

JSM-7500F TYPICAL OPERATION PROCEDURES

Reminders!

- Remember to “sign in” on the sign up sheet.
- **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 MCF staff before you use the SEM to appropriately prepare your sample.
- **MAGNETIC SAMPLES:** please discuss with Dr. Bisrat first – they require different procedure than conventional non-magnetic samples.
- **Login with your user name and password** (Do not share your password with any body! The SEM computer keeps a record of who logged in.)
- Make sure you transfer your files into your own jump drive the same day. MCF will not back up your files and the USERS folder will be cleaned frequently without notice.
- **Please acknowledge the use of the MCF when you publish your paper and e-mail me a copy for our records.**

1. LOADING SAMPLE INTO CHAMBER

- (a) Sign up the login sheet: **Please write your FULL NAME, your ADVISOR’S FULL NAME and a complete ACCOUNT NUMBER legibly.**
- (b) Login to windows with your NetId (NetId@tamu.edu) and NetId pass word. Then select your account and submit.
- (c) Start the SEM software by clicking on “PC_SEM” icon on the desktop and login.
- (d) Turn on the specimen IR chamber camera, IR Camera icon on located on the desk top.
If it does not **turn on**, the procedure for setting up the camera can be found at the end of the SEM procedure manual on the SEM desk.
- (e) 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
- (f) 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:
LEFT click the grey box of any of the stage parameters (x, y, z, R, T) and type in the values manually and select “move”.
- (g) Put on your **GLOVES** and mount your sample on the sample holder.
- (h) 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)
- (i) When the VENT light stops blinking, release the latch on the airlock to open the airlock door.
- (j) Load your sample, close the airlock and press the **EVAC** button until it starts to blink.

- (k) 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
- (l) 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**.
- (m) Retract the exchange rod **FULLY** and tilt up; now the lights on the airlock should be
 VENT – OFF; EVAC – ON; EXCH POSN – ON; HLDR – ON
- Remove your GLOVES now.**
- (n) When the “Select Specimen holder” window pops up select the holder type and insert the
 specimen offset.

