock the washer.

TIP: Obtaining the gauge height is quicker if you start with the sample height slightly high and lower it to the proper height.

SAMPLE LOADING PROCEDURES

The proper loading of the sample holder is paramount. To prevent any damage during loading, the following two conditions must be strictly adhered to.

- The operator will verify that the stage is in the home position prior to placing the sample holder into the exchange chamber.
- The specimen gauge jig must be used to ensure specimen meets proper height requirements before any attempt to load the sample. This is regardless of the type of sample, cross-sectional or planar. (See P. 23)

Once the stage position and sample height is confirmed proceed to loading sample into chamber.

1. Turn the Chamber-view camera and monitor on.
2. Vent the Sample Exchange Chamber of atmosphere. Press AIR.
3. Grasp exchange chamber housing. Swing open till housing is locked into position. An audible click will be heard.
4. With one hand still on the housing, unlock the exchange rod by slightly pushing it forward.
5. Align the male end of the rod with the threaded female sample base. Screw the two together, taking care not to cross-thread, only finger tight.
6. Place one hand on exchange chamber housing, the other hand on the end of exchange rod. Draw the rod back into the locked position. Swing the exchange housing to the closed position.
7. Evacuate the exchange chamber. Press EVAC.
8. Wait till Sample Exchange Chamber high vacuum indicator is green and reading 2 Pa or less. For most semiconductor samples (without photo resist) this takes approximately 60 seconds. At this time the exchange valve indicator light will be flashing yellow.
9. Before you move the MV-1 valve 180 degrees counter-clockwise to open the gate valve, **unlock the sample holder on the rod**, and hold in place momentarily.
10. Open MV-1 gate valve.
11. You should be able see your sample on the monitor – slowly push the rod into the chamber and onto the stage (slight resistance).
12. The sample is completely mounted when the Teflon exchange rod guide is flush with the face of the exchange housing. .
13. The exchange rod and sample are disconnected when the Teflon guide no longer moves away from the housing as the rod is being unscrewed. Unscrew exchange rod.
14. Look through the exchange-housing window. Pull exchange rod back to a point where the rod will clear the MV-1 gate valve. **STOP** - Close MV-1. Pull rod back remaining distance and lock. This procedure ensures that the sample chamber is completely isolated in the unlikely event that the exchange rod o-ring seal should fail.
15. Switch GUN VALVE toggle to AUTO (yellow light will stop flashing) and move to FESEM console.

OBTAIN AN IMAGE

The S-4700 has two image magnification modes.

The high magnification mode is indicated by the lack of LM in the magnification indicator window. It is necessary that the instrument be in the high magnification mode to make adjustments to the Column SetUp and Alignment. The magnification range in this mode is x250 to x500k.

The low magnification mode is indicated by the LM in the magnification indicator window. This mode is used for navigating to areas of interest on the sample. The magnification range in this mode is x35 to x10k.

NOTE: Ensure that the instrument is in the high magnification mode at this time

Set Sample Size

Find the top of the Stage Control [5-axis] window.

CAUTION:

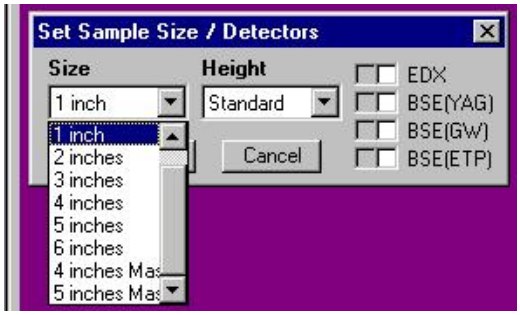
This control limits the movement of the sample within the chamber. It is critical that these limitations are set correctly; failure to do so could result in serious damage to the instrument.

SIZE.

1. Select the appropriate sample stub diameter from the pull-down menu. NanoFab provides two sample stub sizes, 1 inch and 15 mm. For the cross-sectional sample stub the 15-mm diameter size should be selected.

HEIGHT.

Since the specimen assembly was prepared using the sample height gauge, select “Standard”.



Detectors.

