

LEO 1525 FEG SEM

Standard Operating Procedures

Mar. 6, 2012

For additional assistance, please contact the facility manager.

Please contact in case of emergency:

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LEO reservations are made using the EPIC FOM online reservation system. Please follow all EPIC facility rules for using this system. There is a hardware control system on the LEO, so the monitor of the LEO computer will not turn on unless you are logged in. Tampering with or disabling the hardware control may result in revocation of your EPIC privileges.

Note About Saving Your Data: During your session, you may store images in your own folder within the LEO folder on the SEM Server (U:\). The SEM server is accessible through two computers in the specimen prep lab that are labeled "SEM server". You can transfer your data from the SEM server computer to your USB etc. You should never take your data directly from the SEM computer.

FTP Address for connecting to SemServer Network from a personal computer:

129.105.37.228

Username: semuser

Password: \$emu\$er

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Before getting started:

- 1. It is imperative that gloves be worn during all sample exchange procedures. If you cannot find any gloves, please ask!**
- 2. NEVER install any software onto the SEM's PC.**

I. Sample Loading – AIRLOCK

1. Sign into the FOM system.
2. Put on gloves and load sample/s onto a sample holder.
 - a. There are three types for the LEO. A single stub holder, an eight stub holder and a third holder that allows for 45° and 90° stub orientation.

CAUTION: With the round eight-stub sample holder it is possible for the stubs to extend below the lower surface of the holder which will cause the sample holder to get stuck in the SEM. Please make sure no stubs are protruding from the bottom of the holder before insertion into the SEM.
3. On the PC, go to the **Stage** menu and select **Store/Recall**. Double-click on **Exchange** to make sure the stage is in the exchange position. Leave this window open for Step 18.
4. **Tools** menu > **Goto Panel**, double-click **Airlock**. Make sure that **Close Column Chamber Valve** is selected. (This valve between the column and chamber should be closed already, if not, close it.) Leave this window open for Section III.
5. Ensure the following indicator lights on the Airlock panel are on: **Rod Status** and **Gate Closed**.

Note: If the Rod Status light is **not** on, ensure that the exchange rod has been pulled all the way out and the set screw is on tight.
6. Ensure all four buttons on the Airlock panel are deactivated.
7. Activate the **Purge** button. (When the exchange chamber has reached atmosphere, the door should open easily.)
8. Load the sample holder onto the Teflon stage that is in front of the exchange rod.

Note: Make sure that the sample holder is facing the right way.
Note: Do not screw rod into sample holder until step 13.
9. Close the Airlock door and deactivate the **Purge** button.
10. While holding the Airlock door firmly shut, activate the **Pump** button.
11. When the **Proceed** light turns on, activate the **Open** button.
12. If the chamber camera is not shown on the PC screen, click the **Chamber Scope/Detector Control** button to view. 
13. Insert the sample.
 - a. Loosen the set screw on the rod.
 - b. Screw the rod all the way into the sample holder.
 - c. Slide the rod into the chamber and push the sample holder all the way onto the stage. (Check the camera view of the chamber on the monitor to ensure the sample is all the way on.)
 - d. Unscrew the rod from the sample holder and slide it all the way back out until the Rod Status light comes on.
 - e. Tighten the set screw.
14. Deactivate the **Open** button.
15. Activate the **Close** button.
16. When **Gate Closed** light comes ON, deactivate **Close** button.
17. Deactivate **Pump** button.

Note: If the gate closed light does not come on, activate **Pump**, then **Close** again.
18. **Stage** menu > **Store/Recall**. Double-click on **Center**. Close this window.
19. Using the Z-axis joystick control, raise the sample height to the desired level.

CAUTION: At this point, make sure to still keep the sample several millimeters below the pole piece.
20. Align the sample visually with the objective lens horizontally using the Y-axis joystick control. (Left Joystick, Up and Down)
21. Hold down the Left Joystick all the way to the right. (This will move the sample to the far side of the specimen chamber and will make it easier to find your sample once the beam has been turned on.)
22. Go to **Section III**.

II. Sample Loading – CHAMBER VENT (Only for samples too large for the airlock)

1. Sign into the FOM system.
2. Put on gloves and load sample/s onto a sample holder.
 - a. There are three types for the LEO. A single stub holder, an eight stub holder and a third holder that allows for 45° and 90° stub orientation.

