

## SEM OPERATION IN LOW VACUUM MODE

### Instructions for JEOL 5800 LV

The EVAC light of the SEM specimen chamber should be already lit when you approach the SEM & the SEM will have been left in the high vacuum or SEI mode.

Sample display reading:

HT READY	SEI	20KV	X 300,000	WD35
X = + 0.00	Y = +0.00	R= +0.00	Z= +35.0	T = +0.0

WD & Z depend position of sample holder during removal of sample from chamber.

### To load sample:

1. Press VENT and hold for about 1-2 seconds to bring the specimen chamber to atmosphere. Vent button lights up, Evac light goes off.
2. Wait for Beep.
3. UNLOCK specimen chamber door.
4. OPEN specimen chamber door.
5. INSERT specimen holder containing sample into specimen chamber using the tongs. Keep fingers off of specimen holder & specimen – use gloves.
6. CHANGE the magnification from x300,000 (see display panel) to a lower mag by pressing the “instant magnification” INST MAG button. Magnification will instantly change from 300,000x to approximately 100x. You can further lower the magnification if you want using the magnification knob to ~60 - 80x. Lowest possible mag depends on your working distance (WD; see display panel).
7. Use the JOYSTICK to POSITION the sample holder in the specimen chamber.
  - Make sure sample is not too close to detector! Position highest point of sample approximately 10-15 mm from the detector.
  - To center sample, use joystick in X/Y mode (X/Y button is lit). Move it left/right. (If Magnification is too high, X/Y movement is null.
  - To raise/lower sample, use joystick in T/Z mode (T/Z button is lit, and small T/Z button flashes). Move it up/down (towards left monitor or towards you).
  - Note: Stage moves the fastest at the lowest possible magnification.
  - IMPORTANT! Press X/Y button when sample holder is in position. (X/Y button is now lit).
8. CLOSE & LOCK specimen chamber door. \* Write down the WD and position of your sample for future reference.

To start LV mode:

9. Press LV Button located on front left panel of SEM. Display will read 9.9.9 Pa.
10. Press AUTO button.
11. Press EVAC button to evacuate the specimen chamber.
  - LV panel will flash to the number previously set.
  - Suggested LV setting for a wet sample is 150 Pa; for a dry sample try 20 Pa.
  - To change LV setting: turn controller knob to the right (clockwise) to turn the pressure down (i.e., 50 Pa to 7 Pa), and to the left (counter clockwise) to increase pressure in specimen chamber (i.e., 10 Pa to 150 Pa).
  - It takes a while for the pressure to change - be patient! You can move on to the next step before specimen chamber reaches desired vacuum, but remember to listen for beep before saturating the filament.
  - LV panel stops flashing when it reaches the desired vacuum that you set.
12. LOOK at display panel under left monitor. It will change from VENT to PRE-EVAC to EVAC to HT READY.

Sample display panel reading:

HT READY	BE/C	20KV	X 200	WD35
X = + 0.00	Y = +0.00	R= +0.00	Z= +35.0	T = +0.0

WD & Z depend sample height in sample holder & focus setting.

13. TURN UP the brightness & contrast on the two monitors. Don't turn them up all the way.
14. If the display panel shows a different accelerating voltage than you want, use the computer mouse to choose MENU (if not already in this mode). Click on ACCV to choose the accelerating voltage you wish. When finished, a right mouse click or escape will get rid of this submenu. Remember: a high accelerating voltage (30kV) will penetrate samples deeper and damage them more quickly than a low one (10kV).
15. In the Menu mode click on BEIW (left side of screen). Make sure the submenu reads:

Polarity	POSI	When activated, box is yellow.
Image	COMP	
Compo Brt	CRS	
Shadow	ON	

Choose a middle shadow level (click mouse on middle position).

