• JEOL JSM 7500F Instructions version1.6

Log On

1. Fill the LN2 dewar. It will take 20 minutes to cool. This will last for 3 hours.

2. Fill in the logbook.

3. Insert a flash drive into the USB port.

4. When you get the message “Open folder to view files using Window Explorer” select OK, then minimize drive F.

5. Select PC-SEM.

6. Select Guest, no password, then select the Start button.

Sample Insertion

* *Gloves must be worn at all times when touching the stubs, samples, and sample holders. This includes sample mounting and any subsequent touching of the samples, stubs, or holders.*

1. Place the sample in the specimen holder. Position the top of the sample even with the top of the holder. **If you cannot do this, then carefully measure the distance above the top of the holder with the millimeter ruler**

2. Check to make sure the SPEC. EXCHANGE button is illuminated.

3. Select the Condition button at the right side of the screen.

4. Activate the specimen chamber IR camera: Start>Programs>IR Camera

5. Select the Vent button under the image of the sample holder then select OK then **unlatch the door immediately.**

6. Wait until VENT light on the side of the exchange port stops blinking, then open the door.

7. Press the specimen holder into place. It helps to start on one side first.

8. Close and latch the door.

9. Select the EVAC button under the image of the sample holder then select OK.

10. The pump time is variable and depends on any moisture present in the sample.

11. Wait until the EVAC light on the right side of the exchange port stops blinking. Also, the door valve should open on the SEM diagram.

12. Move the specimen exchange rod from the vertical position to the horizontal position. Then allow the vacuum to pull the rod in. Slight gentle pushing may be needed at the end of the travel. If you feel a restriction in the movement - STOP - and contact lab personnel. Push the rod in firmly until it stops moving.

 *Watch the IR camera - you should see the holder on the stage.*

13. Wait until you see the Select specimen holder window appear and **verify that the HLDR light on the right side of the exchange port is illuminated.**

14. Pull the rod all the way back, and while pulling it, move it upward to rest the rod on the metal rest plate. Then continue to move it to the vertical and move it into the lock position.

15.  **Select the proper specimen holder on the Select specimen holder window.** The holder selected should be outlined in red. Then select OK.

16. **Enter the specimen offset value from step 1 if your sample is above the top of the holder**.

**17. Wait until the vacuum recovers to the normal value of 9.6 x 10 -5 Pa.**

*This can take up to ten minutes*

*or more depending on your specimen.*

Startup

1. Make sure the ZFC button at the top of the screen is on (highlighted in green). If it is not, select it to turn it on.

2. Select the accelerating voltage.

3. Select the working distance. The best range is determined by the accelerating voltage, the amount of tilt and which detector being used. Use the following suggestions.

(1) General imaging above 50,000 X with no tilts. Use 5 kV, a working distance of 4.5 mm, and the SEI detector.

(2) General imaging above 50,000 X with some tilt needed. Use 5 kV, a working distance of either 6.0 or 8.0 mm, and the SEI detector.

(3) General imaging of less than 50,000 X with or without tilt. Use 5 kV, a working distance of 15 mm and the LEI detector.

(4) **Imaging of any magnetic sample. Consult with CAM staff! In general, use 5 kV, a working distance of 8 mm, and either the SEI or LEI detector.**

(5) Imaging and EDS analysis. Use 15 to 20 KV, a working distance of 8.0 mm and either the SEI or the LEI detector.

 Then select the working distance below the image, double click, then select OK. The sample will automatically move to the selected working distance; you should watch this on the IR camera.

4. When the holder is in position, close the IR camera.

5. Select an Emission Current of 20 µA. This is best for most imaging. If you have beam sensitive samples, select 10 µA.

6. Select the software Observation button ON. It should go from white to green and the emission current should gradually rise from 0 to either 10 µA or 20 µA over a period of 1 to 2 minutes. When it stops increasing, you may proceed. Doing this turns on the accelerating voltage and gradually increases the gun extraction voltage.

7. Press the GUN button on the column console; it should go out slightly and the yellow light will go off. You should verify that the gun valve opens by watching the black line move under the gun on the SEM column diagram on the screen.

