JEOL 7900FLV

NUANCE
Atomic and Nanoscale Characterization Experimental Center

Northwestern
EXPLORING INNER SPACE
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A. Policies and Introduction

Reservations

JSM-7900F reservations are made using the NUCore online reservation system. Start your reservation before you begin using the instrument. When your session is complete, be sure to end your reservation in NUCore. If you need extra time on the microscope, we recommend ‘extending’ your original reservation, rather than making an additional reservation.

There is a hardware control system on the JSM-7900, so the system will not function unless you are logged in to NUcore. Tampering with or disabling the hardware control may result in revocation of your EPIC privileges.

Saving Your Data

During your session, you may store image data to your own folder within the EPIC_SEM drive (S:\). EDS and WDS data must be saved on the D:\ drive then transferred onto your folder on the S:\ drive. The SEM server is accessible through the computer in the hall. You can transfer your data from this SEM server computer to a USB, etc. You should NEVER take your data directly from the SEM computers.

SEM Rules

1. Please wear gloves when handling any components and samples that will go into the SEM.
2. Do not install any software onto the SEM’s PC.
3. Do not insert any flash drives into any microscope computer!
B. Interface Overview

1) Menu bar and Toolbar
2) a. Electron Image Display
   b. Display Modes
3) a. Beam adjustment tools
   b. Buttons equivalent to tabs (arrow)
4) Stage navigation and tools
5) Stage Coordinates
6) SEM monitor

Toolbar Icons:

- Turn Beam on/off
- Emission or filament toggle
- Scan speeds
- Charge-free scan mode
- Display Freeze
- Auto Focus
- Auto Contrast/Brightness
- Zero the beam shift
- Z Focus Control
- Measurement tools
- Annodization
- Scan rotation on/off
- Chamber camera on/off
Operation panel:

1. Scanning Mode
   a. QUICK VIEW button: fast scanning speeds
   b. FINE VIEW button: slow scanning speeds
   c. RDC Image button: area scan reduced to 1/4

2. Magnification
   a. LDF button: large depth of focus mode for a low magnification imaging
   b. Magnification knob: adjusts magnification

3. Focus
   a. AUTO button: performs the automatic function (auto focus, auto focus + auto stigma, or adjust Z). Function chosen in Setup > Operation Settings > Auto function tab.
   b. Focus knob: adjusts the working distance.

4. Alignment
   a. WOBB button: turns on the wobbler which fluctuates from over/under focus for aperture alignment. Multifunction X/Y knobs adjust aperture position.
   b. Align: opens Electron Beam Alignment tab and starts the wobbler.
   c. STIG: Multifunction X/Y knobs will adjust stigmation
C. Start Up

1. Log in to the NUcore system and begin your reservation
2. If not already logged in, log in to the Guest account on the JEOL software.
3. There is no password
4. On the right hand side of the main computer screen, select Observation for a live inside view of the chamber

D. Preparing sample holder

IMPORTANT!
- If loading multiple samples, they should not vary in height more than about 2–3 mm. Do not put in samples of drastically different heights!
- For any sample holder, you must measure any offset (in mm) from the top of the sample holder to the top of the tallest sample.

1. Single sample holder (12.5 mm)
   a. If using a pin stub, set your pin stub into the stub adapter
   b. Drop your stub or stub/adapter into the cylinder of the single sample holder and tighten the set screws.
   c. Use screw on the bottom of the sample holder to adjust height so that it is as flush with the top as possible.

2. Standard sample holder (32 mm)
   a. If using pin stubs, mount into multi-pin holder and tighten the set screws for each pin stub.
b. Place specimen stub, multi-pin holder, or sample puck into cylinder.

c. Use screw on the bottom of the sample holder to adjust height so that it is as flush with the top as possible.

3. STEM sample holder (NOTE: Requires separate training)

4. GBSH Sample holder (NOTE: Requires separate training)

E. Sample Loading

1. Unlatch the latch on exchange chamber door.

2. Vent the exchange chamber by holding down on the **Vent** button on the side of the exchange chamber or clicking the Vent button in the **SEM Monitor**.
   
   a. Venting is complete when **Vent** button stops blinking.

3. Open the exchange chamber and slide the sample holder in the direction of the arrows on the holder.
   
   a. **NOTE**: Try to keep the exchange chamber door closed and pumped as much as possible.

