Standard Operating Procedure for the JEOL JSM-840A SEM

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Central Facility for Electron Microscopy

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This work is intended a guide to the operation of the **JSM-840A SEM** by the average user. It details

- Sample loading
- Bringing the microscope up to operating conditions
- Alignment of the microscope
- Sample removal
- Returning the microscope to a powered down state
- Relevant safety procedures

A more detailed and thorough account of these procedures as well as a more advanced ones can be found in the JEOL Operating Manual provided by JEOL.

This work is under constant revision and all users are encouraged to contribute any material for review by facility staff.

Emergency Operating Procedure

If a user ever has a problem that he/she is even slightly unsure about, **ASK** someone who knows and can help. There are no penalties or punishments for asking for help but there may be for not reporting damage to equipment which may delay or prevent others from working.

If, at any time, the user perceives a problem that he/she is unable to remedy, the user is to call or find the following individuals:

Lab Manager	12C Walter Scott Engineering Center		2-8762
Faculty Supervisor	318 Walter Scott Engineering Center	2-8762	2-8308

If neither of these individuals are available then the user should do the following:

1. Filament Knob (65) is fully counterclockwise
2. Accelerating Voltage Button (63) is off (unlit)
3. SE brightness (48) is turned fully counterclockwise

If a dire emergency is evident (smoke, fire, sparks.....) the user should pull the main electrical box switch (behind console) to turn off power to entire microscope and notify the proper emergency personnel.

Then, if possible, the user should stay with the microscope while trying to contact the above individuals. If it becomes necessary to leave the microscope then the user should leave a large, legible note on both the microscope and at least one of the above individuals desks stating

- The problem
- When it occurred
- User name and phone number

Qualification Procedures

To become eligible to use the **JSM-840A SEM** it is necessary for the user to do the following:

1. Register desire to become a user with the Laboratory Manager
2. Read and understand the
    - Emergency Operating Procedure
    - Standard Operating Procedure
    - Scheduling Policy
    - Charging Structure
    - Facility Use Policy
3. Pass a written examination on the use of the microscope
4. Pass a "Driving Test" on the microscope with either the Laboratory Manager or Faculty Supervisor present

No persons shall use the microscope without first becoming a qualified user by completing the above steps. **Any person using the microscope who is not a qualified user will be banned from the facility for an indefinite period of time, as determined by the Laboratory Manager or Faculty Supervisor.** This policy, although harsh, is intended to prevent damage to equipment and the subsequent loss in productivity by other users and expense to the center and university.

I Sign in the User-Log with the following information

- Read any previous comments made by the last few users
- Username
- Date
- Time on
- Material being examined including number of specimens
- Type of work to be done (SEI, BEI, EDS, ECP....)
- Vacuum at start

II Loading of the Sample

- *Use only one gloved hand when handling samples and holders*
- *Avoid letting the sample holder or any part of the sample exchange rod touch non-clean surfaces which may be contaminated with hand-oil*
- *Never "blow on" or exhale on samples to dry them, use the IR lamp instead*
- *Always make sure all screws are tight and that you always have a sure grip*
- *Always ask if you have a question*

Placing a Sample in holder

1. Select a holder just large enough to hold the sample
2. Using your one gloved hand place your sample in the holder and position it so the top of the sample is flush with the top of the sample holder.
3. Secure the sample in the holder using set screws and/or carbon paint/tape
4. Ensure a good conductive path from semi-conducting or insulating samples to holder by using carbon paint/tape
5. Allow any carbon paint used to dry under the IR lamp for 2-3 minutes

Inserting Sample (in Holder) into Microscope

6. Set the microscope to the following settings

    - X-control 25mm
    - Y-control 35mm
    - Stage Height (Z-control) 39mm
    - Tilt 00.0 °
    - Rotation 000 Graduates
    - Accelerating Voltage(63) Off (unlit)
    - Filament(65) Fully CCW

