

JSM-7500F TYPICAL OPERATION PROCEDURES

- WEAR GLOVES: always wear gloves when handling your samples, the sample holders, and the specimen exchange rod.
- Ensure that samples are dry and free of excessive out gassing – always discuss a new sample with the I. Center staff before you use the SEM to appropriately prepare your sample.
- MAGNETIC SAMPLES: please discuss with Dr. Gadiant first – they require different procedure than conventional non-magnetic samples.
- You may save your images on the SEM computer but it is highly recommended that you either transfer them to a flash drive or email them to yourself once you are finished on the instrument.

1. Getting started/loading the sample

(a) Sign the logbook: Please write the DATE, the TIME you start using the instrument, your FIRST NAME, your ADVISOR'S LAST NAME, and the ANALYSIS TYPE (SEM/EDS)

(b) SLOWLY fill the Dewar on the instrument with liquid nitrogen until it just overflows.

(c) Turn on the specimen IR chamber camera; IR Camera is minimized on the task bar. If the icon is not on the task bar, open the IR camera from the Desktop shortcut.



NOTE: Ordinarily, the airlock chamber is under high vacuum (9.6×10^{-5} Pa). Before loading, the lights in the airlock button should be: VENT – OFF; EVAC – ON; HLDR – OFF

(d) Check that the stage is in the exchange position - EXCH POSN light on the side of the airlock should be ON and the stage parameters x ,y, z, R & T should read: x=0; y=0; z=38; R=0; T=0

[If not: Click **Spec. Exchange** button next to the SEM diagram on the bottom right of the screen.]

(e) Put on your gloves and mount your sample on the sample holder.

(f) Press and hold the **VENT** button until it starts to blink; the isolation valve closes and N₂ gas vents into the airlock. (The VENT button can also be clicked from the SEM monitor window)

(g) When the **VENT** light stops blinking, release the latch on the airlock to open the airlock door.

(h) Load your sample, close the airlock and press the **EVAC** button until it starts to blink.

(i) Wait until the **EVAC** light stops blinking.

Now the lights in the airlock buttons should be VENT – OFF; EVAC – ON; EXCH POSN – ON; HLDR – OFF

(j) Fully insert the specimen exchange rod, keeping the holder horizontal until it comes to a firm stop and make sure the **HLDR** light is ON.

(k) Retract the exchange rod fully and tilt up. Remove your gloves now.

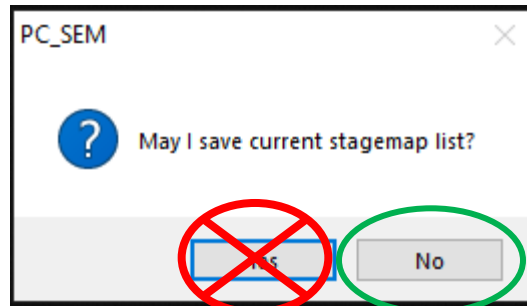
Now the lights on the airlock should be VENT – OFF; EVAC – ON; EXCH POSN – ON; HLDR – ON

(l) When the “Select Specimen holder” window pops up, select the holder type.

(m) Slowly fill the Dewar a second time until it just overflows. Place the cap on the Dewar.

(n) Click **Load** under the Stage Map tab.

****When this dialogue box pops up, click No****



(o) Select the appropriate option from the pop-up. If you are unsure, please ask Cassandra or Dr. Gadiant.

