STANDARD OPERATING PROCEDURE:  
JEOL JSM 7600F SCANNING ELECTRON MICROSCOPE

Purpose of this Instrument:  Structure observation at nanometer scale resolution; quantitative material chemical composition analysis and elemental X-ray mapping; electron beam lithography of complex patterns at nanometer scale precision on resist-coated surfaces.

Location:  Engineering Science Building (ESB) Room G-73 (cleanroom)

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The Shared Research Facilities are operated for the benefit of all researchers.  If you encounter any problems with this piece of equipment, please contact the staff member listed above immediately.  There is never a penalty for asking questions.  If the equipment is not behaving exactly the way it should, contact a staff member.

NOTE:  The purpose of this manual is for general specimen observation with the SEM. For more advanced operations or observations with accessories (e.g., backscatter electron detectors, etc.), please contact a Shared Research Facilities staff member. You may also refer to the JEOL 7600F SEM instruction manual (pdf version), which is located on the SEM computer desktop. Please refer to the specific SOPs for EDS and E-beam Lithography operations.

START UP

1.  Log in your session on the FOM. Write down your name and other required information on the SEM log sheet located in the notebook at the machine.

2.  Start the SEM program PC_SEM (log in as Guest) and the IR Camera program on the computer desktop.

SAMPLE PREPARATION

1.  Mount the specimen on a proper specimen stub with adhesive (e.g., carbon tape, copper tape, silver paste, etc.) available on the sample preparation workbench.

2.  Mount the specimen stub onto a proper specimen holder (stored in the orange colored storage box on the sample preparation workbench). Lock the specimen stub with screws.

3.  Estimate the distance from the top of the specimen holder to the specimen surface. You need to input this value (specimen surface offset, in millimeters) after loading the specimen into the SEM vacuum chamber. IMPORTANT:  It is important that you properly estimate the specimen surface offset, which will be used by the SEM program to determine the proper stage height when you select a working distance (Stage Height = Working Distance + Specimen Surface Offset), as shown in Figure 1. Underestimating the specimen surface offset may cause the specimen in contact with the lens column and lead to specimen and stage damage.
**SAMPLE LOADING**

1. Click on the “SPECIMEN” button on the right side of the PC_SEM program window (Figure 2a). Check the SEM monitor section (Figure 2b) at the lower right corner of the window. If the “Exchange Position” button is not solid green, click on this button to have the stage move to the exchange position.

2. Click on the “VENT” button in the SEM monitor section (Figure 2b) to vent the specimen exchange chamber. The VENT button blinks until the chamber reaches the atmospheric pressure and then becomes solid green.

3. Open the front door of the SEM enclosure by flipping the switch marked “Front door” twice (located on the right hand side of the enclosure).

4. Unlock the locking hook on the right side of the specimen exchange chamber and open it.

5. Slide the specimen holder into the transfer chuck following the direction of the arrow mark on the side of the specimen holder (circled in the Figure 3). **Always keep the arrow towards yourself.**

6. Check to ensure that the airlock O-ring is properly seated in the groove. Remove any dust or debris on the O-ring surface with a clean wiper.

7. Close the door of the specimen exchange chamber and then lock it with the locking hook.

8. Click on the “EVAC” button in the SEM monitor section (Figure 2b). This evacuates the specimen exchange chamber. During the evacuation the “EVAC” button blinks.

9. Wait until “EVAC” button stops blinking and the gate valve between the exchange chamber and main chamber is open.

10. Lift the specimen transfer rod upward. **Rotate until it becomes horizontal while still pulling the rod (up and out).** Insert the rod all the way. Users can look on the IR camera to make sure that the sample is mounted in the chamber. **IMPORTANT:** The user should not have to apply a significant amount of force to the rod. If the rod will not insert, please see a Shared Research Facilities staff member. Forcing the rod to insert into the load lock may damage the o-rings and destroy the vacuum seal.

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*Figure 1. Relationship between the working distance, specimen surface offset and stage height*
Figure 2. (a) PC_SEM program interface; (b) SEM Monitor section

Figure 3. Inserting specimen holder to transfer chuck (from JSM 7600F SEM instruction manual)

11. Pull the specimen transfer rod all the way out until the end of the pipe comes out from the guide (Figure 4). Rotate the rod while still pulling the rod out and up until it attaches to the holding device (rod in vertical position). Release the rod.
**WARNING**: If you do not pull the specimen transfer rod all the way out as shown below, you will damage the rod when you try to rotate the rod, which will result in several days of instrument down time and a service visit from JEOL in addition to the rod repair/replacement cost.