NOTE: These boxes should not be checked. These controls are for advanced users doing X-ray analysis or back-scattered imaging.

The following procedure sets the proper stage and lens conditions that allow the sample to be imaged. It is only from a basic low magnification image can the area of interest be obtained and further high-resolution imaging be accomplished. Fine-tuning of these settings will certainly be necessary in order to obtain the best possible image. This will depend largely on the students understanding of their sample (primarily feature size), basic SEM principles, and the final resolution required.

ELECTRONIC WORKING DISTANCE – COLUMN SETUP

SEM conditions are set in the dialog window

OPERATION MODE:

The five operation modes should be selected according to the working distance and purpose of imaging.

1. Ultra High Resolution. To be used for observation at the highest resolution with a corresponding working distance of 6 mm or less.

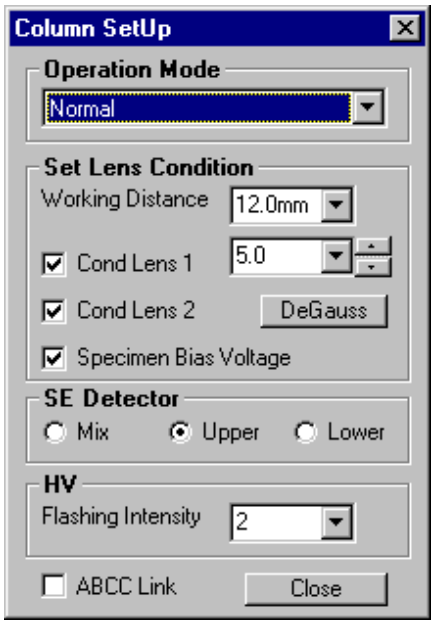
2. Normal. Recommended operational range: 6 mm to 15 mm working distance.

3. Long working distance. To be used for working distances of 15 mm or more, for greater depth of field.

4. Analysis. Used for X-ray analysis and backscattered imaging.

5. Magnet Sample. Used for magnetic specimens.

A majority of users will want to select the high-resolution mode selected. In this operational mode a resolution of 2.5 nm is possible.



SET LENS CONDITION:

1. Working Distance. This is commonly referred to as the distance from the bottom of the pole piece (objective lens) to the top of the sample. The working distance must be set within the range of the selected operational mode.

2. Condenser Lens 1. This should be set at 5.0 and only experienced users should make changes.

3. Condenser Lens 2. This must be checked.

4. Specimen Bias Voltage: This should be checked and only experienced users should make changes.

SE Detector.

This instrument is equipped with two secondary electron detectors; the upper detector is just above the objective lens and the lower detector is located in the specimen chamber. The different signals can be selected independently or mixed. The operator should select the detector best suited for the specimen under observation.

1. The upper detector will provide high resolution of the specimen surface, since the signal is comprised of genuine secondary electrons (an ExB filter is used to energy select). Thus, a short working distance provides the best results.

2. The lower detector receives a signal comprised of secondary and backscattered electrons, providing contrast of specimen surface topology. Using a working distance greater than 7 mm provides the best results for these types of observations – otherwise signal/noise deteriorates with loss of line of sight.

3. The mixed detector provides characteristic of both detectors, however one detector will dominant the other depending upon the working distance.

MECHANICAL WORKING DISTANCE (Z)

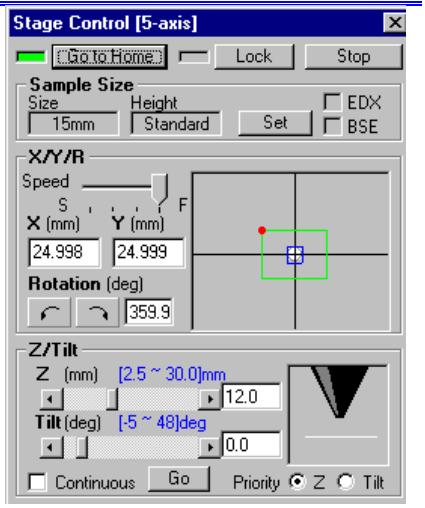
Z/TILT

The selection of the Operation Mode and working distance will determine the crossover point for focus. It is therefore important to have the specimen at this location in order to obtain an image. In which case: The electronic working distance should closely match the mechanical working distance.