(p) While waiting for the vacuum to pump down, double click on Z and enter the working distance for your sample and click **Move** (working distance for each stage map is located on the table uploaded from the stage map—see following image). **Watch the sample move on the IR camera, if it looks like it is about to touch the beam source, tap the trackball to immediately stop the stage.**

PC-SEM

File (F) Edit (E) Function (O) Image Processing (I) Tools (T) Setup (S) Maintenance (M) Help (H)

Observation Accel. Voltage Emission current

OFF ON 2.00kV 0.04kA

Probe Current Contrast Brightness Focus Magnification Stigma X Stigma Y D-Mag RDC

Quick1 Fine1 CF Freeze Freeze Auto ACB Normal Shift ZFC

Text SRT Cursor Spot

Full Image Specimen Observation Comparison

Guide	Navigator	Step Control	Stage Map			
No.	memo	X	Y	R	Z	T
2	2	12.7	1.467	0.0	10.0	0.0
3	3	12.8	8.170	0.0	10.0	0.0
4	4	-0.570	-15.000	0.0	10.0	0.0
5	5	-0.170	-7.191	0.0	10.0	0.0
6	6	1.100	3.985	0.0	10.0	0.0
7	7	0.192	13.200	0.0	10.0	0.0

Save Load New

Edit Mode PQ.Align. PQ.clear

Image File Condition Alignment

Top View Side View

100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100% 100%

Link the stage map display to magnification.

X: 0.000 Y: 0.000 R: 0.0 Z: 10.0 T: 0.0

10 μm JEOL 1/5/2022 X 400 2.00kV LEI SEM WD 9.6mm 13.46.56

Recipe

Standard User

- BEI observation of metals
- BEI observation of MR heads
- Biological
- Conductors(Nonconductive)
- Fine powders(about 1 μm)
- Heavy metals
- Light metals
- Lower detector(High Mag)
- Lower detector(Low Mag)
- Semiconductors(Nonconductive)
- Super fine powders(less than 0.1 μm)
- Cryo observation
- Plastics(with coating)

