

LEO 1525 FEG SEM

Instructions

For additional assistance, please contact the facility manager.

Please contact under emergency:

SEM manager: Mr. Ben Myers, 1-3439 (O), 312-593-8298 (cell)

b-myers3@northwestern.edu

EPIC manager: Dr. Jian-Guo Zheng, 1-7807 (O), 847-675-7387(h),

j-zheng3@northwestern.edu

EPIC director: Prof. Vinayak P Dravid, 7-1363 (O), 847-486-1705 (h),

v-dravid@northwestern.edu

The LEO SEM is currently not connected to the EPIC login system. Please reserve time on the sign up sheet on the door and log your usage on the log sheet.

Note: It is imperative that gloves be worn during all sample exchange procedures. If you cannot find any gloves, please ask!

You are asked to make a copy of your data on your own disk IMMEDIATELY after your session is finished. You may save your data on a 100MB Zip disk or transfer your data by FTP. The data may be deleted at any time without notice. EPIC is not responsible for any data loss.

System Startup

1. There are three types of sample holders this SEM. A single stub holder, an eight stub holder and a third holder that allows for 45° and 90° stub orientation.
 - a) Load a stub by sliding it into a hole in the desired sample holder and tightening the screw.
 - b) Note: On the eight stub sample holder it is possible for the stubs to extend below the lower surface of the holder which may cause the sample holder to get hung up in the SEM. Please check to make sure all stubs are flush with the lower surface or higher.
2. Press the green **On** button on the SEM.
3. Turn on the PC and wait for the system to boot.
4. Once the system boots, you will be prompted for a network password – press **Cancel**.
5. Double click on the **LEO 1525** icon on the desktop to start up the SEM and software.
6. Enter your Login and Password information when prompted.
7. Turn on the Chamber Scope.
8. Under the **Stage** menu, select **Stage Initialise**. When initialization is complete, the stage should be at the following coordinates - X:50.0, Y:50.0, Z:0.516, T:0.0, R:0.0. To view stage location coordinates select Vacuum Status under the Vacuum menu and click on the Stage tab.

Sample Loading (Airlock)

1. Under the **Stage** menu, select **Store/Recall**. On the Store/Recall screen double-click on **Exchange Airlock**. The stage should move to the following coordinates - X:48.124, Y:0.376, Z:4.808, T:0.0, R:333.4.
2. Shut the valve between the column and the chamber by selecting **Goto Panel** under the **Tools** menu and double-click on **Airlock**. Then select **Close Column Chamber Valve** (the valve should already be closed if you are loading your first sample).
3. Verify that the following lights on the Airlock panel are on: Wait, Rod Status and Gate Closed. If the Rod Status light is not on, pull the sample loading rod back fully and the light should come on.
4. Vent the Airlock by pressing the **Purge** button.
5. When the Airlock vents, open the door and load the sample holder onto one of the two Teflon stages (two sample holders may be loaded into the Airlock at one time).
6. Close the Airlock door and press the **Purge** button again to deactivate venting.
7. While holding the Airlock door firmly shut, press the **Pump** button.
8. When the Proceed light turns on, press the **Open** button to open the door between the Airlock and the chamber.
9. Load the sample by first screwing the rod into the sample holder, then sliding the rod into the chamber and place the sample holder on the specimen stage. Then unscrew the rod from the sample holder and slide it back until the Rod Status light comes on.
10. Press the **Open** button again followed by the **Close** button to close the door.
11. Once the door closes and the gate close light is on, press the **Close** button followed by the **Pump** button.
12. Under the **Stage** menu, select **Store/Recall**. On the Store/Recall screen double-click on **Center**. The stage should move to the following coordinates - X:57.377, Y:51.251, Z:7.185, T:0.0, R:332.8.
13. Select **Goto Panel** under the **Tools** menu and double-click on **Airlock**. Then select **Open Column Chamber Valve**. (Note: The column chamber valve will not open when the airlock still has good vacuum – i.e. when the green proceed light is on. To open the airlock valve you will first need to turn the purge button on and off quickly, the wait light should come on and the valve should open)
14. Select Vacuum Status under the Vacuum menu and click on the Gun tab. Set the EHT level.
15. Once the system reaches Vac ready (a green check next to “Vac” on the status bar) click on **EHT** in the status bar and select **EHT On**.

Sample Loading (Chamber Vent)

1. Under the **Stage** menu, select **Store/Recall**. On the Store/Recall screen double-click on **Center**. The stage should move to the following coordinates - X:57.377, Y:51.251, Z:7.185, T:0.0, R:332.8.
2. To load a sample holder that is too large for the Airlock, shut the valve between the column and chamber by selecting **Goto Panel** under the **Tools** menu and double-click on **Airlock**. Then select **Close Column Chamber Valve**.

3. Select Vacuum Status under the Vacuum menu and click on the Vacuum tab. Click on **Vent**.
4. Open the black valve on the Nitrogen tank to vent the chamber.
5. When the chamber vents, close the Nitrogen valve.
6. Slowly open the chamber door by pulling on the black handle.
7. Load the sample holder by sliding it onto the dovetail sample holder.
8. Close the chamber door, hold it shut and click on **Pump** from the Vacuum menu.
9. Select **Goto Panel** under the **Tools** menu and double-click on **Airlock**. Then select **Open Column Chamber Valve** (Note: the sample chamber will have to pump down to base pressure before this is available). Once the chamber reaches vacuum, you will hear the valve open.
10. Select Vacuum Status under the Vacuum menu and click on the Gun tab. Set the EHT level.
11. Once the system reaches Vac ready (a green check next to “Vac” on the status bar) click on **EHT** in the status bar and select **EHT On**.

