JEOL 7900FLV

NUANCE
Atomic and Nanoscale
Characterization Experimental Center

Northwestern
EXPLORING INNER SPACE

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must include the following acknowledgements:

“This work made use of the EPIC facility of Northwestern University’s NUANCE Center, which has received support from the Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource (NSF ECCS-1542205), the MRSEC program (NSF DMR-1720139) at the Materials Research Center, the International Institute for Nanotechnology (IIN), the Keck Foundation, and the State of Illinois, through the IIN”

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Contents

I. 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-7900F, 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.

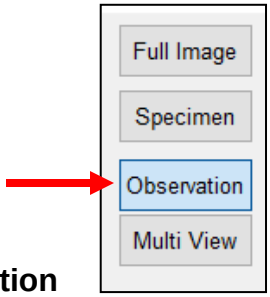
Saving Your Data

During your session, you may store image data to your own folder within the 7900FLV folder on the NUANCE SEM File Server (S:\\). The SEM server is accessible through a computer in the lab. EDS data must first be saved onto the INCA projects folder on the desktop and then transferred onto your folder on the N:\\ drive. You can transfer your data from the SEM server computer to a USB, etc. You should **NEVER** take your data directly from either the SEM or EDS computers.

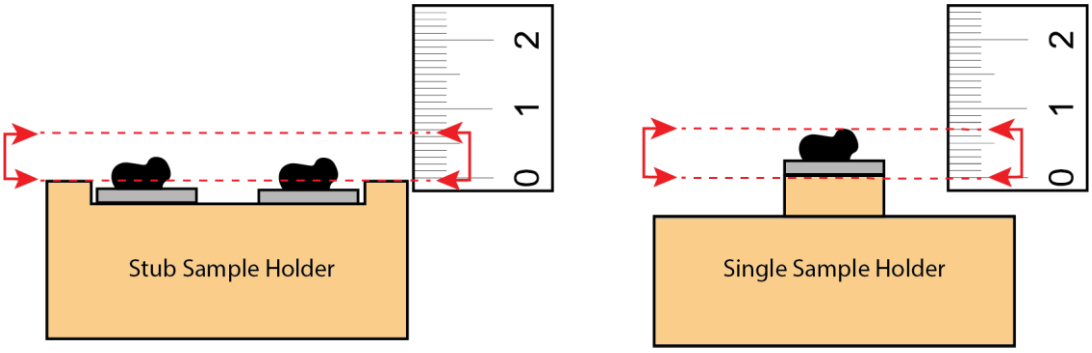
SEM Rules

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!

II. Sample Loading



1. Begin your reservation in NUcore
2. Log into the Guest account on the JEOL software
    a. There is no password
3. On the right-hand side of the main computer screen, select **Observation**
4. Prepare sample holder
    a. Single sample holder
        i. Set your stub in the single puck holder and tighten set screw
        ii. Drop puck with stub attached in the single sample holder
        iii. Use screw on the bottom on the sample holder to adjust height so that it is flush with the top



        iv. **IMPORTANT! Measure any offset (in mm) with ruler from the top of the sample holder to the top of the sample**

    b. Stub sample holder
        i. Place stubs in top of holder
        ii. Tighten set screws on all sides with flat head screw driver
        iii. Place stub holder in sample holder base
        iv. **NOTE**: The stubs should not vary in height than more than about 2-3 mm. ***Do not put in samples at drastically different heights***
        v. **IMPORTANT! Measure any offset (in mm) with ruler from the top of the sample holder to the top of the sample**

c. Puck sample holder

i. Load similarly to single stub holder. Do not remove small screws!

ii. **IMPORTANT! Measure any offset (in mm) with ruler from the top of the sample holder to the top of the sample**

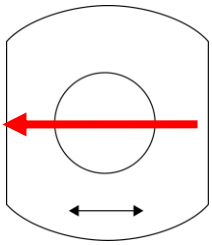
d. **STEM** sample holder (NOTE: Requires separate training)