8. Turn the magnification knob to the lowest setting.

9. Move the sample to the correct focal plane by focusing using the outer ring on the trackball.

*This focuses by moving the sample Z position to the proper focal plane. This is essential - do not use the focus knob at this point. You may move the sample using the trackball if needed. You may need to adjust contrast and brightness by pressing the ACB button.*

10. After focusing with the outer ring, you may now focus using the focus knob.

Image Viewing

1. The image contrast and brightness may be adjusted automatically at any time by selecting the blue ACB button at the top of the screen or by selecting the ACB button on the Operation Panel.

2. Sample movements X and Y:

(1) To make large movements, use the X +, X-, Y+, and Y- on the Specimen Stage Panel. The C button (coarse, when lighted), toggles between course and fine movements.

or

(2) Use the trackball

(3) for fine movements, left click and hold the mouse to drag the image. Use this only at magnifications of 10,000 X and higher.

*The X, Y, R (rotation), Z, and T (tilt) coordinates can be read on the green oval and gray bars at the right side of the screen.*

*To view your location on the stub, select the Stage Map tab*

*Right click anything on the image and select Stage Move to Center to center the item*

3. To go to the low magnification mode, press the Low Mag button on the Operation Panel or select LM on the screen. The WD readout on the image is not accurate in this mode.

4. To tilt the sample, select the Tilt + or - buttons on the stage panel.

5. To rotate the sample, select the Rotate + or - buttons on the stage panel. The sample can also be rotated by deselecting the ZFC button at the top of the screen; it should be highlighted in white. Then use the outer ring on the trackball to rotate the sample.

6. The full screen image may be viewed at any time by selecting the Full Image button on the right side of the screen. To go back to reduced view, select the Standard button at the top.

7. Five imaging modes buttons are displayed at the top of the screen. They may be selected at needed.

(1) Quick 1. This is a rapid mode, with 16 frame averaging. It is good for watching stage movements and some alignment procedures. It will be grainy in most situations.

(2) Quick 2. This is also a rapid mode, with 32 frame averaging. It will give better image quality than Quick 1.

(3) Fine 1. This is a slower mode without frame averaging. It will give a better quality image but is not appropriate when moving the sample.

(4) Fine 2. This is slightly slower than Fine 1.

(5) Freeze. This combines both a slower speed and 128 frame averaging. It provides the best image quality and simulates what you would obtain from a photograph. It saves the resulting image until you select the button again to unfreeze it.

8. Periodically check the emission current. If is has dropped significantly from 10 µA.or 20 µA., then select the Reset button.

9. The Probe Current should normally be set at 7, except if you are doing EDS.

Astigmatism Correction

1. Make sure the STIG button on the Operation Panel is on (illuminated in green).

2. Using the focus knob and the X and Y stigmator knobs, correct the astigmatism.

Changing the Accelerating Voltage

1. Select the software Observation button OFF. It should turn from white to gray. The emission current should go to 0. This insures that the electron beam is off.

2. Select the accelerating voltage.

3. Select the software Observation button ON. It should go from white to gray; the emission current should gradually rise from 0 to either 10 µA or 20 µA over a period of 1 to 2 minutes. When it stops increasing, you may proceed.

Saving an Image

1. To take a photo, first select one of the ACB buttons.

2. Then select the Photo button at the top of the screen. It takes approximately one minute.

3. When the Save as: panel appears, do the following:

(1) select Save in: drive F:, the flash drive

(2) give your image a name in File name:

(3) check the 🞏 export box to keep the information bar at the bottom of the image.

4. When finished, select the Freeze button at the top to unfreeze the image (it will go from green to white).

5. To change the Photo conditions, select Setup>Operation Settings> Image/Scan. You may change the Speed to any of the settings. We recommend Photo 1 (1m 4s) or Photo 2 (2m 8s). However, for charging samples, you may want to select shorter setting. For Image Size, we recommend 2560x 1920. You may if you wish, change it to 1280 x 960 or 5120 x 3820.

*Do not change any of the other conditions under Operation Settings!*

Printing Images

1. To see a “thumbnail” display of your current images, select the Image File button at the far right side of the screen.