7. If using a sample holder larger than 32mm in diameter skip to step 8a below
8. Using your one gloved hand push the large circular plastic plate on the sample exchange rod to the end of its travel (furthest from the orange handle-knob), locking the clips onto the plastic plate assembly.
9. Using your one gloved hand screw the sample holder onto the sample exchange rod
10. Place the rod-holder assembly in to the airlock chamber, verifying the plastic plate is flush
11. While holding the end of the rod with one hand use the other to depress the Vacuum Operation Button on the top of airlock
12. The button should stay lit during the airlock pump-down, approximately 1 minute
13. When the airlock is at sufficient vacuum for exchange the light should extinguish
14. Grasp the Specimen Exchange Chamber Isolation Valve Knob and rotate it towards you (CCW) 90 ° and then pull it to the right (away from airlock) until the valve is completely open and you cannot pull any further. Then rotate the knob 90 ° away from you (CW) to lock the isolation valve in the open position
15. Look into the chamber through the plastic plate on the exchange rod, there should be a light on inside, illuminating the stage. If there is no light confirm the settings in step 6; if there is still no light then contact the Lab Manager. If no help is available then skip to the section on removal of the specimen and get help afterwards.
16. Push the handle of the exchange rod so as to insert the holder into the chamber, being careful not to strike the airlock door or any other features inside the microscope
17. Insert the holder onto the stage being sure to lock the dovetails together and being sure that the holder is pushed back as far as possible

18. Unscrew the rod from the holder by turning the orange handle-knob CCW for a large number of turns, being certain that when finished it is fully disconnected from the holder.
19. Withdraw the rod, locking it in its fully retracted position, secured with the clips on the plastic plate
20. Grasp the Specimen Chamber Isolation Valve Knob and rotate it 90 ° towards you (CCW) to unlock it from the open position
21. Verify that the specimen exchange rod is fully withdrawn and slowly close the isolation valve by sliding it to your left (towards the airlock). After the valve is fully to the left then rotate the Knob 90 ° away (CW) from you to lock it in the closed position
22. While holding the exchange rod depress the Vacuum Operation Button to vent the airlock chamber to atmospheric pressure. After venting is complete the rod will be loose, remove it from the airlock using your gloved hand and push the plastic plate towards the orange handle-knob. Place the rod back it its holder (tube beside the chamber)

Larger Sample Instructions

8a Depress the Specimen Vent Button on the panel on the bottom of the microscope and wait for the chamber to vent
9a When the chamber is vented the door will "pop" open and you should hear gas escaping
10a Grasp the bottom and top of the chamber door and pull slowly, being careful not to strike any features inside the chamber
11a Using your one gloved hand place the sample holder onto the stage making sure it is secured
12a Slowly close the door, being careful not to strike any features inside the chamber

13a While holding the door closed depress the Specimen Vent Button, the chamber should begin to pump down (approximate time 5-10 minutes)

III Bringing the Microscope to Operating Conditions

- *Never turn the filament knob past its limit and always turn it slowly (2 minutes for full range)*
- *Never move any of the stage controls past their limits*
- *Never tilt a sample beyond the specified limits indicated on the specimen chamber door*
- *Never operate the microscope if the pressure rises to a level >1x10^{-6} torr as read on the gauge in the back of the microscope*
- *Always ask if you have a question*

1. Examine the panel located on the bottom of the microscope column and check that none of the error lights (Red LEDs) are lit. If any are lit then refer to the emergency procedures section for instructions
2. Adjust the following controls to the indicated settings:

• Character-mag/off switch(1)	**Mag**
• Character-white/off/mask switch(2)	**White**
• WFM(3)	**Lit**
• CRT Contrast Knob(7)	**Orange Arrow**
• CRT Bright Knob(6)	**Orange Arrow**
• IMS-SEI(L,R) buttons(9,10)	**Lit**
• SE Image-Contrast Knob(47)	**8**
• Scan Mode-Pic Button (37)	**Lit**
• Scan Speed-SR Button(41)	**Lit**
• EOS mode-SEM Button(54)	**Lit**
• Magnification(45 or 46)	**100x**
• Gun Bias Switch(29)	**Auto**
• Detector(30)	**SEI**
• Detector-PMT Link(32)	**On**
• Detector-Collector(33)	**On**
• Probe Current Coarse Knob(61) and Fine(62)	**Orange Arrow**
• Alarm/AEM switch (right side of column)	**Alarm**
• Objective Aperture (on column)	**2**

3. Select the accelerating voltage desired by pressing the appropriate +/- buttons(64) on the console

4. Adjust the Coarse Focus Knob(57) to match the Stage Height (Z) displayed on the Stage Height Control Knob (should be 39mm at this point)
5. **Check that the pressure inside the chamber is < $1x10^{-6}$ Torr, if it is not then wait until it is**
6. Turn on the accelerating voltage by depressing the Accelerating Voltage Button(63)
7. Slowly turn the Filament Knob(65) CW until the stop is reached, it should take about 2 minutes to completely turn up
8. While rotating the filament knob watch the CRT and this signal it is displaying. The signal should be a line whose level will rise as you reach the upper limit on the Filament Knob(65)
9. Use the SE Image-Brightness(48) and Contrast(47) Knobs to adjust the signal so its peaks reach the uppermost line and its lowest signals reach the lowermost line with the average level between the 2nd and 3rd lines from the bottom. This will insure proper contrast to produce good quality images
10. Depress the Normal Button(5) which will then display a SEI image of the specimen
11. Translate the sample until its highest point comes into view (as determined before inserting the specimen into the microscope)
12. Adjust the Coarse Focus Knob(57) until focus is achieved (finest features observed)
13. Note the Working Distance displayed on the bottom right of the CRT, this is the real distance from the Objective Lens and the top of the sample. There may or may not be a difference between this value and the value displayed on the Stage Height Control, not this difference and take it into account when translating the specimen in the future.
14. Adjust the Stage Height Control to whatever is needed for the sample being examined (taking into account the differential in step) and adjust focus, brightness and contrast levels as per steps , and respectively

The microscope is now at operating conditions; proceed with the alignment

IV Alignment

Alignment of the Objective Aperture

1. Increase the Magnification(46) to well in excess of the level you wish to image at
2. Insert the appropriate Objective Aperture as indicated by the OPTI-AP display (23)
3. Depress the Wobbler Button(59) and adjust the amplitude to match the orange arrow
4. Depress the Beam-up Button(38) and adjust the cross-hair with the X and Y Position Controls(34,35) so the crossover rests near an easily discernible feature on the sample
5. Adjust the position of the Objective Aperture using the X and Y Control Knobs(on Column) on the aperture assembly. Only adjust one direction at a time and adjust them until the movement of the image stops in that direction. It may be helpful to press the D-Mag Button(4) during the procedure

Stigmation of the Image

6. Refocus with the Fine Focus Knob(58) to get the finest features in the image
7. Rotate the Fine Focus Knob(58) so as to be under and then over focused. If directional blurring is observed (at right angles to each other) then the image has an astigmatism.
8. Refocus with the Fine Focus Knob (58) and then adjust the Stigmator Controls (X and Y, 55 and 56 respectively), one at a time until the finest features in the image are observed
9. Repeat step, it may be helpful to increase magnification or use the D-Mag Button(4)
10. The microscope is now aligned, if problems still exist or you are unhappy with the image please refer to the troubleshooting section and if that fails, ASK!