Set Conditions

Accel. Voltage (kV) 2.00

Probe Current LC HC Z

Image Rotation 0.0

Dynamic Focus

Focus Depth 1 Std. 11

Column Mode SEM LM GB-H GB-L

r-Filter SB 0

SE SB BE

LUT Standard LUT

SEM Monitor

Vacuum 9.9E-5 Pa

Spec. Surface Offset 0.0 mm

Spec. Exchange

Home Position

VENT EVAC

Gun

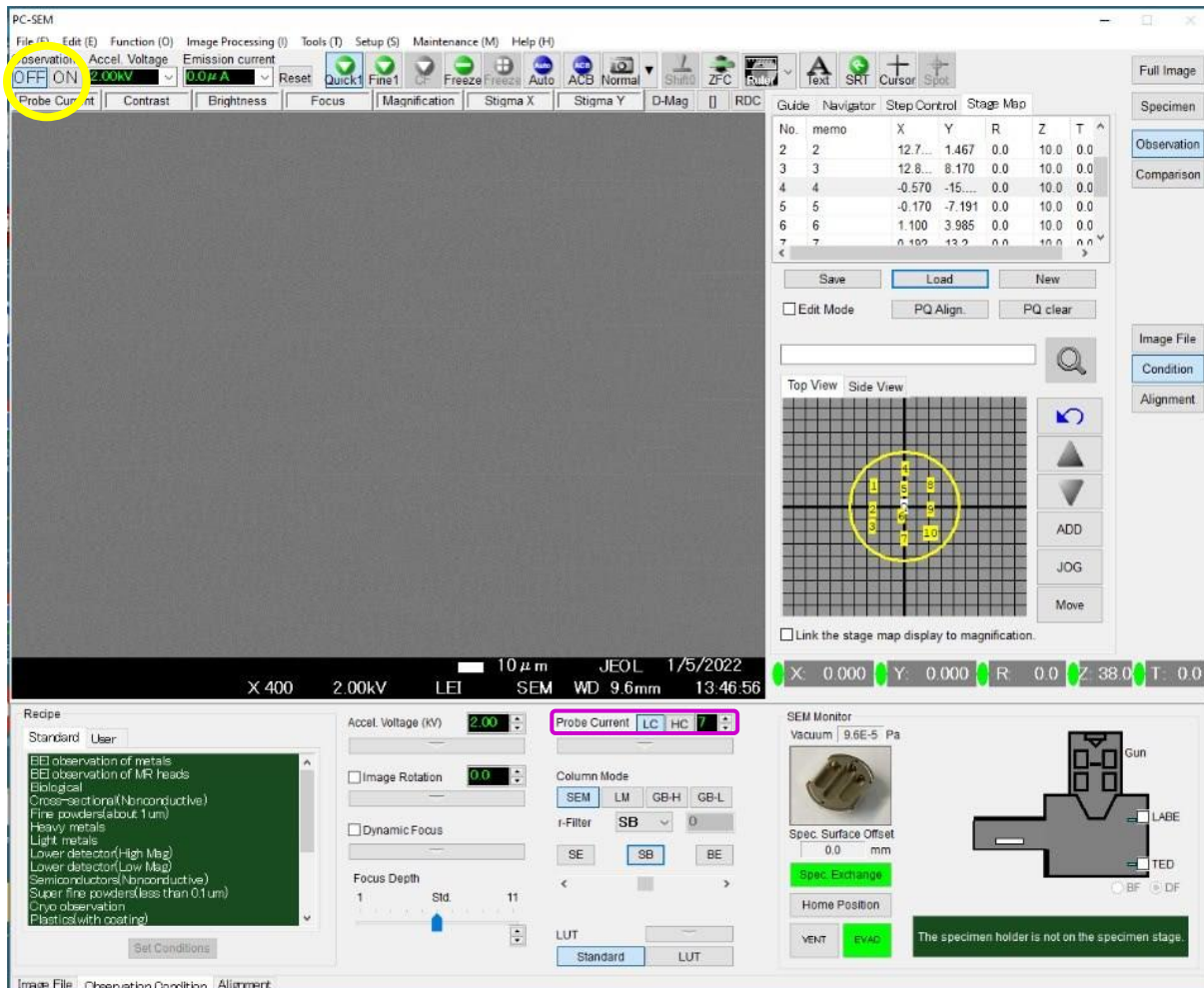
LASE

TED

BF DF

The specimen holder is not on the specimen stage.

Image File Observation Condition Alignment



2. Finding the desired area on the sample

- Wait until the Vacuum pressure reads 9.6×10^{-6} Pa.
- Select/Input the value for Acceleration voltage (acc. vol.). Start with 2kV for imaging or 15kV for EDS – if you need to increase or decrease the acc. vol at some point during your work, make sure you do it in steps of 2kV to avoid damaging the gun.
- Turn on the **Acceleration voltage**
- Watch the Emission Current readout increase. Wait for proper Emission Current 20 μ A.
- Check the **Probe Current** (under the image window). If you are using 2kV, the LC button should be selected. If you are using 15kV or higher for EDS, the HC button should be selected.
- Use Low Magnification Mode (LM) to find area of interest on sample and have it centered. (*operational panel: **LOW MAG** button*)

- (g) Auto adjust contrast and Brightness
(operational panel: **ACB** button)
- (h) Focus just enough to identify site of interest on your sample
(operational panel: **FOCUS** knob)
- (i) Turn off LM and select SEM Mode (LOW MAG button light off).

3. Correcting Astigmatism using the Operation Panel

- (a) Stop focus at center of over and under focus (image may not be sharp but has no directionality of focus).
- (b) Adjust the X & Y knobs on the operation panel to obtain the sharpest image.
- (c) Increase magnification and focus the image with the **FOCUS** knob checking for astigmatism
- (d) Usually several iterations of steps (a) – (c) are required before acquiring a crisp image.

Note: Focusing and astigmatism are generally necessary for any change in operating parameters except for magnification. In fact, for a good image acquisition it is best to adjust these controls at an exaggerated magnification (at least 50kX more than the desired magnification), then reduce the magnification to the desired level.