2. Select the Browse button at the bottom and select Drive (F:), then select OK.

3. Select File>Report

4. Select Setup> 2 images

5. Select thumbnail images from the image filing area, left click and drag them into the report page, then left click again to paste them into the report.

6. Then select File> Print> Print on the Layout Sheet.

7. When finished, close the Layout Sheet, then select No.

8. Select the Condition button on the right side to display operation conditions.

Sample Removal

1. **Press the GUN button on the column console; it should go in slightly and will turn yellow.** You should verify that the gun valve closes by watching the black line move under the gun on the SEM column diagram on the screen.

2. Select the software Observation button OFF. It should turn from white to gray. The emission current should go to 0. This insures that the electron beam is off.

3. Open the specimen chamber IR camera.

4. Select the software Spec. Exchange button in the lower right hand corner of the screen.

**Wait until the EXCH POSN button on the side of the**

**exchange port is illuminated.**

5. Move the specimen exchange rod from the vertical position to the horizontal position. Then allow the vacuum to pull the rod in. Slight gentle pushing may be needed at the end of the travel. If you feel a restriction in the movement - STOP - and contact lab personnel. Push the rod in firmly until it stops moving.

6. Pull backward on the exchange rod. You should be able to see the sample holder move with the rod on the IR camera.

7. Pull the rod all the way back, and while pulling it, move it upward to rest the rod on the metal rest plate. Then continue to move it to the vertical and move it into the lock position.

8. Close the IR camera by selecting X

9. Select the Vent button under the image of the sample holder then select OK then **unlatch the door immediately.**

10. Wait until the door vents (approximately 20 seconds). The VENT light on the side of the exchange port will stop blinking.

11. Then open the door and remove the sample.

12. Close and latch the door.

13. Select the EVAC button under the image of the sample holder then select OK.

Shutdown after Sample Removal

1. Select File then select Exit

2. When you get the JEOL screen, select Exit

3. Select Start> My Computer> right click on drive F: then select Eject. Remove you flash drive and close My Computer.

4. Select Start, then select Log Off, then select Log Off again.

5. Fill in the logbook.

**Appendix**

Tilt Limits

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Holder mm | 1.5 mm | 2 mm | 3 mm | 4.5 mm | 6 mm | 8 mm\* | 15 mm\* | 20 mm\* |
| 12.5 | 0° | 0° | -5 to + 5° | -5 to + 15° | -5 to + 20° | -5 to + 25° | -5 to + 55° | -5 to + 60° |
| 32 | 0° | 0° | -5 to + 5° | -5 to + 15° | -5 to + 20° | -5 to + 25° | -5 to + 45° | -5 to + 55° |

\*Use the LEI detector instead of the SEI detector. Consult CAM staff for more information.

Gentle Beam Mode - electron beam deceleration

1. If a sample is currently being viewed, select the software Observation button OFF. It should turn from white to gray. The emission current should go to 0. This turns the electron beam off.

2. Select either GB-H (sample high position) or GB-L (sample low position).

|  |  |  |  |
| --- | --- | --- | --- |
| Mode | Working Distance | Tilt  | Landing Voltage |
| GB-H | 1.5 mm | 0 | 0.1 to 2.8 kV |
| GB-L | 2.0 to 8.0 mm | as allowed by WD | 0.6 to 4 kV |

3. Select the Observation button ON

4. If you want to make changes to the standard conditions, select the bar under the Accelerating Voltage in the Observations Conditions area at the bottom of the screen to open the dialog.

R-filter - The R-filter is an energy filter. It works with the upper detector only, the SEI detector

1. Normally, the SB button should be selected, filter off.

2. The Slider bar adjusts the intensity in the mode you have selected.

3. The value in the box to the right of r-filter indicates the filter intensity.

4. SE button. SE only mode, default slider value is 100, i.e. full voltage on the filter to filter out as many backscattered electrons as possible. The slider can be adjusted from 100 to 0 as needed.

5. SB button. Filter off, all SE and BSE are detected. The slider can be adjusted to the left to SE mode, or to the right BSE mode.