To saturate the filament:

16. First verify the filament knob is at the seven o'clock position (i.e., off). Press **SPOT SIZE** button (on extreme right of keyboard). The button will light up green when activated. **CRS, SIZE** and **FINE** will light up orange, and **SIZE** will flash on & off. This button may have been on **TONE**.
17. Check the number of chevrons (>>) visible in the display panel which may look as follows:

HT READY	BE/C	20KV	x 300,000	WD34
SPOT SIZE	>>>>>>>	>>>>>>>	> - - - - -	- - - - -

18. Press the **HT** button (extreme right of keyboard). This button will light up orange when activated. The filament light to its immediate left will light up and flash continuously.
19. Press **DMAG** button on top left keyboard. The button will light up green when activated.
20. Press **LSP1** button (to right of **DMAG** button). The button will light up green when activated, and a line appears on the left monitor.
21. Make sure that at least 12 chevrons are lit (ballpark setting: anywhere in the middle set of chevrons). Change spot size by rotating the knobs for **CRS** and **FINE**. Spot size is dependent on the chosen accelerating voltage you are working with. Go to **TONE**.
22. **SLOWLY** saturate the filament by slowly turning the filament knob clockwise from a 7 o'clock position to approximately 2 o'clock. Notice the filament light stops flashing. What you need to look for in the left monitor as you rotate the filament knob so that you know the filament is saturated:
  - Observe the horizontal line on the left monitor.
  - As you slowly saturate the filament, the line should become wavy (noisy).
  - When the panel display reads 12 o'clock, the line should be very wavy at this point. If not press **TONE** button (is lit green), and rotate knob controlling contrast (**CONT**) & knob controlling brightness (**BRT**) until the line on the left monitor is wavy. Don't let the line to go to the top of the screen & become flat - adjust using **BRT** & **CONT** knobs.
  - As you continue to slowly turn the filament knob, the wavy line should:
    - 1) increase in amplitude, then 2) decrease amplitude, then 3) increase again. At the point of the second increase the filament is saturated.
  - **IMPORTANT!** Do not turn the filament knob past 2 o'clock. Look at the panel display which should show **L.C.** and a number (in  $\mu\text{A}$ ) instead of **HT ready**. If  $\mu\text{A}$  doesn't increase with knob rotation you have saturated the filament!!! Back off.

A sample display panel in LV mode with filament saturated. Record start time on filament. Close canopy lid.

L.C	BE/C	83 $\mu\text{A}$	20KV	X 200	WD35
X = + 0.00	Y = +0.00	R= +0.00	Z= +35.0	T = +0.0	

To get an image:

\*Insure that nothing or no one is touching/against the column console. Image quality is greatly affected.

23. Press the **SL1** button. The line on the left monitor should disappear.
  - If the picture is too bright or dark; in **TONE** mode, rotate the **BRT** and/or **CONT** knobs, so that sample is visible. An easier way to adjust is listed below.
  - If you press **SL1** again, a graphical display (called a wave form monitor) will appear overlaying the image in the left monitor. Adjust **BRT** & **CONT** so that they are centered on the graph.
  - The sample is most likely out of focus at this time.
24. Press the **COARSE** button above the focus knob (right side of keyboard). This will let you quickly focus on the sample.
25. Increase magnification by 5-10 times or more (e.g., from 150x to 550x). Use coarse focus to get a sharp image.
26. When you get the best focus on the sample, press the **COARSE** button again (coarse button is no longer lit) – this lets the focus knob adjust with fine focus.
27. Press the **STIG** button and use **X** & **Y** knobs to get a sharply focused image; then return to the desired low mag (e.g., 150x). When you change **ACCV**, recheck filament saturation and stigmat.
28. Press the **SL2** button once. Observe the image - if it is acceptable, you can go to step #31 or #32. If not, press **SL2** button again to obtain wave form monitor function, then use fine focus to get sample in focus, and adjust **CON** & **BRT**.
29. Press **SL2** button (now in regular raster mode) and use fine focus if necessary.
30. Press **DMAG** button (not lit) to get a full screen view. (Optional).