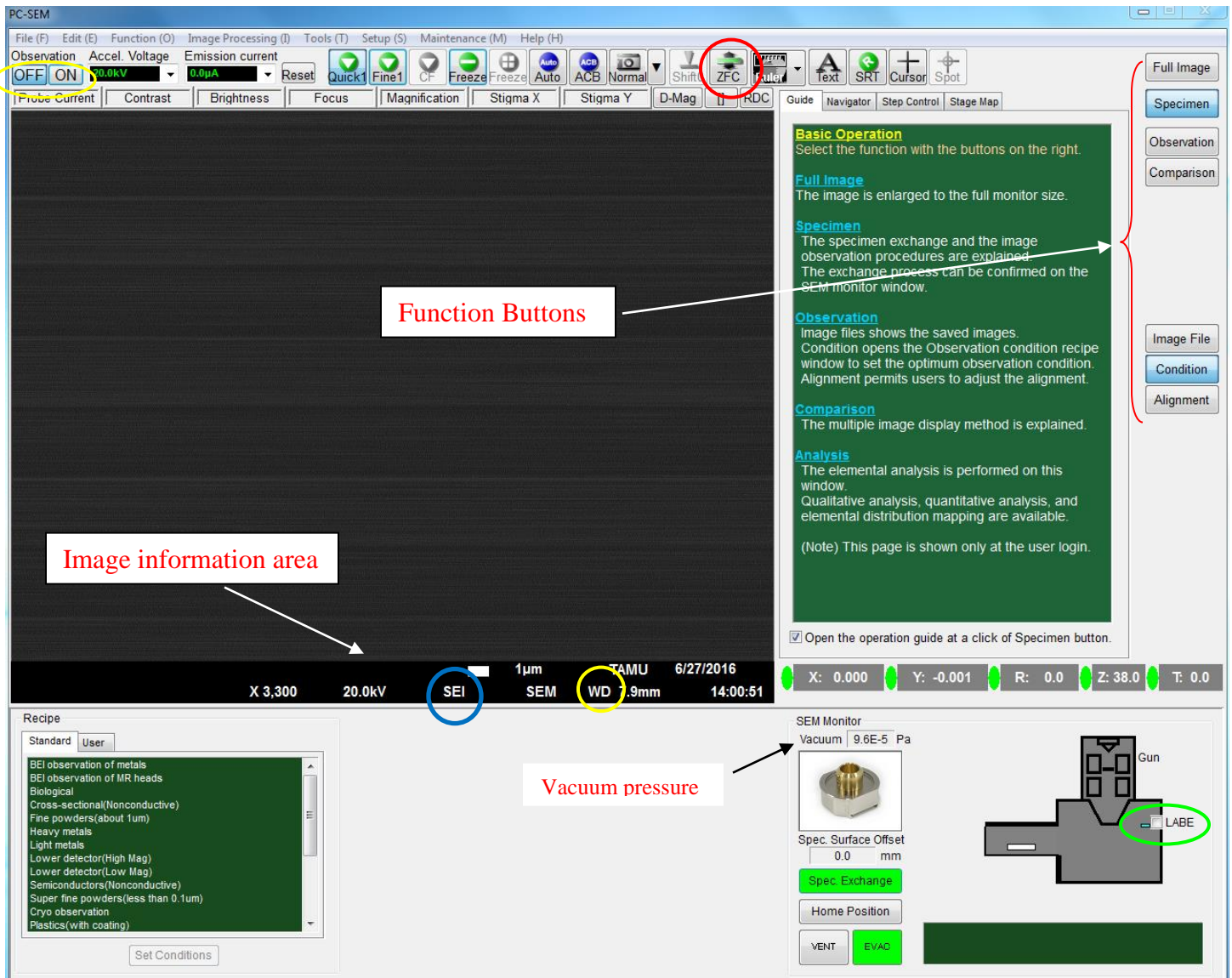


Figure 1. PC- SEM window

2. FINDING THE DESIRED AREA ON THE SAMPLE

- (a) Wait until the Vacuum pressure reads $3.6 \times 10^{-4} \text{Pa}$ or better.
- (b) Select/Input the value for Acceleration voltage (acc. vol.). Start with 5kV – 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.
- (c) Turn on the Acceleration voltage (*circled in yellow* on **Fig.1**): [ON]
- (d) Watch the Emission Current readout increase. Wait for proper Emission Current usually 10 μ A or 20 μ A.
- (e) Check the Probe Current setting, use the following guideline
FUNCTION BUTTONS (**Fig.1**): [Observation] Probe Current (generally 8 is a good choice).
- (f) Use Low Magnification Mode (LM) to find area of interest on sample and have it centered.
(KNOBSET on the “operational panel”: LOW MAG knob)
- (g) Auto adjust contrast and Brightness
(KNOBSET on the “operational panel”: ACB button)
- (h) Focus just enough to identify site of interest on your sample
(KNOBSET on the “operational panel”: FOCUS knob)
- (i) Turn off LM and select SEM Mode (LOW MAG button light off).

3. 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 LBE detector.

- (a) Select the desired WD
IMAGE INFO AREA: 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 as shown in **Fig.1**)
- (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** (*circled in blue in Fig.1*). 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**] (*circled in red on Fig.1*) use **TRACKBALL RING** until the sample surface comes into focus.
- (g) Turn off Stage focusing [ZFC]

4. ALIGNING THE BEAM using the “Operation Panel”

- (a) Set magnification to ~5000X – 50000X → KNOBSET: MAGNIFICATION knob
- (b) Focus the image the best you can → KNOBSET: FOCUS knob
- (c) Turn **Wobbler on** → “*Operation Panel*”: **WOBB** button.
- (d) If image shifts, adjust to stop image shifting → KNOBSET: X, Y knob
- (e) Turn **Wobbler off** → “*Operation Panel*”: **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

5. CORRECTING THE ASTIGMATION 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.

6. 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 full name at
C: images\users\
C: images\users\

- (a) *Using the NORMAL mode (recommended for non-charging specimens)*
 - (i) Set exposure visually
KNOBSET: ACB button and/or IMAGE 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

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

7. SHUTTING DOWN

(a) **Exit INCA software first if you have run EDS analysis.**

(b) Turn the Accelerating Voltage off [Observation]:[OFF]

(c) Make sure the IR camera is turned on.

(d) 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.)

(e) Move the stage to the exchange position

a. Click the Function button: Specimen button in the main menu

b. Click the “**Specimen Exchange**” button in the SEM monitor window

Make sure the stage parameters read as follows: $x=0$; $y=0$; $z=38$; $R=0$; $T=0$

(f) Before unloading, the lights in the airlock button should be:

VENT – OFF; EVAC – ON; EXCH POSN – ON; HLDR – ON

(g) **Wear gloves!** Fully insert the specimen exchange rod keeping it horizontal until you feel it comes to a firm stop.

(h) Fully retract the specimen exchange rod and confirm that the HOLDR light goes out.

(i) When the rod is FULLY retracted, tilt up the exchange rod and secure it to the black clip.

(j) Press and hold the VENT button momentarily until it starts to blink (the isolation valve closes; N₂ gas vents into the airlock).

(k) When the VENT button light stops blinking, unlock the airlock chamber by releasing the latch.

Open the airlock door.

(l) Remove the specimen.

(m) 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

(n) Make sure the pump is pumping down well and is in the 10⁻³ Pa range.

(o) Exit the SEM monitor: go to FILE ⇒ Exit and turn off the IR Camera.

(p) Exit the PC_SEM software.

(q) **Log off** windows and remove your user name.

(r) Complete the login sheet.

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 (**circled in green** on **Fig. 1.**), 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 LBE detector and you won’t be able to see any signal, you will see a white screen)
5. Now set detector to LBE: IMAGE INFO AREA: [SEI] ⇒ [LBE]
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 LBE detector: in the SEM Monitor Window uncheck the LBE 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: [LBE] ⇒ [SEI]

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. 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]

3. 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

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Ideally, these different facets will augment each other and can be published together or separately as appropriate. In any case, the two parties should discuss the best way to disseminate the information and must recognize and respect each other's' contributions either by authorship or acknowledgement. In the case of co-authorship, a contribution to the writing of a paper is both a privilege and requirement. Cases of disagreement or potential conflict arising from prior agreements (or more likely, lack of prior agreements) will be resolved through the Office of the Vice-President for Research in consultation with the Director of the MCF, or an ad hoc committee of their choosing.

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- Use of facilities only (PI and associates carry out the research) requires acknowledgement of facilities in all resulting publications and presentations. The following acknowledgement should be used: "***Use of the TAMU Materials Characterization Facility is acknowledged.***"
- Provision of routine procedures and methods by MCF Staff requires acknowledgement of facilities and individuals in the resulting publications. The following acknowledgement should be used: "***Use of the TAMU Materials Characterization Facility and (Dr./Mr./Ms.) _____ are acknowledged.***"
- Contributions of MCF Staff that include ***development and implementation of original techniques, experimental design, interpretation, or other significant intellectual input require co-authorship*** and acknowledgement of facilities. The following acknowledgement should be used: "***Use of the TAMU Materials Characterization Facility is acknowledged.***"