1. Locate the Z/Tilt section of the Stage Control window.
2. Set the working distance or Z so it matches the distance set in the Column SetUp working distance selection. This can be done using the slider selector or by typing in the distance.

CAUTION: The stage does not physically move till the **Go** button is selected. For working distances below 6 mm, after **Go** is clicked move the cursor to the **Stop** button in the upper right of the widow and observe the chamber monitor. If the stage does not stop or appears to go beyond the desired working distance, click **Stop**.

3. Once the stage is at the desired working distance the operator has the option of locking the stage. This will provide a better image at higher magnification by reducing transmitted turbo pump vibrations to the stage. However this action disables movement in the Z-axis and the ability to tilt the stage. Those options gray out. Click the **Lock** button at the top of the Stage Control window.



NOTE: Tilting the sample is not required to obtain an image. Only experienced users can change both the Z and tilt simultaneously.

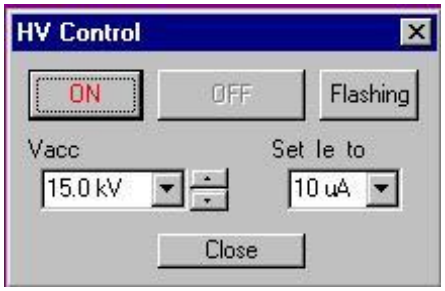
When the stage lock is engaged – you will hear a thud. The mechanical lock will cause temporary drift of the stage and most likely a loss in focus. You will have to re-focus after the stage drift stabilizes. The locking mechanism is engaged while the box is red. To unlock, the operator should click **Release.** Click on the **Go to Home** button, this will automatically return the stage to its home position for sample exchange.

APPLYING HIGH VOLTAGE

HV CONTROL

1. To open the HV Control window (lower right), click within the HV indicator window (upper right). Both windows can enable the high voltage (ON), however it is only within the HV Control that the acceleration voltage and emission current are adjustable. (ON will change to SET after voltage is on)
2. The acceleration voltage (V_{acc}) is adjustable according to the needs of the operator from the pull-down menu. **It is the responsibility of the operator to use accelerating voltages that are appropriate for their sample.**
3. The emission current (I_e) is adjustable according to the needs of the operator from the pull-down menu. **It is the responsibility of the operator to use emission currents that are appropriate for their sample.**

NOTE: Before acceleration voltage is enabled a warning to verify the sample size will be displayed. This warning reminds the operator of the importance of having set the proper sample size and height. With these condition properly satisfied a fail-safe is established that prevents the specimen from coming into contact with other objects in the chamber.



4. In the HV Indicator window note the extraction voltage (Vext) and record it on the log sheet.

After the HV has completed ramping on you should have some type of image that may or may not be in focus. Switch the magnification mode to low; this should reduce the magnification to x35.

If no image is seen, the contrast or brightness may not be adjusted correctly. Locate the **Auto Contrast and Brightness Control** button,  in the Auto section of the Scanning Image Screen. This button provides a good baseline contrast and brightness for imaging; however do not use it exclusively to control contrast and brightness of images. Slider controls for contrast and brightness are located directly above the Scanning Screen. These allow for a fine degree of control to give the best results.

Then use the analog control pad, which has four knobs; Alignment/Stigmation (X &Y), Course Focus, and Fine Focus to continue fine tweaking of the image. Use the Course Focus and then Fine Focus so that some feature on the sample can be identified, this will enable you to orientate your sample and establish the current location being imaged. The ease of this step will be determined by the feature sizes on your sample. Smooth surfaces are more difficult to image at this point than rough surfaces.

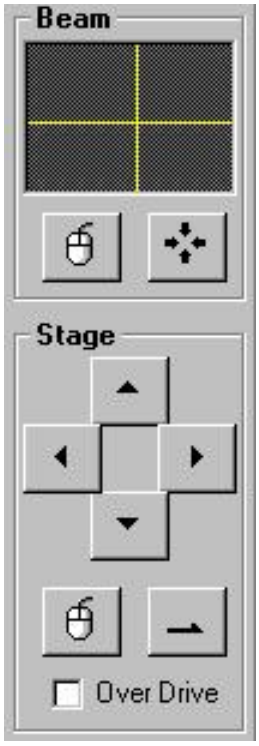
Magnification is adjusted by placing the cursor over the magnification indicator window, left click and hold. Moving the cursor to the left or right will decrease or increase the magnification respectively.