6. BE button. BSE dominant mode, default slider value is 60. The slider can be adjusted from 100 to 0 as needed.

Users are not to do any of the following:

1. Change any of the parameters under Operation Settings other than Image/Scan information.

2. Select any of the recipe conditions on the screen. Doing so will change microscope parameters to these conditions automatically and can result in damage to the SEM. These settings have little value for anyone trained in SEM. A full chart of the information is on top of the computer.

Emergency Stop - In the event of an emergency, such as a fire, flood, smoke, etc., please push the red emergency stop button on the lower right side of the operation console.

Stop Here

Sample Preparation Guidelines for the 7500F SEM

1. Mounting materials. The only adhesives allowed are properly dried carbon cement, properly dried rapid cure epoxy cement and the special carbon tabs (not carbon tape) provided by the Center for Advanced Microscopy. **All other materials are specifically prohibited.** For the best results, use the minimum amount of adhesive possible.

2. Adhesive drying times.

Carbon cement - 4 hours at room temperature

Rapid cure epoxy - 12 hours at room temperature.

3. Never use carbon cement to mount samples like glass cover slips, silicon wafers, etc. It will not dry. Use only the rapid cure epoxy on samples like these.

4. Although the special carbon tabs are approved for use in the 7500F, they may not provide the best results above 100,000X and may take up to 40 minutes to stabilize in the vacuum.

5. For the best resolution, the smaller the sample the better the results. All samples will release residual materials in a vacuum (outgas) when placed in the ultrahigh vacuum of the 7500F and this will impact resolution. Also, thermal expansion and contraction is more of a problem with larger samples.

**6. Gloves must be used at all times when touching samples and stubs.** The cleaner the conditions, the better the results.

7. Only stubs provided by the Center for Advanced Microscopy are to be used. These stubs have been ultrasonically cleaned.

8. Sample height. All samples on a given stub should be the same height. If not, there will be problems in selecting the correct working distance. If samples vary in height, then mount the samples on several stubs, each with the same height samples.

9. Stub information

(1) For samples up to 25 mm diameter and up to 10 mm thick, use 25 mm diameter stubs.

samples 0.0 mm to 5 mm thick - use stubs 10 mm in height

samples 2.0 mm to 10 mm thick - use stubs 5 mm in height.

(2) For samples up to 12.5 mm diameter and up to 7.5 mm thick, use 12.5 mm diameter stubs. samples 0.0 mm to 1 mm thick - use stubs 10 mm in height

samples 0.0 mm to 7.5 mm thick - use stubs 5 mm in height.

10. Store your samples in a vacuum for the best results.

**11. Samples with magnetic materials present special problems.** Consult CAM staff for assistance before placing these samples in the 7500F.

Low Magnification Imaging

Images collected in the Low Mag Mode will not have resolution equivalent to the Normal Mode. If low magnification images are needed, much better results will be obtained by increasing the working distance to 20 mm and using the LEI detector. The lowest magnification at the 20 mm working distance is 100X.

Oxford EDS System on the JEOL 7500F

1. Log on to the 7500F, open PC\_SEM.exe, then select Guest.

2. Start the Oxford computer if turned off and wait until you get the log on screen. It can take up to two minutes for the keyboard/mouse switch to activate.

3. Log on under Operator. There is no password.

4. Insert your flash drive if you need to save information.

5. Start the AZtec program.

6. It is not necessary to do a quant optimization

7. You do not have to enter the accelerating voltage and working distance. The INCA program obtains this information from the 7500F.

8. The working distance on the 7500F must be 8 mm.

9. The IR camera on the 7500F must be closed.

10. Set your dead time in Microscope Setup before going to Acquire Spectrum.

11. To control the dead time, use the Probe Current adjustment in the 7500F; higher numbers give more probe current and a higher dead time.

12. If you save anything as a Word File, the default setting is Word 2013.

13. When finished, close the AZtec program, select Start>Log Off.

If you are interested in the best analytical spatial resolution in EDS (for instance, in mapping), select an accelerating voltage of 8 kV. For many of the elements, you will then have to use the low energy L lines as opposed to the higher energy K lines. This is usually not a problem because the detector resolution is significantly better at the lower X-ray energies.