![Figure 4. Pull the transfer rod all the way out until the end of the pipe comes out from the guide (from JSM 7600F SEM instruction manual)]()

12. Close the front door of the SEM enclosure.
13. The SEM specimen holder selection dialog will appear on screen. Select the SEM holder you are using from the list. Type in the specimen surface offset value (noted in Sample Preparation step 3) at the bottom section of the dialog. This value is used by the system to determine the proper Z height for a selected working distance setting (see Step 2 in next section).
14. Monitor the vacuum reading in the main chamber in the SEM monitor section (Figure 2b). Wait until the pressure in the main chamber goes down to $9.6 \times 10^{-5}$ Pa before starting the observation.

**WARNING**: Starting the observation by turning on the electron beam before reaching the ultimate vacuum level will shorten the lifetime of the filament and cause unnecessary downtime of the system for filament replacement.

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**SAMPLE OBSERVATION**

1. Set the desired Accelerating Voltage on the top left corner of the SEM program menu (Figure 5). You can type in any Accelerating Voltage value between 0.5 and 30 kV if the desired value is not shown in the list.

   **NOTE**: You will get better image resolution by using high accelerating voltage but it will also cause charging issues if the sample is not conductive. Select a high Accelerating Voltage (e.g., 20-30 kV) for conductive samples. For nonconductive samples, select a lower Accelerating Voltage (e.g., <10 kV) to reduce the charging effect. Contact a Shared Research Facilities staff member if you need help.

2. Select a working distance (WD) between 6–8 mm at the bottom of the image observation window (Figure 5). The stage will automatically rise to the set Z height based on the given specimen surface offset (see Figure 1). Monitor the motion of the stage in the IR camera (Figure 1).

   **NOTE**: Generally you will get better image resolution at a smaller working distance. However, when operated at a very small working distance (e.g., 1.5 mm), the sample may accidently hit the lens column (especially if the specimen surface offset is underestimated, see Figure 1) and result in sample and stage damage. A **working distance between 6-8 mm is sufficient for general observation**. Contact a Shared Research Facilities staff member if you need help.
3. Click on the “ON” button in the SEM program menu bar under “Observation” (Figure 5) to turn on the irradiation of the electron beam on the specimen. The electron beam will start scanning on the sample surface and the SEM image window will become brighter. If the SEM image window remains solid black after turning on the beam, the image scanning is being frozen (the “Freeze” button above the image window in Figure 5 is in solid green). Click on the freeze button to unfreeze the scanning (Freeze button turns to grey).

4. Check the current magnification displayed under the image window (Figure 5). Turn the MAGNIFICATION knob on the operational panel (Figure 6a) counter clockwise until the magnification value does not decrease.

5. Click on “Quick 2” button above the image window (Figure 5) to observe the sample with rapid scan rate. Focus on the sample surface with the FOCUS knob on the operation panel (Figure 6a) until you observe some feature on the sample surface.

6. If you could not see anything on your sample surface, press the “LOW MAG” button on the operation panel (Figure 6a) to observe the sample at Low Magnification mode. Turn the MAGNIFICATION knob counter clockwise until the magnification decreases to 25×. Move the specimen around and locate the site of interest on the specimen. Move the site of interest to the center of the screen with the X, Y motion control on the specimen stage control panel (Figure 6b) or the track ball. Gradually increase the magnification to over 1,000× while keeping the site of interest in the center of the image window. Finally, press the “LOW MAG” button again to return to regular SEM observation mode.
7. Press the “ACB” button to automatically adjust the image brightness and contrast. You may also manually adjust the image brightness and contrast by turning the BRIGHTNESS and CONTRAST knobs on the operational panel (Figure 6a).

8. Click on the “Fine 1” or “Fine 2” buttons above the image window (Figure 5) for slow scan observation (better resolution than rapid scan Quick 1/Quick 2).

![Figure 6. (a) Operation panel; (b) Specimen stage control panel](image)

**BEAM ALIGNMENT**

**NOTE:** Monitor the position of an object on the sample surface while turning the FOCUS knob back and forth. If you notice the object moves on the screen as you change the focus, the electron beam is not aligned. You need to perform the beam alignment steps as follow to align the electron beam with respect to the objective aperture.

1. Press the “WOBB” button on the operation panel (Figure 6a). The image may move periodically along X and/or Y directions as the focus changes periodically.

2. Adjust the X and/or Y knobs on the operation panel (Figure 6a) to minimize the image movement.

3. Press the “WOBB” button again to stop the wobbling when the image shift is minimized. The image should remain stationary when the focus changes periodically.