CAUTION: With the round eight-stub sample holder it is possible for the stubs to extend below the lower surface of the holder which will cause the sample holder to get stuck in the SEM. Please make sure no stubs are protruding from the bottom of the holder before insertion into the SEM.
3. On the PC, go to the **Stage** menu and select **Store/Recall**. Double-click on **Center**. Close this window.
4. **Tools** menu > **Goto Panel** and double-click on **Airlock**. Make sure that **Close Column Chamber Valve** is selected. (This valve between the column and chamber should be closed already, if not, close it.) Keep this window open for Section III.
5. **Vacuum** menu > **Vacuum Status**, click on the **Vacuum** tab. Click on **Vent**. (Leave this window open for step 10.)
6. To vent the chamber:
 - a. Open the valve on top of the Nitrogen tank.
 - b. Open the small green valve on the regulator attached to the Nitrogen tank.
7. When the chamber vents, **CLOSE both Nitrogen valves**.
8. Slowly open the chamber door by pulling on the black handle.
9. Load the sample holder by sliding it fully onto the dovetail on the stage.
10. Close the chamber door, hold it shut and on the PC click **Pump** from the previously opened **Vacuum** menu.

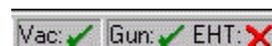
Note: It usually takes at least 20 minutes for the chamber to pump down to proper vacuum.
11. Using the Z-axis joystick control, raise the sample height to the desired level.

CAUTION: At this point, make sure to still keep the sample several millimeters below the pole piece.
12. Align the sample visually with the objective lens horizontally using the Y-axis joystick control.
13. Move the sample to the far side of the specimen chamber by using the X-axis joystick control. (This will make it easier to find your sample once the beam has been turned on.)

III. Start Up

1. The **Column Chamber Valve** (Isolation Valve) will not open when the airlock still has good vacuum (i.e. when the green **Proceed** light is on). To remedy, simply turn the **Purge** button on and off quickly. The **Wait** light should come on and the Column Chamber Valve should now be unlocked.
2. If Airlock menu is not already open, go to **Tools > Goto Panel**. Double-click on **Airlock**. Select **Open Column Chamber Valve**. Close window.
3. **Vacuum > Vacuum Status**. Record the **System Vacuum** and **Gun Vacuum** readings on the LEO data sheet. Keep window open.
4. In the same **Vacuum Status** window, select the **Gun** tab. Record the **Extractor I** reading on the LEO data sheet.

CAUTION: The “leave gun on at shutdown” option should always be checked, and **NEVER** touch the **Filament I Target** or the **Extractor V Target** settings.
5. At the bottom of that tab, you may set the EHT (Accelerating Voltage) level with the slider at the very bottom. (You may also type in the desired number by double clicking on the **EHT Target** text window.) Keep window open.
6. Once the system reaches adequate vacuum, a green check will appear next to “Vac” on the status bar in the lower right corner of the screen. To



turn on the accelerating voltage, left click on **EHT** in the status bar in the lower right of the screen and select **EHT On**.

7. Once the EHT has ramped up, record the new EHT number from the Gun tab of the Vacuum Status window on the LEO data sheet.
8. Choose the desired secondary electron detector (Inlens or SE2) by clicking the Detector Toggle button. 
 - a. **Inlens** (upper) detector for high resolution (needs short working distances).
 - b. **SE** (lower) detector for large samples, lower mags and lots of Depth of Field. Has lower resolution than the Upper detector.
 - c. **RBSD** detector for compositional contrast. See **Section V** in this SOP for instructions.
9. Click on the **Magnification/Focus** button and decrease magnification **all** the way (Hold down left mouse button and drag mouse to the left). 
10. Click on the **Brightness/Contrast** button and adjust with the mouse until you can see some signal (noise) on the screen. 
11. Locate your sample by moving the stage in the $-X$ direction (Left joystick to the left).
12. After you find the sample, focus on the surface at about 2,000X. *Once you have a large surface feature in focus it is OK to change the working distance.*
13. **To set a shorter working distance safely:**
 - a. Double-click **WD** below the data bar and enter in a shorter working distance (i.e. 5mm).
 - b. Raise the stage up using the Z controller until the image comes into focus.
CAUTION: Be **VERY CAREFUL** not to crash the sample into the lens. There is **NO** software control over this stage. **YOU** are the only thing that will stop the stage before it hits the lens.
 - c. Continue setting a shorter working distance a little bit at a time and bringing the sample into focus with the Z joystick until you are satisfied with the distance (usually around 4-5mm is good for Inlens (upper) mode).

Note: For RBSD and SE modes the WD should be at least 10mm.