4. Taking photos of images

Acquisition of images can be performed using the FREEZE or NORMAL mode. Once acquisition is completed, save it in your folder. Create a folder with your name at
C: users\yourgroupname

- (a) Using the NORMAL mode (recommended for non-charging specimens)
 - (i) Set exposure visually
KNOBSET: **ACB** button and/or **CONTRAST, BRIGHTNESS** knobs
 - (ii) Press **PHOTO** on the KNOBSET or **NORMAL** on the software button for slow acquisition
Important: make sure the Export box is checked in the “save as” window to ensure all the image information is saved as part of the image.
- (b) Using the FREEZE mode (recommended for charging specimens)
 - (i) Check if the “Freeze button set to integration” box is checked (setup\ operation settings\Image scan\Integration)
 - (ii) Press the FREEZE button (it will blink until it finishes acquisition)
 - (iii) Once it stops blinking, go to FILE >SAVE AS and save your file in the appropriate folder or click **NORMAL** on the software

Important: You can use the **LUT** (Look Up Table) to adjust the contrast and brightness of the frozen image before saving it.

5. Shutting Down

- (a) Turn the Accelerating Voltage off [Observation]:[OFF]
- (b) Make sure the IR camera is turned on.
- (c) Retract the LABE detector if used uncheck the LABE check box
(Note: Make sure that the top of your sample is at least 8mm away before you retract the LABE.)
- (d) Move the stage to the exchange position: Click the **Spec. Exchange** button in the SEM monitor window. Make sure the stage parameters read as follows: x=0; y=0; z=38; R=0; T=0

**Note: Before unloading, the lights in the airlock button should be:
VENT – OFF; EVAC – ON; EXCH POSN – ON; HLDR – ON**

- (e) Wear gloves! Fully insert the specimen exchange rod keeping it horizontal until you feel it comes to a firm stop.
- (f) Fully retract the specimen exchange rod and confirm that the HOLDR light goes out.
- (g) When the rod is fully retracted, tilt up the exchange rod and secure it to the black clip.
- (h) Press and hold the **VENT** button momentarily until it starts to blink (the isolation valve closes; N₂ gas vents into the airlock).
- (i) When the VENT button light stops blinking, unlock the airlock chamber by releasing the latch. Open the airlock door.
- (j) Remove the specimen.
- (k) Close and lock the airlock chamber and press the **EVAC** button until the light starts blinking.

**When the EVAC light stops blinking, the airlock button should be:
VENT – OFF; EVAC – ON; EXCH POSN – ON; HLDR – OFF**

- (l) Make sure the pump is pumping down well and is in the 10⁻³ Pa range.
- (m) Record the time you finish in the login book.

ADDITIONAL OPERATIONAL STEPS AND TIPS
(Jeol Applications Laboratory, p.93)

