

SEM Instructions (JEOL JSM-6500 F)

User: Emcenter-sem

Password: ***** (See system administrator)

Domain: CVMBSDOM

A. Sample Preparation (3 holders)

1. Holds 1 stub, most tilt
2. Bulk holder – deepest, use for pin stubs
3. 2BH: multiple samples (eight 10mm stubs or six 12mm stubs)
 - Wear powder-free gloves
 - Use a minimal amount of carbon sticky tape to attach samples
 - If your sample is mounted on a non-conducting substrate, make sure you ground the surface of your sample to the holder. You can use graphite or silver paste for this (best) or single-sided copper tape. (Copper tape doesn't need to outgas prior to inserting into the SEM.)
 - Height adjustments are on the holder bottoms; the top of the sample should be as tall as the holder. Use height gauge to ensure proper height of sample.

B. Loading a sample

1. Open the instrument control program, click OK to bring up the specimen exchange window (6th button from the right at the top of the screen, counting the “?” [Help] button as first.)
2. Select the correct holder.
3. Click on the “Exchange” or “Home” buttons if the EXCH POSN light under the specimen entry lock is not on. This will modify the x and y positions, as well as the rotation, to their exchange positions.
4. Adjust tilt and z manually.
 - Specimen exchange position: tilt to 0 (manual adjustment), z to 10mm (manual adjustment), x and y are motorized and will adjust automatically.
5. Turn on the vacuum gauge (“PVG” button, 2nd button from right).
6. Make sure the specimen exchange position button is lit and the gun valve is closed (“GUN VALVE” button lit).
7. Unclip door
8. Press vent chamber button until it flashes.
9. Load sample holders so that ↔ axis is parallel to the axis of insertion (i.e points into chamber).
10. Check o-ring seal around door and clip door shut.
11. Press evacuation button until it flashes. It will flash in pre-evac, and will light continuously when sample is ready for insertion (i.e. the gate valve to the main chamber is open).
12. Wait until the main chamber get to < 1e-3 Pa (i.e. 9.63e-04Pa) before inserting the sample.
13. Turn on the chamber-scope screen and light.

- When you turn on the IR chamber-scope light, you will hear a couple beeps. This is normal, and is the EDS detector being saturated with IR light.
14. To load the sample:
 - Drop the black handle to the horizontal with a slow, smooth, continuous motion. (You may hear some hissing sounds during this process. These are normal, and are from motion of the air table that supports the beam column.)
 - Slide the handle in.
 - “HLDR” button will light when the sample is in. (It might take a second or two for this light to come on.)
 - Make sure pin on the bottom of the rod is not twisted!
 - Pull arm out all the way until it reaches the end of its travel. You should see the joint at the end of the rod.
 - Lift arm up into original position.
 15. Turn on the instrument control panel (the 7th button from the right).
 16. When the chamber is $<2e-4$ Pa (i.e. $1.91e-04$), open the gun valve by pressing the “GUN VALVE CLOSE” button for a second. It will take a few seconds for the light to go out, and you should hear the valve opening.
 17. Turn on the SEI detector in the instrument control window. You are now imaging, and being charged for beam time (\$30/hour).

C. Settings

Set the instrument control panel as follows:

- HT (high tension): Use a low kV for delicate sample (i.e. 1kV for PDMS), change by stepping in increments of 5 (bottom of instrument control panel screen).
- Col mode: SEM
- OL aperture: 3 unless you require very high resolution, then use OL aperture 4.
- Mode: 3 (0 or 1 for low kV work)
- Focus: controls z working distance, start at 10.
- Probe current: affects spot size, start at medium (6 – 13). [Other choices are small (1 – 6) and large (13-18).]
- Scan rotation: initially off.
- DFC (dynamic focus control): off

D. Alignment

1. Find a spot off of your sample, on the conducting stub or holder to do your alignment
2. Focus, using the working distance (manual coarse Z) knob on the beam column, not the focus knobs on the console.
3. Press ALIGN button on the console.
4. The align window will appear on the screen.
 - a. Clear all settings (gun and aperature)
 - b. OL aperture:
 - Set magnification to 3,000x to 10,000x
 - Probe current at 7
 - Turn HT wobbler on (separate box) and adjust amplification

- Pick a feature
 - Minimize x and y motion in the center of the screen by turning the x and y knobs on the actual aperture (i.e. on the beam column, not on the console).
 - The image should appear to be moving in and out of focus, but not up and down or side to side.
 - Turn HT wobbler off
- c. Gun alignment
- Set magnification to the lowest setting and probe current to the highest setting (i.e. 18).
 - Click “Gun alignment” button in “Align” window.
 - Turn x knob in the negative direction until your image disappears – not this number. Now turn the x knob in the positive direction until the image again disappears – note this number. Take the difference between the two numbers and divide by two, then subtract the results from your positive setting. This should now be the x setting of highest intensity. Repeat this procedure for the y setting. Your gun should now be aligned. Typical x and y values at 15 kV are less than 20.
 - If you cannot see a change in brightness, set to the middle of the brightest range.
- d. Return probe current to 7.
- e. Repeat step b.
- f. Stig center X
- Increase magnification to $> 20,000\times$
 - Turn wobbler on, on console. (Not the HT wobbler.)
 - Adjust x and y knobs on console so the image is not moving.
- g. Stig center Y
- Same as for “Stig center X”.
- h. Now you can close the alignment window and find a spot you want to image on your sample. Here you will do a fine focus and then adjust the OL stigmator by turning the x and y knobs on the console. This is an iterative process, so you will want to do this step several times until the image comes into sharp focus. At this point you are ready to take images. Repeat this step for each new area you image.
5. Once the sample is aligned in the reduced image, choose fine view in full screen mode and hit FREEZE button on the control panel to take a picture, or just press the PHOTO button on the console.
- E. Saving images
1. Save as .tif, bitmaps or JPEGs on G drive. (If you plan to publish an image, do not use JPEG to save the image.)
 2. Export images, don’t save them. (Exporting retains the scale bar; saving them does not.)
 3. Move files to server (L drive) when finished, or to your USB drive.
- F. Shutdown
1. Gun valve closed

2. RBEI out (retract backscatter detector, if relevant)
3. Leave system at 15kV and high magnification (less strain on scan lens)
4. SEI detector off
5. Specimen exchange window.
 - Exchange 1, z = 10, tilt = 0, et cetera
 - Exchange position light comes on
 - Retrieve sample.
6. Unlatch chamber
7. Push “VENT” (closes gate valve).
8. Remove sample from instrument.
9. Close door.
10. Press “EVAC”.
11. Wait for vacuum to reach $<5e-04$ Pa.
12. Exit program. (Don't save stage points.)
13. Log off.
14. Turn chamber scope light and screen off.