

# JEOL JSM-6460LV

## Scanning Electron Microscope

**Warning:** This instrument may only be operated by those who have been trained by AAF staff and have read and signed the AAF laboratory policies.

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**Appendix I:** Back Scatter Detector

**Appendix II:** Low Vacuum Mode

**Appendix III:** Filament Replacement

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### **Basic SEM procedure/Imaging Instructions:**

#### **Login:**

SEM Imaging Computer (left monitor): no password

EDS Computer (right monitor): username: Labuser password: Abc123

#### **Sample Preparation, Mounting, and SEM startup:**

1. Only clean samples are allowed inside the analysis chamber. Use ethanol or isopropyl alcohol to clean oils and grease off of samples.
2. Sample holders are located in blue tool box.
  - a. Mount sample to holder. (ask AAF staff if unsure of which holder to use)
  - b. Measure length that sample extends beyond the sample holder. This value must be entered into software in next step so as to avoid damage to SEM.
3. Open SEM software. (SEM main menu)
  - a. Check in the software that the Z-height value (bottom of black screen) is approximately 45mm. If not, click on Z-value, type in 45, press enter key. Or user can Go to: "STAGE"-> "FILE" -> "EXCHANGE" (highlighted) -> "GO"
  - b. Go to SETUP → FUNDAMENTAL SETUP → AUTO FUNCTION (tab)

- Input z-height value of your sample above the sample holder (specimen height).
  - Click okay and close the stage control window.
- c. On the main software window click the STAGE icon → HOLDER
    - Choose the type of holder you are using.
    - In stage centered window blue line is sample height position, green line is sample holder position (blue line should be above green line).
    - Close window.
  - d. Go to desktop and open “IRCamera” software. Double click.
4. Press VENT CHAMBER button on front of SEM (remove sample door clamp before).
  5. After VENT CHAMBER light stops blinking, open the chamber door.
    - Do you need to use the Backscatter Detector (BSE)?
      - If yes, see that the backscatter detector is properly positioned below the pole piece (if unsure, see Appendix II *and* AAF staff).
      - If no, see that the backscatter detector is lifted away from the pole piece (if unsure, see AAF staff).
  6. Insert sample with the special black handle tongs or carefully by hand. NOTE: the sample holder can only be inserted one way (make sure sample is fully inserted). If it doesn't seem to fit, do not force it!
  7. Close the chamber door and clamp down.
  8. Press EVAC button on front of SEM and **allow to pump down for 7 minutes**.
  9. After 7 minutes, the high voltage “HT” icon at the top left of the JEOL SEM software should say “ready”.
    - a. If it does, click the icon.

### **SEM Imaging and Saving Image Files:**

10. Move sample to location under electron beam. Check sample (xy) position via stage icon (move Z to ~25mm or sample to at least 15mm below pole piece— see IR image).
11. Focus on sample at about 50X (coarse focus, then fine focus) and click “ABC” (Auto Brightness & Contrast)

12. Check working distance of focused sample.
13. Choose an accelerating voltage. To adjust, click on the “acc. volt” value near bottom of the screen and double click on the value you desire, **15kV normal** (Higher kV sharpens image, but possible damage to sample. Lower kV more surface detail).
14. Choose a spot size (generally 35). To adjust, click on the spot size value and adjust the slider bar.
15. Now that the sample is focused, working distance (WD) can be adjusted.
  - \*\*\*Make sure the WD (when focused on highest point on sample) is NEVER below 10mm. If it is, catastrophic damage to backscatter detector can occur\*\*
  - Based on the current WD value, adjust the Z-value so that WD will be 13mm after refocusing.
  - Emergency stop for stage movement is to push down on black joystick.
  - For example:
    - If currently Z-value = 45mm, WD = 22mm
    - Adjust Z-value to 36mm, refocus and check that WD now = 13mm
16. Sample should now be in focus and WD= 13mm.
  - a. Magnification can be adjusted as desired
  - b. Fine focus with knobs on control board.
  - c. Adjust X & Y stigmators with knobs on control board.
17. At top of screen there are four options for image scan rate (scan 1, 2, 3, 4)
  - a. Scan rate 2 is generally chosen for focusing on sample.
  - b. Scan rate 3 or 4 (slower) are chosen for image capture.
18. To capture an image, click the “Freeze” button. Then, save the image to your folder.
19. If image moves (up/down or left/right) while focusing, adjust wobble.
20. Should be able to get  $\geq 20,000\times$  magnification images.

### **SEM Procedures After Imaging is Complete:**

21. Return sample WD to 45mm.
22. Return sample to exchange position “Stage” -> “File” -> “Exchange” (highlighted) -> “GO”
23. Adjust magnification to 50,000X

24. Turn off electron beam by clicking “HT” button at top left of screen.
25. Press “VENT” on front of instrument. Wait until “VENT” button stops blinking and then can open chamber door.
26. Remove sample using special black handle tongs or carefully by hand.
27. Close chamber door and press “EVAC” again. (**Chamber must always be under vacuum except when installing or removing sample.**)
28. Fill out JEOL SEM Logbook with user’s name, PI’s name, sample type, time/date.