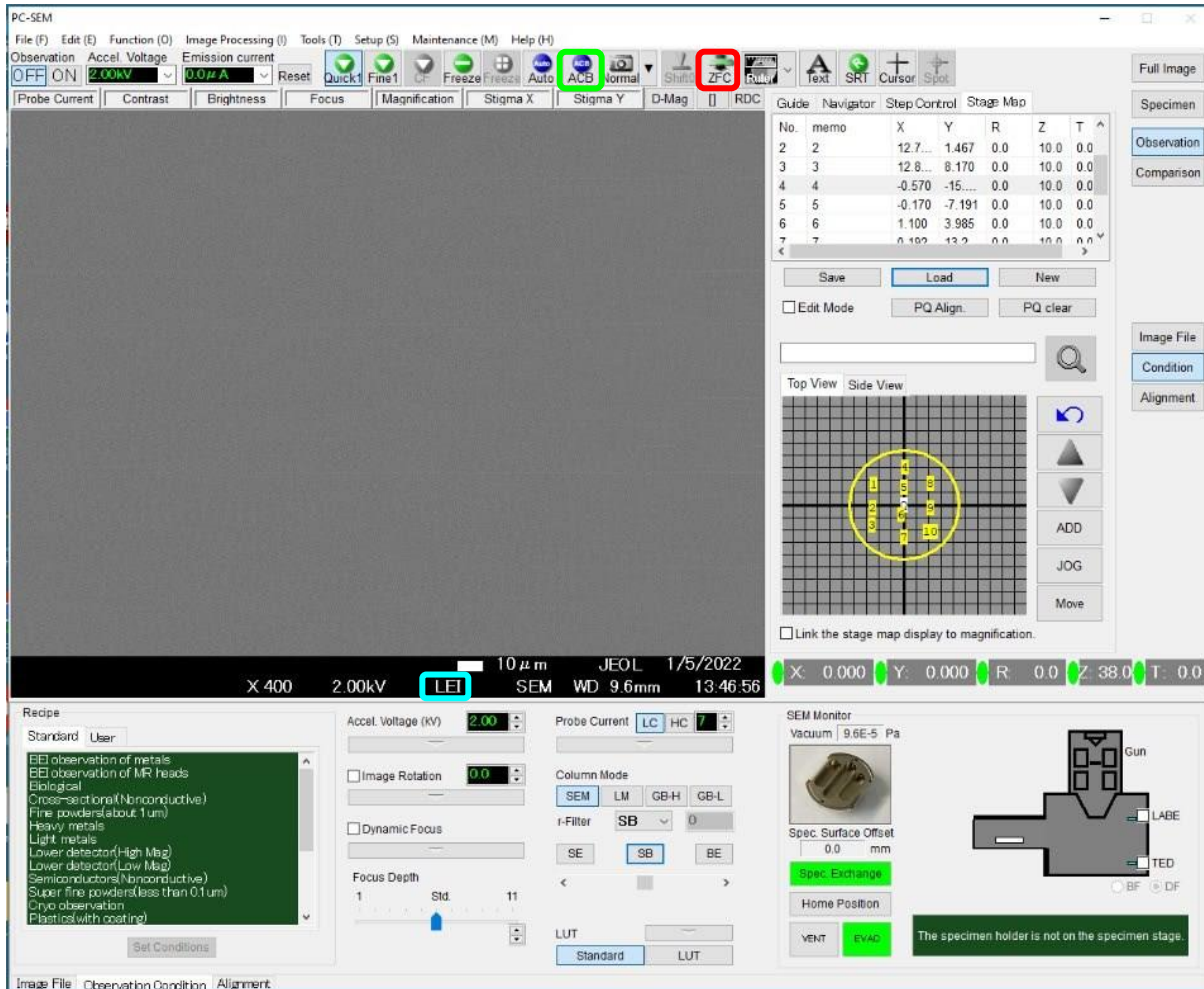
1. CHOOSING BETWEEN THE TWO SECONDARY ELECTRON DETECTORS (LEI) Vs (SEI)

- for best/high resolution: SEI especially at Low KV 4.5 – 6mm WD
- for good resolution: SEI, LEI 8mm WD

2. SETTING THE APPROPRIATE WORKING DISTANCE (WD)

Be mindful of the actual distance between the top of the sample and the lens! Make sure the IR camera is on whenever you are making any Z movement or inserting and retracting the LAGE detector.

- (a) Select the desired WD left click [WD] and select (WD= 15mm is recommended for a start)
(**image info area** is the black bottom of the image viewing part of the screen)
- (b) Make sure you are at the lowest magnification possible for the selected WD in SEM mode.
- (c) Move stage from the exchange position close to the selected WD as follows
STAGE INFO AREA: Left click Z, input a value close to the desired value (WD + offset+ 3mm)
- (d) Adjust contrast and brightness using auto contrast/brightness, **ACB**.
- (e) If the contrast of the image is too high (if the image looks too bright or too dark), change your detector to **LEI**. If the image looks too dark (not enough electron yield), you will need to increase the probe current.
- (f) Bring image into focus with Stage Z control (manual control of stage) as follows: Activate **[ZFC]** use **TRACKBALL RING** until the sample surface comes into focus.
- (g) Turn off Stage focusing [ZFC]