Shut Down and Sample Removal (Airlock)

1. On the status bar, click on **EHT** and select **EHT Off**.
2. Shut the valve between the column and the chamber by selecting **Goto Panel** under the **Tools** menu and double-click on **Airlock**. Then select **Close Column Chamber Valve**.
3. Turn on the chamberscope if necessary.
4. Under the **Stage** menu, select **Store/Recall**. On the Store/Recall screen double-click on **Exchange Airlock**. The stage should move to the following coordinates - X:48.124, Y:0.376, Z:4.808, T:0.0, R:333.4.
5. Pump down the Airlock by pressing **Pump** on the Airlock panel.
6. When the Proceed light turns on, press the **Open** button to open the door between the Airlock and the chamber.
7. Extend the rod into the chamber and screw it into the sample holder.
8. Pull the rod back and slide sample holder onto the Teflon stage.
9. Unscrew the rod from the sample holder and pull it back until the rod status light comes on.
10. Press the **Open** button followed by the **Close** button on the Airlock panel.
11. When the Gate Closed light comes on, press **Close** again then press **Pump**.
12. Press the **Purge** button.
13. When the Airlock vents, open the door and remove the sample holder.
14. Close the Airlock door and while holding it firmly shut, press the **Purge** button followed by the **Pump** button.
15. When the Proceed light turns on, press **Pump** again.
16. Close the software interface and shutdown the PC.
17. Once the PC is shut down, press the **Standby** button on the SEM.
18. **Log your time on the LEO 1525 Log Sheet.**

Shut Down and Sample Removal (Chamber Vent)

1. See steps 1-8 from Sample Loading (Chamber Vent)
2. Wait for the system to pump down to 1e-5 Torr before shutting down.

3. Close the software interface and shutdown the PC.
4. Once the PC is shut down, press the **Standby** button on the SEM.
5. **Log your time on the LEO 1525 Log Sheet.**

General Operation and Alignment

1. Using the Z-axis joystick control, raise the sample height to the desired level. (A shorter working distance will provide better resolution, but poor depth of focus)
2. Align the sample visually with the objective lens horizontally using the Y-axis joystick control.
3. Move the sample to the far side of the objective lens using the X-axis joystick control.
4. Switch to the desired detector (Inlens, SE2 or RBSD). For short working distances, use the Inlens (upper) detector and for longer working distances, use the SE (lower) detector. Use the RBSD detector for compositional contrast.
5. Click on the Brightness/Contrast button and adjust until you can see some signal (noise) on the screen.
6. Click on the Magnification/Focus button and decrease magnification all the way.
7. Locate your sample by moving the stage in the $-X$ direction (joystick to the left).
8. After you find the sample, focus on the surface, increase the magnification to at least 20,000X and find a suitable feature for alignment.
9. Select Vacuum Status under the Vacuum menu and click on the Apertures tab.
10. Click on Gun Align and then click on Emission.
11. Adjust the gun alignment to line up the emission image (bright spot) with the cross hairs. If the crosshairs are not present, you can turn them by selecting User Preferences under the tools menu and clicking on SEM Conditions.
12. After gun alignment, click on Normal, then click on Mag/Focus and focus the image.
13. Click on Aperture Align, turn on the focus wobble and adjust the wobble amplitude (30% is a good starting point).
14. Align the aperture to eliminate any translation (wobbling) of the image.
15. Turn off the focus wobble, click on Mag/Focus and focus the image.
16. Correct for astigmatism by clicking on Stigmatism and adjusting the stigmators for the sharpest image. When the astigmatism is corrected, you should not see any stretching of the image when out of focus.
17. If you change EHT level, aperture size or working distance you will probably need to realign the SEM.

Image Capture

1. To capture an image, first find the are of interest using the X/Y joystick controls.
2. Focus the image at a higher magnification than you wish to capture.
3. Zoom out to the desired magnification.
4. In the Vacuum Status menu, select the Scanning tab.
5. Select the image resolution (1024x768 is a reasonable size).
6. Select the type of noise reduction – pixel averaging or frame integration.
7. For frame integration, select the dwell time.

8. Select the scan speed.
9. For frame integration, the image will automatically freeze when the integration is complete.
10. For pixel averaging, select Freeze = End Frame and press the Freeze button. The image will freeze when the current frame is finished scanning.
11. Image annotation options can be found by selection Annotation under the Edit menu.
12. To save the image select Save Image under the File menu, select the appropriate directory, enter the file name and save.

Other Important Information

1. When inserting and retracting the backscatter detector (RBSD), go very slowly and be careful not to dump the vacuum. When retracting, make sure you pull the detector all the way out until the pin drops.
2. Always remember to shut the valve to the nitrogen tank after using the Chamber Vent method for sampling loading.
3. Always record the system information (Ext I, vacuum, etc.) on the logsheet when the EHT is on. These values will be very different when the voltage is off.
4. Never install any software on the LEO SEM computer.