**Promptly report any issues to AAF Staff**

## **Appendix I: Back Scatter Detector (BEIW)**

Under typical SEM imaging, a secondary electron detector is used. Secondary electron detectors require high vacuum and provide surface topography contrast for imaging. Image formation is due to inelastic collision of high energy electrons which impact the sample surface (data from top 5-50nm). Other high energy electrons from the electron beam do not interact with the sample surface. These elastically scattered electrons (data from top 50-300nm) are scattered at large angles to the sample surface and are useful for elemental contrast (z-contrast). BSE detectors do not require high vacuum and therefore in addition to providing z-contrast, are used for imaging under low-vacuum mode (appendix II).

### **3-segment detector (segments A,B, and C)**

Topo = surface topography (A-B)

Compo = Z contrast (A+B)

Shadow = Enhances Topo mode

1. Only one detector can be used simultaneously, either secondary electron detector (SEI), or backscatter electron detector (BEIW).
2. To use the BEIW, make sure that the detector is properly positioned underneath the pole piece.
3. Follow same steps outlined in main procedure through step #8.
4. Then, click on "Signal" at the bottom of the JEOL SEM software window.
5. Select BEIW and double click.
6. When finished, set the detector type back to SEI and follow same steps as outlined in main procedure to turn off beam, remove sample, etc.

## **Appendix II: Low Vacuum Mode**

Low vacuum mode allows for imaging of non-conductive samples without the need for sputter coating. Low vacuum mode can also be used for samples with moderate vapor pressure (see AAF staff for more information). Only the back scatter electron detector (BEIW) can be used when in low vacuum mode.

1. Follow steps outline in main procedure through step 7 EXCEPT that electrical isolation sample holder must be used.

**\*\*Do not press EVAC until low vacuum mode AND BEIW is set up!\*\***

2. On JEOL SEM software click "Signal" and change to BEIW.
3. On JEOL SEM software click "Vac. Mode" at bottom of screen.
4. Will be prompted: "Are you sure you want to change to Low Vacuum mode?"
  - Click Yes.
5. Start low vacuum mode at about 30-40Pa. (conversion: 1Pa = 7.5mTorr)
6. Now, press "Evac" button on front of instrument.
7. Once Evac button stops blinking, the high voltage "HT" icon at the top left of the JEOL SEM software should say "ready".  
If it does, click the icon.
8. Now, main procedure guidelines for imaging and SEM shutdown can be followed.
9. As a final step, once you are done and sample is removed from chamber adjust the "Signal" back to SEI and "Vac. Mode" back to High Vacuum.

## Appendix III:

### JEOL JSM-6460LV Filament Replacement:

Filament replacement is necessary every few months of regular use or when system vacuum is poor. JEOL SEM software will indicate filament burnt out with an error message. Additionally, “LC current” (load current) display value will be low or near zero mA. Load current (mA) during regular use will be about five times the accelerating voltage (kV) value being used.

1. If microscope was being used for imaging for any length of time before burn-out the electron gun (filament and Wehnelt cap) will be very hot.
  - Allow the electron gun to cool for several minutes under *vented* conditions before attempting to replace the filament.
2. Using nitrile inspection gloves, open the electron gun on top of the column and remove the Wehnelt by pulling it straight out.
3. Disassemble the Wehnelt by loosening the external set screws that hold the ceramic base of the filament in place and pull the filament out by the electrode prongs.
4. Visually inspect the filament using a stereo microscope to verify the mode of failure (See JEOL manual page 5-7 for more information).
5. Clean the Wehnelt cap with metal polish followed by isopropyl alcohol rinse.
6. Install a new filament in the reverse procedure:
  - a. **Important:** Make certain that the spacer is seated all the way down in the Wehnelt cap and that scribe mark is facing the correct direction.
  - b. Insert the new filament with the guide groove facing the correct direction.
  - c. Visually check the filament position using a stereo microscope. The Filament tip should automatically be centered and positioned slightly below the cone tip hole in the Wehnelt cap.
7. Open the electron gun on the top of the column.
8. Align the guide groove on the Wehnelt with the guide pin on the electron gun and then push in the Wehnelt until it clicks into position.
9. Close the electron gun cap making sure that the o-ring is seated properly.
10. Press EVAC on the front of the system.
11. Once the EVAC button stops flashing and the “HT ready” icon appears on the main software window, click the icon to turn HT on (accelerating voltage).
12. Perform gun alignment (use semi-auto gun align, NOT auto).
  - a. Manually move scroll bars to fine tune the gun alignment.