STAGE MOVEMENT

There are a number of methods for controlling the stage to observe the area of interest.

BEAM

This control, rather than moving the stage, alters the location of the beam raster. The yellow axis indicates the current location of the raster for a given area. By clicking the mouse button, a yellow hand will appear when the cursor is above the current image. This hand will allow for the operator to click and drag an area of interest to the center of the screen. The button with arrows pointing center, will re-center the raster. This method of specimen movement works best at very high magnification, when fine adjustment is required.



STAGE

This control physically changes the location of the stage in the X and Y direction by clicking on the arrow button for the desired direction. The mouse button is similar to the one described above, however the hand that appears over the image does move the stage.

Typing the coordinates into the X-axis or Y-axis indicator located in the Stage Control window can also alter the stage position: however this method is not very practical for most applications.

ROTATION

Rotation of the stage is achieved by entering the desired degree of rotation into the indicator window or the curved arrows. This control is located within the Stage Control window.

Once the general area of imaging is located, change to high magnification mode and ensure the lowest magnification is set for this mode. This is generally x250 or x500, depending on the working distance.

Course focus and then fine focus.

HIGH RESOLUTION IMAGING

The methodology for going from low magnification to high magnification/resolution is straightforward. The level of magnification which this methodology begins depends largely on feature size of the sample, however x500 or x1k is a good starting point. Generally at lower magnification, when the Course/Fine Focus is adjusted the image will shift. This is a result of the objective aperture not being properly aligned. This motion can also occur when adjusting the stigmation and this alignment is not properly adjusted. The following alignment procedure corrects these conditions.

ALIGNMENT

When any of the alignment modes, **Beam Align, Aperture Align, Stigma Align X/Y**, are dotted, the left two knobs (**Stigma/Alignment X/Y**) on the analog control pad are strictly for Alignment correction.

BEAM ALIGN

A circular image will appear, place the image in the center of the target using the **Alignment** knobs.

APERTURE ALIGN

This process is to adjust the center of the objective lens; this is a critical step in obtaining a quality image.

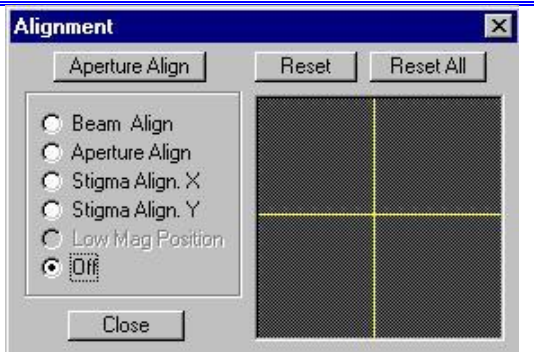
1. Find a unique feature on the specimen; relocate it to the center of the screen and focus.
2. Click the **Aperture Align**, the focus conditions will change periodically causing the image to move.
3. Adjust the **Stigma/Alignment** knobs to minimize the motion of the feature. At lower magnifications control of this motion will be limited to the center of the image.

STIGMA ALIGN X/Y

This alignment procedure operates in the same fashion as **Aperture Align**; however image motion will generally not be noticeable until 50KX mag. When minimizing the motion of **Stigma Align X** use both X and Y knobs on the analog pad. This condition applies for **Stigma Align Y** as well.

OFF

This setting stops all alignment adjustment and returns the **Stigma/Alignment** knobs to stigmation adjustment. After the initial alignment procedure, fine focus as necessary.



TIP: Adjustment will be facilitated by displaying the cross-hairs cursor with **AREA MARKER** turned on by checking the box in the image window.