4. Close the door and latch the exchange chamber, and then hold down the **EVAC** button on the side of the exchange chamber or click **EVAC** in the **SEM Monitor**.
   
   a. Pumping complete when **EVAC** button stops blinking

   b. **NOTE**: The SEM monitor will show the gate valve between the main chamber and the sample exchange chamber has opened so the sample can be inserted.

   c. The system will automatically snap a picture of the holder for navigation.

5. Select **Spec. Exchange** button if not already selected (green) under the **SEM Monitor**.

6. Lower the exchange rod (rod will read “up/down only”)

7. Pull rod out slightly and turn right (clockwise) until it says
“in/out only.” Rod will go back in slightly.

8. Slowly push rod all the way in
   a. Rod will reach a stopping point a couple inches from being fully inserted and needs to be pushed in further.
   b. HLDR light on the stage panel should turn on and the Specimen Holder window should appear if holder is properly loaded. If holder isn’t detected, try pulling out and inserting more slowly.

9. Remove exchange rod, turn to the left (counterclockwise) until it says “up/right only” and return to the upright position.

10. In the Specimen holder pop-up window, select the sample holder you are using and input any offset from your specimen height and holder.
   a. It is imperative you input the correct offset to avoid crashing into the objective lens when you bring your sample to your desired working distance.

11. Wait for the vacuum to reach at least 7E-4 Pa on the SEM Monitor.

F. Setting Imaging Conditions

1. Set the accelerating voltage level either below the Menu Bar or within the Operation Condition tab of the bottom left section.
   a. High accelerating voltage: better resolution, loss of surface sensitivity, and increased charging effects
      i. Low Vacuum: high voltage will minimize beam skirting by the chamber gas

2. Select your probe current in the Operation Condition tab
   a. Smaller probe current: better resolution, but less signal (noisier image).
   b. Most common probe current is medium (8)
3. Ensure the LED (Lower Electron Detector) is selected  
   a. **NOTE**: If using other a different detector or mode, refer to the section *Detector Options*.

4. Select the ZFC button in the **Toolbar** if not already selected.

5. Select a WD of 10 mm at the bottom of the electron image display.
   a. A window will appear asking if you want to move to this distance or cancel.
      i. Selecting OK will link the stage to the WD, moving the stage up so that the position specified by your measured offset is in focus at the set WD. **If you did not measure the offset, please remove your sample and measure it!**
      ii. Selecting Cancel will set the WD but will not move the stage.

6. If the vacuum is <7E-4, turn the beam on by selecting **ON** under **Observation** (top left under the **Menu Bar**).
   a. **Note**: If you have a porous or large sample, this may take up to 10 min. Do NOT turn on the beam until the vacuum has recovered.

7. Move to your sample by A) using the trackball, B) right clicking on your sample in the Navigator tab (top right) and choosing “move to center”, or C) right clicking in the SEM image to “move to center.”

8. Focus using the stage focus control wheel around the trackball
   a. This adjusts the stage height until the sample is focused at the set WD. Best for coarse adjustments.
   b. Using the focus knob will change the WD without
moving the stage. Best for fine adjustments.

9. Ensure the Z-height is 10 mm or greater.

G. Alignments

1. Find a particle or textured surface to focus on and focus at a magnification of at least 10,000 x.

2. Activate the Wobbler in the Electron Beam Alignments tab or press the WOBB button on the Operation Panel.
   a. Use the X and Y knobs in the Alignment section of the operation panel to counteract any translation in the image until your feature remains stationary.

3. Select OL stigmator on the computer or press the STIG button on the operation panel to return the X/Y multifunction knobs to controlling stigmation.

4. **Important!**: Correct for astigmatism using X/Y multifunction knobs
   a. Press RDC Image button to reduce the scanning area
   b. Focus the beam up and down and identify the two directions of stretching. Find the middle point between the two directions of stretching (where it is in best focus).
   c. Adjust one X/Y knob at a time until you achieve the clearest image. Adjust focus if necessary and repeat with other knob.
H. **Image Capture**

1. Select the camera button that says “Normal” in the toolbar.
2. To change image capture settings, select Setup (S) > Operation Settings > Scan Setting
3. Change any Photo button option
   a. NOTE: Do not change any other settings in this window!
4. When prompted, save your images to the folder named with your NetID on the EPIC_SEM drive (S:).