3. ALIGNING THE BEAM using the “Operation Panel”

- (a) Set magnification to ~5000X – 50000X.....MAGNIFICATION knob
- (b) Focus the image the best you can.....FOCUS knob (c) Turn Wobbler on.....WOBB button.
- (d) If image shifts, adjust to stop image shifting.....X, Y knob
- (e) Turn Wobbler off.....WOBB button
- (f) Increase the magnification and then focus to check for any astigmatism
Using the **FOCUS** knob, check for astigmatism by going through, over & under focus while looking for directionality (or streaking) of focus in the image (over and under focus directionality will be at right angle to each other).
- (g) Correct Astigmatism if necessary.....see step 5 below

4. REDUCING THE EFFECT OF SAMPLE CHARGING IN THE IMAGE

- Reduce the accelerating voltage: *Make sure you change the acceleration voltage in steps of 2kV and wait for few seconds for it to stabilize between each step.* (Otherwise, it will shorten the lifetime of the gun or even damage the gun)
- Use LBE detector (since BEs have more energy than SEs your image is less susceptible to Charging)
- One can also use a BE imaging at low accelerating voltage (at or < 2kV) using GB mode
- Reduce the probe current [Observation]:[Condition]:Probe current slider
- Use integration to acquire image KNOBSET: FREEZE button
- Use the Lower Detector (LEI) For WD longer than 6mm
- Change to GB mode (see step 3 below)
- Reduce the emission current Emission Current: [select desired current 20, 10, 5, 2]

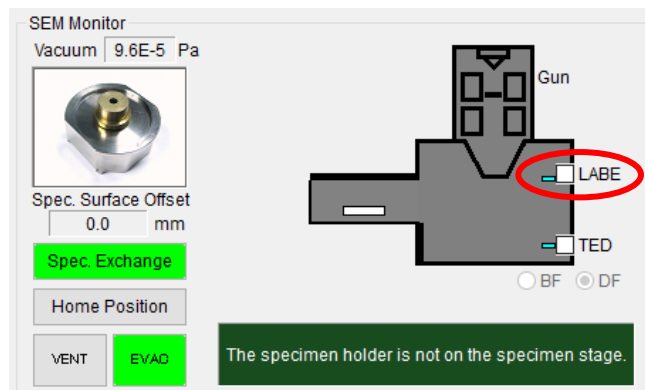
5. WHAT TO DO IF SIGNAL IS WEAK

If your image looks darker or not clear even after adjusting the contrast and brightness, you may have weak signal (not enough electron yield) and so try the following:

- Check if the emission current has reduced from what you have started, if so reset by clicking the reset button
- increase the probe current
- increase emission current to 20 μ A if using different
- if working at WD > 6mm, try using LEI instead of SEI

BACKSCATTERED ELECTRON (LBE) IMAGING

1. Make sure you have the camera on.
2. Make sure there is at least 8mm gap between the objective lens and your sample surface. If you have been working at shorter WD, then lower the stage to have 8mm gap using ZFC before you insert the LBE detector. (Once you insert the detector, use ZFC to bring up the stage to your desired WD.)
3. Insert LBE Detector in: in the SEM Monitor Window select the LBE detector check box, wait till the Gun valve opens.



4. First using the SEI mode,
 - (a) choose the sample location
 - (b) adjust magnification, focus and astigmatism
 - (c) adjust contrast and brightness (ACB)
 - (d) make sure the **IR Camera is off** (other wise it will “blind” the LABE detector and you won’t be able to see any signal, you will see a white screen)
5. Now set detector to LABE: IMAGE INFO AREA: [SEI] • [LABE]
6. Set scan speed to slow: KNOBSET: FINE VIEW button
7. Adjust the Brightness and Contrast
8. If the contrast is too weak, it may be necessary to increase the Probe Current to a large number, at least PC 10, or you may need to increase the Acc. voltage.
9. View and/or acquire BE image using the FREEZE or PHOTO mode.
10. When finished retract the LABE detector: in the SEM Monitor Window uncheck the LABE detector box.
(Make sure you have at least 8mm gap between the objective lens and your sample surface.)
11. Set detector selector back to SEI: IMAGE INFO AREA: [LABE] → [SEI]