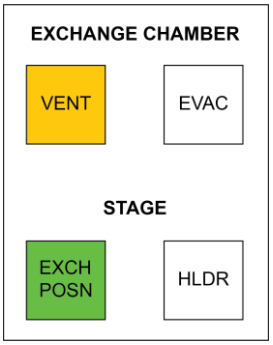
e. **GBSH** Sample holder (NOTE: Requires separate training)

5. Unlatch the latch on exchange chamber door

6. Vent the exchange chamber by holding down on the **Vent** button

7. Swing open the door and slide sample holder on making sure that the arrows are pointing the right direction (see figure) – should slide in parallel to the arrows





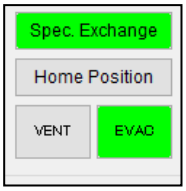
8. **NOTE**: Try to keep the exchange chamber door closed and pumped as much as possible

9. Close the door, latch, and select the **EVAC** button

10. The system will automatically snap a picture for navigation

11. Once the **EVAC** light stops blinking, the exchange chamber is pumped down

12. Ensure the stage is in the **Spec. Exchange** position



a. If it is not, please select this button on the screen until it is solid green

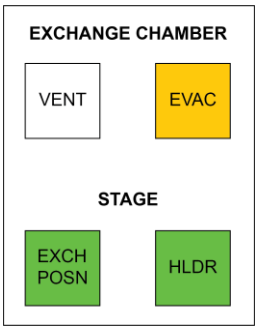
13. Lower the exchange rod

14. Turn rod to the right and slowly and carefully push the rod in all the way

a. **NOTE**: Look for the **HLDR** light to turn on the **STAGE** panel. When this light is on, the sample holder is connected to the stage

b. Do not do this too fast! The stage will not register the holder.

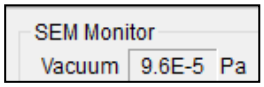
15. Remove exchange rod, turn to the left, and push back to upright position

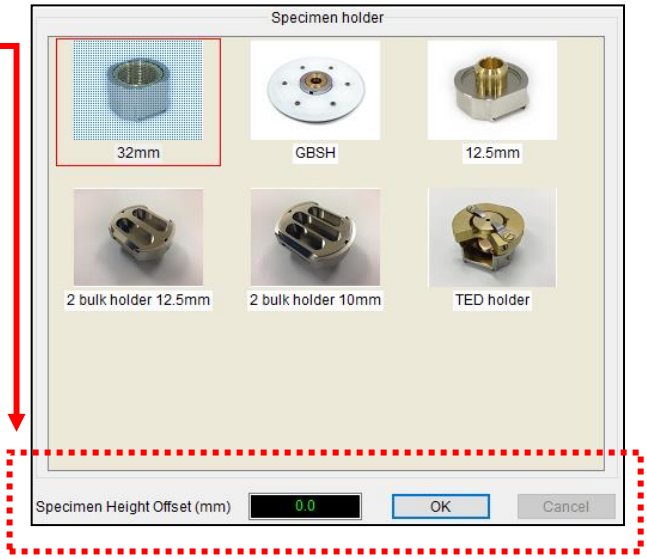


16. When prompted, select the sample holder you are using and indicate any offset

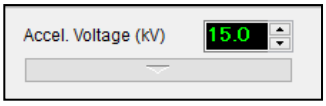
   a. **NOTE**: It is imperative that you select the appropriate offset to avoid crashing into the objective lens (See step 4)

17. Wait for the vacuum to reach at least **2E-4** before continuing

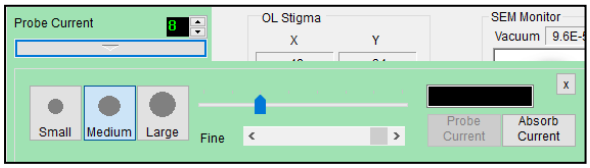




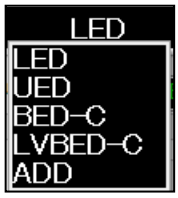
III. Start Up



1. Set your accelerating voltage either at the top of the screen, or the bottom panel
2. Select your probe current
    a. A smaller probe current will provide better resolution, but will result in a noisier image
    b. A large probe current will result in worse resolution, but higher signal/contrast. This will be useful for EDS
    c. Most common probe current is medium (8)