**NOTE:** If you are having problems with the beam alignment, please contact a Shared Research Facilities staff member for help.

**ASTIGMATISM CORRECTION**

**NOTE:** If the image expands diagonally (blurs in an oblique direction, as shown in Figure 6) when you try to focus it, the image has astigmatism that needs to be corrected.

1. Find some small features (e.g., particles) on the specimen surface.

2. Use the FOCUS knob to adjust the focus to the condition where directional blurring disappears (the image is blurry but does not slant in any direction, as shown in the middle figure in Figure 7). To find the focus condition, you need to turn the focus knob back and forth to find the two positions that the image slants
in two orthogonal directions, as shown in Figure 7. Then, the position half way in between these two slanting condition positions is the focused position.

3. Press the “STIG” button on the operation panel (Figure 6a).

4. Adjust the X and Y knobs (Figure 6a) on the operation panel to maximize the sharpness of the image.

5. Increase the magnification. Repeat steps 2–4 for sharper image up to the magnification that you want to record the image. Typically, one should first correct the astigmatism at low magnification (e.g., 5,000×), then at a medium magnification (e.g., 20,000×) and finally at a high magnification (e.g., 50,000 × – 100,000×).

![Figure 7. Astigmatism correction (from JSM 7600F SEM instruction manual)](image)

**SAMPLE ROTATION**

**Stage Rotation:**

1. You can rotate the sample stage by specifying the angle of rotation in the stage position dialog box as follow:
   - Click on any cell in the stage position display section (X, Y, R, Z, T bar) to display the Set stage position dialog box (Figure 8a).
   - Enter the desired degree of rotation.
   - Click the “Move” button on the dialog box and the stage will start rotating to the specified angle.

2. You can manually rotate the specimen stage using the “ROTATION” buttons (-R and +R) on the specimen stage control panel (Figure 6b).
**Image Rotation:**

You can rotate the SEM image on screen as follow:

- Click on “Observation” button on the right side of the PC_SEM main window.
- Select the “Image Rotation” check box under the image window.
- Click on the **-** button and the Image Rotation dialog appears (Figure 8b).
- Click on the Horizontal or Vertical setting buttons (Figure 8b). Draw a line at the place where you want the image to be horizontal/vertical. The image will be rotated accordingly.
- You can also directly type in the desired rotation angle.
- When finished, uncheck the “Image Rotation” check box.

**NOTE:** The stage motion is not affected by image rotation. If you move the stage when the image is rotated, the stage will move along the X-Y directions before image rotation.

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**SAMPLE TILT**

**WARNING:** Tilting the sample holder without enough clearance between the specimen holder and the lens column can result in instrument damage.

1. Lower the stage Z height (*e.g.*, to 15 mm) to provide more clearance for tilting.
2. Click on any cell in the stage position display section to display the Set stage position dialog box (Figure 8a).
3. Enter the desired tilt angle in the T (deg) section (Figure 8a).
4. Click on “Move” on the dialog box and the stage will tilt to the desired angle. Monitor the stage tilt in the IR camera. **For emergency stop of the stage motion, press the Stop button in the Stage Control window or move the trackball in any direction.**
5. You can also manually tilt the specimen stage using the “TILT” buttons (-T and +T) on the specimen stage control panel (Figure 6b).
6. Raise the stage to the desired height for sample observation. Monitor the stage position in the IR camera to ensure that the sample holder does not hit the lens column above. **For emergency stop of the stage motion, press the Stop button in the Stage Control window or move the trackball in any direction.**
7. Focus on the specimen surface for observation.
8. When you finish observation, lower the stage height (e.g., to 15 mm).
9. Enter 0° for Tilt in the Stage position dialog box and click on “Move”. You may also use the TILT buttons to manually tilt the specimen stage back to zero position.

**IMAGE RECORDING**

1. Set a proper field of view in the observation window.
2. Select *Setup > Operation Settings* from the menu bar to open the Operation Settings dialog. Click on the “Image/Scan” tab. Verify the setting in the “Photo button” section: Speed is Photo 4 (slowest speed), Image Size is 1280 × 960, Image format is TIFF.
3. Click on the “Set” button to apply the changes.
4. In the same Operation Settings dialog, click on the “Photo & Print Data” tab. Select the check boxes for the information that you want to display on your saved SEM image (e.g., Date, Magnification, Accel. Voltage, Signal, Working Distance, etc.).
5. Click on the “Set” button to apply the changes. Close the Operation Setting dialog.
6. Press the “PHOTO” button on the operation panel or click on the “Photo” button above the image window to record a slow scan SEM image.
7. After the image freezes (freeze button becomes solid green), the Save As dialog will appear. Set the proper image file storage settings (e.g., create a new folder for the SEM session). Make sure the “Export” box is checked on the Save As dialog. Save the image.
8. Click on the “Freeze” button to restart the observation.