V Removing A Sample

Powering Down the Microscope

- Never remove a sample with the voltage or filament on
- Always restore the stage to an untitled, unrotated and centered position before removal of specimen
- Always wear a glove on one hand when manipulating samples and holders

1. Change the following controls to the indicated settings:

    - X-Control 25mm
    - Y-Control 35mm
    - Stage Height(Z-control) 39mm
    - Tilt 0.00
    - Rotation 000 Graduates

1. Slowly turn down the Filament Knob(65) (CCW) until it is completely CCW, taking about 3 minutes to do so
2. Depress the Accelerating Voltage Button(63) and make sure it is unlit
3. Lower the Magnification(46) to its minimum
4. Turn the SE Brightness Knob(48) fully CCW

Removing the Sample Holder from Microscope

5. For samples or holder >32mm in diameter skip to step
6. Using your one gloved hand push the large circular plastic plate on the sample exchange rod to the end of its travel (furthest from the orange handle-knob), locking the clips onto the plastic plate assembly.
7. Place the rod in to the airlock chamber, verifying the plastic plate is flush
8. While holding the end of the rod with one hand use the other to depress the Vacuum Operation Button on the top of airlock
9. The button should stay lit during the airlock pumpdown, approximately 1 minute
10. When the airlock is at sufficient vacuum for exchange the light should extinguish
11. Grasp the Specimen Exchange Chamber Isolation Valve Knob and rotate it towards you (CCW) 90 and then pull it to the right (away from airlock) until the valve is completely open and you cannot pull any further. Then rotate the knob 90 away from you (CW) to lock the isolation valve in the open position

12. Look into the chamber through the plastic plate on the exchange rod, there should be a light on inside, illuminating the stage and holder. If there is no light confirm the settings in step; if there is still no light then contact the Lab Manager. If no help is available then skip to step
13. Push the handle of the exchange rod so as to insert the rod into the chamber, being careful not to strike the airlock door or any other features inside the microscope
14. Slowly insert the specimen exchange rod until the thread on the end of it matches the threaded hole in the specimen holder
15. Screw the rod in to the holder by rotating it CW being sure it is fully screwed in and tight
16. Withdraw the rod-holder assembly, locking it in its fully retracted position, secured with the clips on the plastic plate
17. Grasp the Specimen Chamber Isolation Valve Knob and rotate it 90 towards you (CCW) to unlock it from the open position
18. Verify that the rod-holder assembly is fully withdrawn and slowly close the isolation valve by sliding it to your left (towards the airlock). After the valve is fully to the left then rotate the Knob 90 away (CW) from you to lock it in the closed position
19. While holding the exchange rod depress the Vacuum Operation Button to vent the airlock chamber to atmospheric pressure. After venting is complete the rod-holder assembly will be loose, remove it from the airlock using your gloved hand and push the plastic plate towards the orange handle-knob.
20. Using your one gloved hand unscrew the sample holder from the sample exchange rod
21. Place the rod back it its holder (tube beside the chamber)

Larger Sample Instructions

6a Depress the Specimen Vent Button on the panel on the bottom of the microscope and wait for the chamber to vent

7a When the chamber is vented the door will "pop" open and you should hear gas escaping

8a Grasp the bottom and top of the chamber door and pull slowly, being careful not to strike any features inside the chamber

9a Using your one gloved hand remove the sample holder from the stage making sure to not strike any features inside the chamber

10a Slowly close the door, being careful not to strike any features inside the chamber

11a While holding the door closed depress the Specimen Vent Button, the chamber should begin to pump down (approximate time 5-10 minutes)

VI Shutdown Procedure

1. Be sure that all samples are removed from the microscope
2. Turn off the monitors on the Compaq and Kevex units (to the right of the console)
3. Sign off on the Userlog with the following information:
    - Date off
    - Type of work that was done (SEI, BEI, EDS, ECP....)
    - Film and other consumable used
    - Vacuum at finish
    - Accelerating Voltage Used
    - Emission current at saturation
    - **ANY PROBLEMS ENCOUNTERED, NO MATTER HOW SMALL**
4. Remove all samples and possessions from the SEM room and clean up used film...etc
5. Turn off room lights and shut door on way out
6. Fill out NCMN Charge Sheet and turn in to the Lab Manager or his/her mailbox (located across from the 2 computers in the main room of 12C)