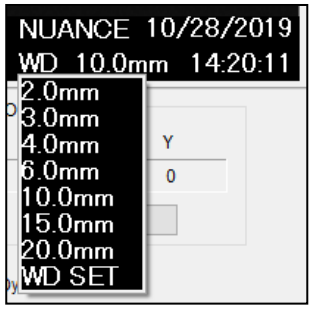


3. Ensure that the LED (Lower Electron Detector) is selected by clicking the detector in the scanning window, or in the menu below
    a. **NOTE**: If using other detecors or modes, please see section on detector options
    b. See detector option on page 10



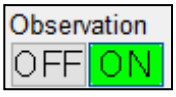
4. Open the Chamber camera window
5. Select the **ZFC button** (if not already selected)

6. Select a WD of **10 mm**



7. A window will appear asking if you want to move to this working distance. Select Ok
    a. **NOTE: If you did not measure the offset in your height, please remove and remeasure**
    b. **NOTE: to stop stage from moving, select any button on the stage control button panel on the desk (except the "c" button**

8. The Z height will change until it matches 10 mm
9. Close the chamber camera (camera does not work when beam is on)
10. Turn **Observation ON** if vacuum down to **4E-4**
    a. If you have a porous sample or a large sample, this may take up to 10 min. Do NOT turn on the beam until the vacuum has recovered.



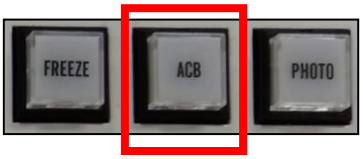
11. Select a **Quick 2** scan speed

   a. Note: Quick 1 is set for auto alignments and should never be changed. Quick 2 is faster than Quick 1

12. Use the Navigator window to center over sample

   a. Right click – Move stage to center

13. Adjust brightness and contrast



   a. Use knobs on knob set
   b. Select ACB button on computer interface
   c. Select ACB button on knob set

14. Decrease magnification all the way using magnification knob on the knob set

15. Roll the track ball or right click on screen to center over a small particle on sample surface

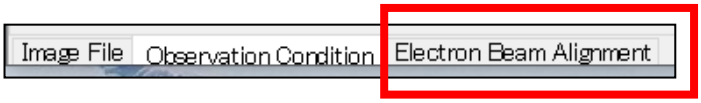
16. Start by focusing using the stage focus control by turning the wheel around the track ball

   a. This adjusts the height of the stage so that the sample is in focus at WD 10 mm

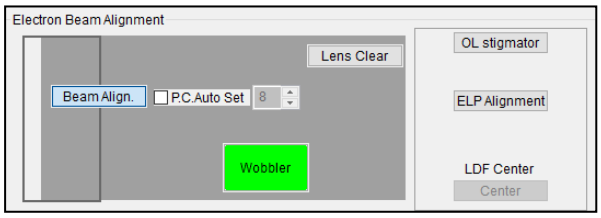
17. Check the Z: height to ensure that it is 10 mm or higher

IV. Alignments

1. Find a particle or some texture on the surface of your sample
    a. Move around your sample by right clicking > move to center on the sample navigation image or in the live scanning window
    b. Stage movements can also be made with the track ball
2. Increase magnification to at least 10,000x
3. Focus the beam with the focus knob on the knob set (no the stage focus)
4. Open the **Electron Beam Alignments** window



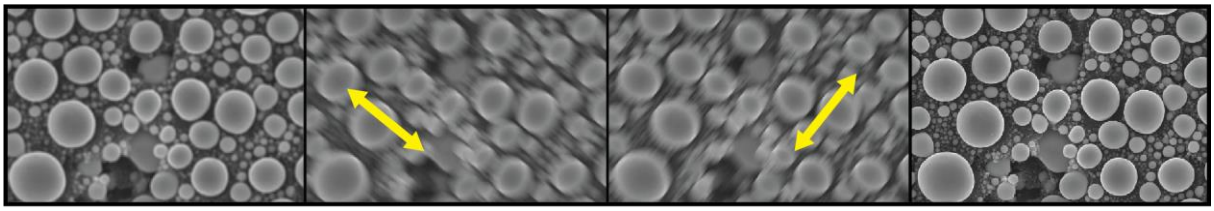
5. Select **Beam Align.** Button and ensure Wobbler is on
    a. Use the X and Y knobs on the knob set to adjust the translation seen in the image