**NOTE:** Users are responsible for copying their images off the SEM computer or hard drive. The WVU Shared Research Facilities Cleanroom is a multi-user facility, and cannot guarantee that image files may not be corrupted or deleted.

**SAMPLE UNLOADING**

1. Set the magnification to 300,000x.
2. Click on the “Specimen” button on the upper right side of the PC_SEM program window.
3. Click on the “OFF” button under “Observation” on top of the program window to shut off the electron beam.
4. Click on the “Exchange Position” button in the SEM monitor section of the program window (Figure 2). The user should see the sample stage move to the exchange position (X=0; Y=0; R=0; Z=38 mm) in the IR camera window. Wait until the “Exchange Position” button turns solid green.
5. Open the front door of the SEM enclosure by flipping the front door switch twice.
6. Lift the specimen transfer rod upward. Rotate until it becomes horizontal while still pulling the rod up and out. Insert the rod all the way.
7. **Pull the specimen transfer rod all the way out until the end of the pipe comes out from the guide** (see Figure 4). Rotate the rod **while still pulling the rod out and up** until it attaches to the holding device (rod in vertical position). Release the rod.

   **WARNING:** If you do not pull the specimen transfer rod all the way out, you will damage it when you try to rotate the rod. Such damage will result in several days of instrument down time and a service visit from JEOL in addition to the rod repair/replacement cost.

8. Click on the “VENT” button in the SEM monitor section (Figure 2b) of the program window to vent the specimen exchange chamber. The “VENT” button blinks until the chamber reaches atmospheric pressure and then becomes solid green.

9. Unlock the locking hook. Open the specimen exchange chamber and take out the specimen holder by slightly lifting it up.

10. Check to ensure that the airlock O-ring is properly seated in the groove. Remove any dust or debris on the O-ring surface with a clean wiper.

11. Close the door of the specimen exchange chamber and lock it with the locking hook.

12. Close the front door of the SEM enclosure.

13. Click on the “EVAC” button in the SEM monitor section (Figure 2b) of the program window to evacuate the specimen exchange chamber. During the evacuation process, the “EVAC” button blinks. Wait until it stops blinking and turns solid green.

**SHUT DOWN**

1. Remove your sample from the specimen stub.
2. Remove the specimen stub from the specimen holder.
3. Remove adhesive from the specimen stub. Clean the specimen stub with wiper and alcohol supplied on the specimen preparation workbench.
4. Store the specimen stub in the tool box and the specimen holder in the orange holder storage box.
5. Clean up the SEM specimen preparation workbench by putting away all holders, adhesive materials, and tools. Make sure you remove all your samples.
6. Copy all desired image files off the SEM computer or hard drive.
**EMERGENCY PROCEDURES**

If a user ever has a problem, even if slightly unsure, ASK someone who knows and can help. There are no penalties for asking for help but there may be for not reporting damage to the equipment that may delay or prevent others from working.

If, at any time, the user needs to contact someone for help, call or locate the following staff of the Shared Research Facilities (SRF):

- **Weiqiang Ding**
  - Office: ESB G75D
  - Phone: (304) 685-1938 cell
- **Marcela Redigolo**
  - Office: ESB G75D
  - Office phone: (304) 293-9973
  - Cell: (304) 680-3007
- **Kolin Brown**
  - Office: ESB G75D
  - Phone: (304) 366-6551 cell

If no one is available and the SEM is not acting as expected, the user should do the following:

- Shut OFF the electron beam (OFF button under “Observation” on top of the PC_SEM program window)
- If the PC_SEM program freezes, open the front door of the SEM enclosure chamber and press the GUN button in front of the specimen exchange chamber to shut off the electron beam.
- Exit the PC_SEM program
- Shut down the computer

Then, if possible, the user should stay with the SEM while trying to contact the above individuals. If it becomes necessary to leave the instrument then the user should leave a large, legible note on both the SEM and at least one of the above individuals’ offices, stating:

- The problem (describe what happened and steps taken)
- When it occurred (date and time)
- User name and phone number

If a dangerous situation is evident (smoke, fire, sparks, etc.), ONLY if it is safe to do so, the user should press the RED emergency off button at the front of the SEM control console to turn OFF power to the entire SEM system and evacuate all persons from the Cleanroom. The user should leave the facility and contact emergency personnel as soon as possible from a safe place.