I. **Detector Options**

1. **LED – Lower Electron Detector**
   a. Ideal for displaying topography, surface detail, and shape.
   b. Most efficient at WD > 6 mm. Signal decreases at shorter WD.
   c. The LED will show less charging artifacts than UED.
2. **UED – Upper Electron Detector**
   a. Ideal for high resolution imaging at low kV.
   b. Most efficient at short working distances < 6mm (minimum is 2 mm).
   c. Can be used for both SE and BSE imaging using energy filter.
   d. Used for Gentle Beam mode.
3. **RBED (BED-C) – (Retractable) Backscatter Electron Detector**
   a. Retractable backscatter detector–inserts at the base of the pole piece.
   b. Ideal for atomic-number contrast and topographic contrast
   c. Can be used at any working distance (min. is 2 mm).
4. LVBED-C – Low Vacuum Backscatter Electron Detector
   a. The LVBED is a backscatter detector used in low vacuum operation that is inserted into the tip of the pole piece.
   b. This detector can be used in both LV and HV modes
   c. See next section on LV operation

J. Low Vacuum Mode
   *Note:* You may want to adjust your beam under high vacuum mode with a conductive specimen prior to switching to low vacuum mode.

1. Make sure the beam is turned off by selecting the Observation Off button.

2. Choose degree of vacuum you would like in the specimen chamber and click Set.
   a. Pressure can range from 10 to 300 Pa.
      i. Higher pressure: less charging, worse resolution.

3. Click the LV button to switch to Low Vacuum Mode, click OK.
   a. LV button will turn green when process is completed, and the SEM monitor will turn a lighter shade of grey.
   b. The LV Orifice (LVBED detector) will automatically be inserted at the base of the pole piece and will be reflected in the SEM monitor.

4. Press the Observation On button to turn the beam on

5. To return to High Vacuum mode, press the HV button.
   a. You can then choose in the pop up to have the LV Orifice retracted after changing to HV mode or to leave it inserted.
K. Gentle Beam Mode

**Note:** GB Mode allows for high resolution imaging at extremely low accelerating voltages by applying a bias voltage to the specimen to decelerate a focused higher kV beam to a (gentler) lower kV beam.

1. Set your **accelerating voltage** to your desired final landing voltage.
2. Under **Observation Mode**, switch to GB mode.
   a. **Note:** When in GB mode, the background color for the accelerating voltage will turn from black to white.
3. Refocus and adjust stigmators as necessary.
4. Open the sub-panel for the Accel. Voltage. Choose the desired Specimen Voltage (the bias).
   a. **Note:** The Gun Volt value shows the accelerating voltage of the beam prior to deceleration.
5. Return to **SEM mode** when finished.

L. EDS with Aztec

1. Select an appropriate accelerating voltage
   a. Usually between 15–30 kV, can be lower.
   b. Accelerating voltage should be ~2.1x the highest energy peak you expect.
2. Pause the chamber scope.
3. Increase probe current to improve signal (10–14).
4. Focus sample at an 8 mm working distance. This is the analytical working distance for this microscope.
5. See separate AZtec manual for further instructions.

M. Shut Down and Sample Removal

1. Turn the beam off by selecting OFF under **Observation** (top
left under the Menu Bar).

2. Move the stage to the Spec. Exchange Position under the SEM Monitor

3. Bring sample into the exchange chamber
   a. Lower the exchange rod (rod will read “up/down only”)
   b. Pull rod out slightly and turn right (clockwise) until it says “in/out only.” Rod will go back in slightly.
   c. Slowly push rod all the way in
      i. Rod will reach a stopping point a couple inches from being fully inserted and needs to be pushed in further.
   d. Remove exchange rod, turn to the left (counterclockwise) until it says “up/right only” and return to the upright position.
   e. Ensure the HLDR light turns off

4. Unlatch the exchange chamber door

5. Hold down on the VENT button

6. When venting has completed, remove the sample holder from the exchange chamber

7. Close the door and hold down on the EVAC button

8. The EXCH POSN light should be green and the EVAC light should be solid before you leave.

9. Do not unscrew any set screws too much! They can get lost very easily!

10. END RESEVATION in the NUcore system