6. Select **OL stigmator** button on computer **OR STIG** button on knob set to exit the wobbler
7. Select the reduced area window by selecting the RDC button on the knob set

8. **Auto Alignments**
    a. In the Setup (S) > Operation Settings > Scan Setting make sure Quick1 is set to Speed 2, Ave. 1. If these settings are not set, auto alignments will not work
    b. Press F12

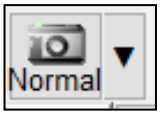
9. **IMPORTANT**! Adjust your STIG
    a. Using the focus knob, focus the beam up and down and look for stretching in two directions at 90 degrees of each other. Then find the middle of the stretching and adjust your STIG knobs (X and Y) one at a time until you see the clearest image



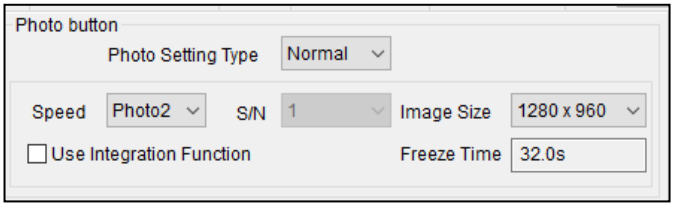
10. Select the **RDC** button to exit reduced area window

V. Image Capture

11. Select the camera button that says “Normal”



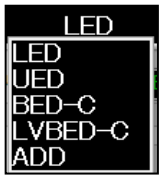
12. To change image capture settings, select Setup (S) > Operation Settings > Scan Setting
13. Change any Photo button option
    a. NOTE: Do not change any other settings in this window!



14. When prompted, save your images to the folder named with your NetID on the EPIC_SEM drive (S:)

VI. Detector Options

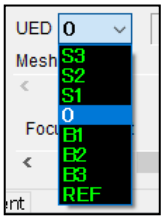
1. LED – Lower Electron Detector



a. The LED can be selected from the detector drop down menu. It is always in the chamber and does not need to be inserted
   i. You must be at or above 6 mm WD to use LED

b. LED Utility
   i. Ideal for displaying topography, surface detail, and shape
   ii. The LED will show less charging than UED

2. UED – Upper Electron Detector

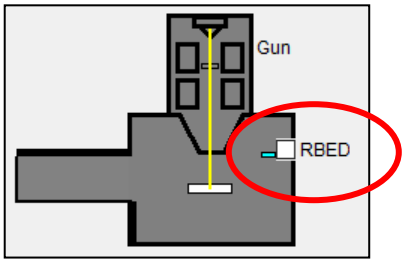


a. The UED can be selected from the detector drop down menu. It is always in the chamber and does not need to be inserted
   i. Must be closer than 6 mm WD

b. UED Utility
   i. Most efficient at short working distances (minimum is 2 mm)
   ii. Can be used for both SE and BSE imaging using energy filter

3. RBED (BED-C) – (Retractable) Backscatter Electron Detector

a. BED-C must be inserted into the chamber at the base of the pole piece
   i. Move to 10 mm WD
   ii. Turn the beam (observation) OFF
   iii. On the column/chamber schematic, check the box next to RBED
   iv. Turn the beam back on

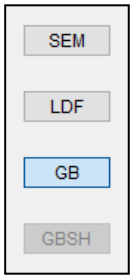


b. BED-C Utility
   i. Ideal for atomic-number contrast and topographic contrast
   ii. Can be used at any working distance (minimum is 2 mm)

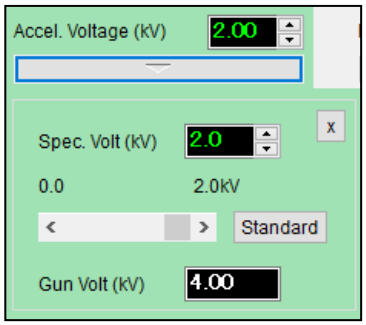
4. **LVBED-C – Low Vacuum Backscatter Electron Detector**
    a. The LVBED is a backscatter detector used in low vacuum operation that is inserted into the tip of the pole piece
    b. This detector can be used in both LV and HV modes
    c. See section on LV operation for instruction