TIP: If a particular alignment mode is difficult to adjust click **Reset**. This should cause considerable motion of the image, but is a good starting point. Only experienced users should use **RESET ALL**

NOTE: That the **Low Mag Position** is grayed-out. This adjusts the visual field at low magnifications in the Low Magnification mode.

NOTE: The dark field with yellow cross hairs is the digital equivalent to the analog knobs. This feature is difficult to use and lacks the sensitivity of the analog input.

MAGNIFICATION

The S-4700 has the ability to make large jumps in magnification without losing much resolution. However incremental increases in magnification between focus, alignment, stigmation adjustment is preferred and will provide the best resolution.

Increase magnification to x10k, by left-clicking and moving the cursor across the indicator window.

Fine focus and repeat the above alignment procedure.

STIGMATION

Astigmatism in the electron probe is a polarization in either X and/or Y direction of the image with changes in focus. In essence the probe is not Gaussian in shape. It is most noticeable with circular or curved features that elongate. Astigmatism is caused by aberrations in the magnetic lenses.

This problem is corrected with stigmators; these coils are controlled by the **Stigma/Alignment** knobs on the analog pad. After fine focusing (i.e. getting best possible image), adjust the image with the X and Y stigmator knobs. The idea is to improve the quality of the image by moving the knobs clockwise and counter clockwise slowly till the image becomes clearer. Adjust one knob at a time can help facilitate this process – in conjunction with fine focus adjustments.

NOTE: For magnification > 50KX, it is critical that the stigmation alignment done in the alignment procedure is performed as best as possible. This will ensure a better response from the stigmator coils.

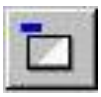
Fine focus and continue to increase to the target magnification – repeat above procedure as necessary.

SCAN SPEED

Once a target magnification is obtained, the scan speed can be reduced to improve image clarity. For simplicity all work has been conducted in the **FAST1** scan speed. There are six different scan speeds available; however the three below are the most useful.

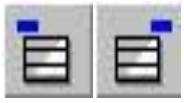
FAST1

Used for normal operations; increase magnification, alignment, focusing, and stigmation. Additionally, frame by frame micrographs can be taken in the fast scan speed, but this is rather difficult and time consuming.



SLOW3/SLOW4

Used to view the image with a much higher degree of clarity, either speed is recommended for high quality micrographs.



METHODOLOGY SUMMARY

This summary provides a simple step-by-step guide of the methodology described above, starting from a magnification of 35X in the low magnification (L) mode.

1. Coarse Focus, with analog input.
2. Fine Focus, with analog input.
3. Change to high magnification (H) mode.
4. Adjust brightness and contrast with ABCC, if necessary.
5. Coarse and/or Fine Focus.
6. Increase magnification to 500X or 1KX.
7. Fine focus.
8. Adjust Beam Alignment.
9. Turn on Area Marker.
10. Center a unique object.
11. Adjust Alignment, minimize motion.
12. Adjust Stigmation Alignment X, normally no motion is seen below 50KX.
13. Adjust Stigmation Alignment Y, normally no motion is seen below 50KX.
14. Alignment Off.
15. Fine Focus.
16. Increase magnification to 5KX.
17. Fine Focus.
18. Repeat steps 8 thru 14.
19. Fine Focus.
20. Adjust Stigmation X.
21. Adjust Stigmation Y.
22. Fine Focus.
23. Repeat steps 16 thru 22 until the desired magnification is reached.

IMAGE CAPTURING

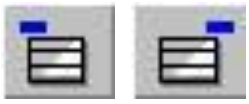
Images captured on the S-4700 are to be saved to the IMAGES folder on the FESEM PC desktop:

The path is C:\WINNT\PROFILES\ADMINISTRATOR\DESKTOP\IMAGES.

After your session is over you must FTP the images to the Gateway PC c/o FileZilla - you will need to use either your ASURITE or ASUAD to login. Hostname is fesem-ex.eas.asu.edu with Port 990.