IV. Alignment

1. Find a suitable feature for alignment and increase the magnification to a higher magnification than what you want to take your images at.
Note: Alignments should always be done at higher magnification than what you want to take your images at.
2. **Vacuum > Vacuum Status**, click on the **Apertures** tab.
3. Click on **Gun Align**, then click **Emission**.
4. Adjust the gun alignment with the sliders to line up the emission image (bright spot) with the crosshairs.
 - a. If the crosshairs are not present, you can turn them on by going to **Tools > User Preferences > SEM Conditions**.
5. After gun alignment, click **Normal**, then **Mag/Focus** and focus the image.
6. Click **Aperture Align** and turn on the focus wobble.
 - a. Use the slider bar to adjust the wobble amplitude (30% is a good starting point).
7. Using the mouse, align the aperture to eliminate any translation (wobbling) of the image.
8. Turn off the focus wobble.
9. Click on **Stigmatism**. Focus the image (right mouse button) and then adjust the stigmators (left mouse button) for the sharpest image.
 - a. When the astigmatism is corrected, the image should not stretch when focus is changed.
NOTE: If you change EHT level, aperture size or working distance you will probably need to realign the SEM.

V. Robinson Backscatter Detector

1. Move the sample down to the bottom of the specimen chamber. (Right joystick down)
DANGER: The sample needs to be well out of the way when the detector is inserted into the chamber during the next step. If it is not, horrible things will happen to the detector, and by extension - **YOU**.
2. Carefully insert the detector into chamber.
 - a. Loosen top set screw.
 - b. Lift up on the skinny bar running underneath the detector.
 - c. Carefully guide unit into chamber.
CAUTION: If the detector is bumped or handled too roughly, the vacuum in the chamber can be broken.
3. Select the RBSD detector by clicking on **Detection > Detectors > RBSD**.
4. Voltage should be at least 10 or above.
5. To get an image, it helps to set the brightness at 75% and then increase the contrast.
6. When finished, retract the detector.
CAUTION: Make sure the detector is retracted all the way until the rod drops and locks into place.
7. Tighten set screw.

VI. Image Capture

1. Focus and adjust stigmators at a higher magnification than you wish to capture. 
2. Zoom out to the desired magnification.
3. To change the image resolution.
 - a. **Vacuum > Vacuum Status**, select the **Scanning** tab. (1024x768 is a reasonable size.)
4. To take a slow scan (for high res images)
 - a. Click on scan speed 4.
 - b. As the image scans, click the Pause button. This will freeze the frame after the current scan finishes. 
5. For Frame Integration (good for charging samples)
 - a. Click on scan speed 5.
 - b. Image will freeze once integration is complete.
6. Image annotation options can be added through **Edit > Annotation**.
7. To remove the data bar. **View > Hide Data Zone**.
8. To save the image. **File > Save Image**. **ALWAYS** save images to the **U:** drive.

VII. Shut Down and Sample Removal - AIRLOCK

1. On the status bar in the lower-right of the screen, left-click on **EHT** and select **EHT Off**.
2. Go to **Tools** menu, select **Goto Panel** and double-click on **Airlock**. Select **Close Column Chamber Valve** to isolate the column from the chamber.
3. Activate **Pump** on the Airlock panel.
4. Go to **Stage** menu, select **Store/Recall** and double-click **Exchange**.
5. When the **Proceed** light turns on, activate the **Open** button to open the door between the Airlock and the chamber.
6. Extend the rod into the chamber and screw it into the sample holder.
7. Pull the rod back and slide sample holder onto the Teflon stage.
8. Unscrew the rod from the sample holder and pull it back until the rod status light comes on.
9. Deactivate the **Open** button and activate the **Close** button on the Airlock panel.
10. When the **Gate Closed** light comes on, deactivate the **Close** button.
11. Deactivate the **Pump** button.
12. Activate the **Purge** button.

13. When the Airlock vents, open the door and remove the sample holder.
14. Deactivate the **Purge** button.
15. Close the Airlock door and while holding it firmly shut, activate the **Pump** button.
16. When the **Proceed** light turns on, deactivate the **Pump**.
Note: The Airlock should be under vacuum, but **do not** leave the pump on when you leave the microscope.
17. Log your time out the LEO data sheet.
18. Log out of the FOM system.

VIII. Shut Down and Sample Removal – CHAMBER VENT

1. On the status bar, click on **EHT** and select **EHT Off**.
2. See steps 4-11 from **Section II. Sample Loading - Chamber Vent**
3. Wait for the system to pump down to 1e-5 Torr before logging out.
4. Log out of the FOM system.

IX. Putting the System into Standby Mode (If the computer freezes)

1. Close the software interface and shutdown the PC.
2. Press the **Standby** button on the SEM.
3. Bring the SEM out of standby mode:
 - a. Load your sample using step one of the *Sample Loading* instructions
 - b. Press the green **On** button on the SEM.
 - c. Turn on the PC and wait for the system to boot.
 - d. Once the system boots, you will be prompted for a network password – the username and password are both “**semuser**”
 - e. Double-click on the **LEO 1525** icon on the desktop to start up the SEM and software.
 - f. Enter your Login and Password information when prompted.
 - g. Turn on the Chamber Scope.
 - h. Go to **Stage** menu, select **Stage Initialize**.