VII. Operation Modes

1. **SEM**
    a. The mode used for imaging with LED, UED, LVBED or BED-C



2. **LDF** - Large Depth of Field Mode
    a. Allows you to see more of sample for navigating.
    b. Can only use LED
3. **GB** – Gentle Beam
    a. Gentle beam applies a negative bias to the stage resulting in a higher landing energy of the beam on your sample. Having a higher beam energy down the column will reduce lens aberrations and increase low kV resolution
        i. Allows super surface sensitive analysis (for imaging)

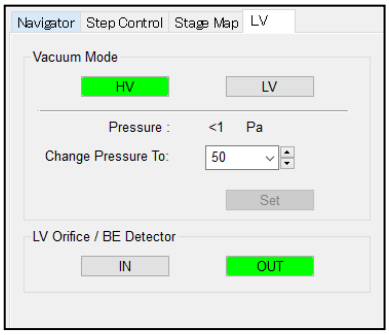


    b. Select GB mode
    c. Select the Accel. Voltage (kV) as landing voltage
    d. Select the Spec. Voltage (deceleration/bias on stage)
        i. The system will set the gun voltage so that it is Accel. Voltage – Spec. Voltage
    e. Example: Desired 2 kV landing voltage with 2 kV deceleration. The system will set Gun Volt to 4 kV so that the landing energy is 2 kV
    f. GB mode can use the LED or UED

4. **GBSH**
    a. **GBSH is used for up to 5 kV beam deceleration on sample. This requires a special sample holder and additional training. Please contact EPIC Staff**

VIII. Low Vacuum Mode

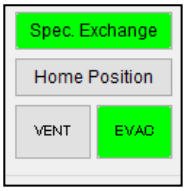
1. Turn **Observation** OFF
2. Select the **LV** menu
3. Set desired chamber pressure (in Pa)
4. Select the **LV** button
    a. Confirm LV mode when window pops up
5. The SEM will lower the stage and insert the LVBED-C orifice
    a. You cannot use any other detector in this mode
6. Set accelerating voltage and probe current
7. Turn **Observation ON**
8. **NOTE**: the LV detector S/N is better at slower scan speeds
9. **Exit LV mode when done!**
    a. To exit LV mode, select HV button under Vacuum Mode
    b. Select to remove LVBED-C
    c. **NOTE**: You can leave in the LVBED-C detector to do HV imaging.

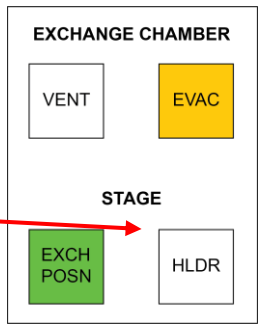


IX. EDS with AZtec

1. Select an appropriate accelerating voltage (usually between 15-30 kV)
2. Pause the chamber scope
3. Increase probe current (10 -14)
4. Make sure sample is in focus at 10 mm working distance. This is the analytical working distance for this microscope

X. Shut Down and Sample Removal





1. Turn **Observation OFF**
2. Return to HV mode if using LV mode
3. Move the stage to the **Spec. Exchange** Position
4. Lower the exchange rod
5. Rotate rod to the right and glide into the chamber
6. Once rod is all the way in, retract it all the way and lock into position by rotating back to the left
7. Ensure the **HLDR** light on the panel is off
8. Raise the exchange rod to resting position
9. Unlatch the exchange chamber door
10. Hold down on the VENT button
11. Remove the sample holder from the exchange chamber
12. Close the door and hold down on the **EVAC** button
13. The **EXCH POSN** light should be green and the **EVAC** light should be solid before you leave
14. Do not unscrew any set screws too much! They can get lost very easily!
15. Exit the SEM software
    a. Got to File (F) > Exit
16. **END RESEVATION in the NUcore system**