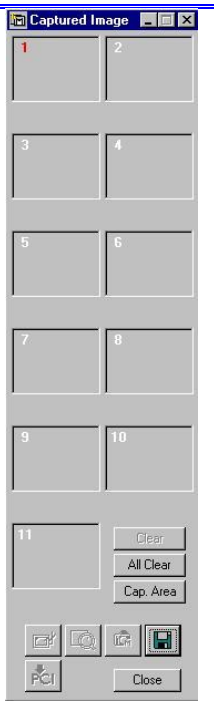
The corresponding folder on the EDX desktop is called DATA. Both these folders are for temporary use only – once you FTP the files to the Gateway PC – you should delete them. These folders will be checking periodically and files older than two months will be deleted. You will also need to download WinSCP onto your lap-top/PC to retrieve your files from the Gateway PC.

1. When the operator is satisfied with the quality of the image, click either (slow 3) or (slow 4) for recording scan speed, this is particularly useful for high-resolution images. The actual recorded scan speed can be selected from the Setup Image menu – 80s scan speed typically results in good quality images if your sample and stage are stable.



2. To capture an image the operator clicks the capture execution button from the Scan tool bar on the Scanning Image window.

CAPTURED IMAGE



Upon completion of the scan, the Captured Image window will open. This provides a location for captured images; it is possible to store up to eleven images before it becomes necessary to save the images to the Image Manager.

 Saves images

Clear: Clears image data (after operator confirmation)
All Clear: Clears the image memory.
Close: Terminates dialog.

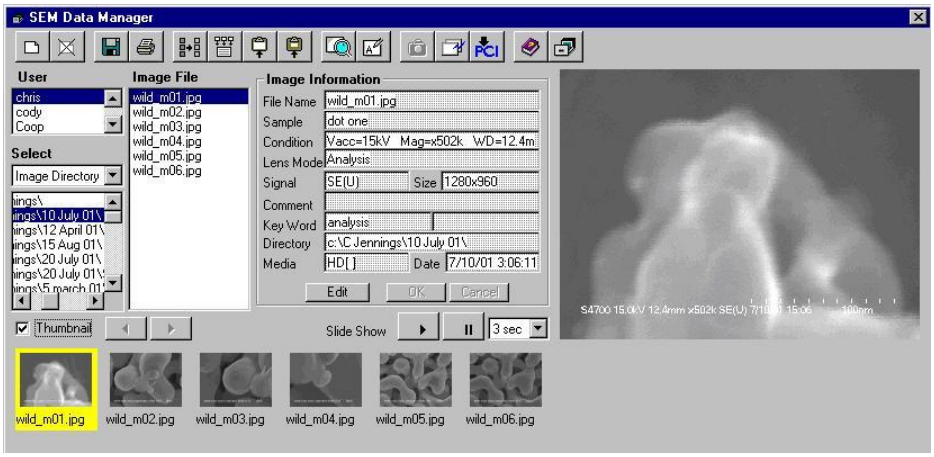
SAVING IMAGE

To save, click the SAVE button in the Captured Image window. This opens the Save Image window.

Before saving, arrange the following conditions.
Input File Name.
Select File type.
Select drive: C:\WINNT\PROFILES\ADMINISTRATOR\DESKTOP\IMAGES
Select user from among the already registered ones.
Save Option: Save all.
Click Save.

SEM DATA MANAGER

This opens the SEM Data Manager window, identical to File/Open/Image Manager in the menu bar. This window allows you to transfer to PCI and print images, in addition to viewing the text data that accompanies each image.



Once the image manager is opened, click the upper left button (Make New User) in the Image Manager window. The input window will appear. Enter user name (8 characters), click OK.

REVISION HISTORY

Effective Date	Originator	DESCRIPTION OF REVISION	Issue
12/12/2001	Chris Jennings	Initial Release	A
5/24/2005	Andy Ngo	Update Contact Information	B
4/25/2006	James Barfoot	Update Contact Information	C
1/22/2008	Stefan Myhajlenko	Update Contact Information	D
9/30/2009	Stefan Myhajlenko	Update Contact Information	E
6/21/2012	Stefan Myhajlenko	Update Contact Information	F
4/2/2015	Stefan Myhajlenko	Update Gateway PC File Transfer Protocol	G
1/28/2020	Kevin Nordquist	General update for contacts and organizational names/links	H
7/16/2024	Kevin Nordquist	Rearrange sections to follow video flow	I