To move the specimen:

31. If you are in the wrong area of the specimen, you may move the specimen by using the joy stick. **BEFORE USING JOYSTICK:**
  - Make sure the **X/Y** button on the extreme left of keyboard is lit.
  - Make sure the display panel reads **T = +0.00** (see ¶ #12). If display panel says **Spot Size**, press **Tone** button to see sample position on display panel.
  - Moving joy stick to left makes image move left, and you can see what was out of view to the right.
  - Moving joy stick up (towards left monitor) makes image shift up, and you can see what was below the current view.

To take a video print picture:

32. In SL2 mode, obtain a good image that is in focus with appropriate contrast & brightness.
33. Press SL2 button twice to see a graphical display overlaying the image in the left monitor. In TONE mode adjust BRT and CONT so that they are centered on the graph.
34. Press SL3 and wait for one full screen (raster).
35. Press the Freeze L button to freeze the good image on left monitor. (Can also freeze right image and print while continuing to work with left screen).
36. Select L button under “select” on panel near Polaroid camera. (or R if froze R image).
37. Press PRINT under “stored image”.
38. Remove video print from bottom right front of SEM. Picture tears off.

To take a Polaroid picture (photo):

39. Repeat steps #32 - #36 above.
40. Use Polaroid film Type 52 at F16, Pro 400 at F16, or Type 55 at F8.
41. Verify that Polaroid camera lever is in L position.
42. Insert film in camera. Leave lever in L position.
43. Pull film out.
44. Press “Stored” PHOTO. Photo light is on.
45. Wait for exposure by watching orange photo light go out & you’ll also hear a beep.
46. Push film back in.
47. Move lever to P position.
48. Pull film out.
49. Wait a specified number of seconds as indicated on film envelope.
50. Leave lever in P position.

To take save image to computer disk:

51. Repeat steps #32 - #34.
52. Insert disk into disk drive (Disk label faces left).
53. Click on ALBUM in computer screen.
54. Click on SAVE.
55. Answer questions in submenu (filename, screen choice, TIFF/Bitmap).
56. Wait for image to load on disk. In SL3 mode, only one image fits on disk. In other modes up to 4 images can fit on disk. Icon appears on computer screen.

To change the sample:

57. Slowly turn down the filament knob. Press the orange HT button to turn off the high tension. The HT button should no longer be lit. Keep track of filament time.
58. Press VENT on the specimen chamber.
59. Remove old sample and add new one: SEE steps #1 - 8 above. Be careful of specimen positioning.
60. Press EVAC button.
61. When display panel says HT Ready you can press the HT button to turn on the high tension.
62. Resume steps #23+ above to get an image of the new sample.

To shut down:

63. Slowly turn FILAMENT KNOB counter clockwise back to 7 o'clock. Notice orange filament light next to HT button flashes. Keep track of filament time.
64. Press HT button if still on. It's orange light should be off, as well as that of the filament.
65. Turn MAGNIFICATION KNOB all the way to 300,000x. See display panel to verify.
66. Press DMAG button to turn it off, if you haven't done so already. It should not be lit.
67. Press Freeze L or R buttons so that they are not lit.
68. Press TONE button if it isn't already activated (should be lit green).
69. Press SL3 button. Should light up green.
70. Leave computer in MENU mode.
71. Turn down brightness and contrast on two monitors.
72. Press VENT.
73. Wait for beep.
74. Unlock and open specimen chamber door.
75. Lower specimen holder using joystick in T/Z mode. Look while doing this.
76. Press X/Y button so that it is lit green.
77. Close and lock specimen chamber door.
78. Press LV button (lit up) on front left panel to turn off LV (button no longer lit).
79. Press EVAC. Notice display mode shows SEI instead of BE/C.
80. IMPORTANT! CLEAN UP the area.
81. Scope is now in SEI mode. Leave scope in this mode.

A sample display panel when you are finished.

HT READY	SEI	20KV	X 300,000	WD34
X = +19.10	Y = +10.09	R= +0.00	Z= +35.0	T = +0.0

Note: WD & Z depend on where you left sample holder when you took the sample out. Only "controls" lit (green) are SL3, X/Y, and TONE.