

**FEI XL-30 SIRION
SCANNING ELECTRON MICROSCOPE**

Instruction Manual
V02.08

- UCSB Nanofabrication Facility -

1. Preparing your sample.

- a. If the sample that you want to analyze with the Sirion SEM is non-conductive, there are basically two options available to you:
- i. **EASY:** The standard procedure requires that the sample surface be coated with a thin, $\sim 100\text{\AA}$ metallic film (normally gold). There is an easy-to-use, bench-top Au-Pd sputter coater located just outside the Nanofab Sirion SEM room in Bay 1 provided for just this purpose. Note however that you should use a Cu-Be clip to secure your sample to the mount when loading – this will provide a direct connection between the coated surface of your sample to electrical ground.
 - ii. **DIFFICULT:** The XL-30 Sirion machine has the ability to perform high-resolution low voltage ($\leq 1\text{kV}$) imaging. Apparently!, there does exist a voltage (on the order of a few hundred eV or so) below which the number of electrons leaving the surface (backscattered, secondary, etc from multiple scattering events) can equal the number of electrons coming in from the incident beam (a highly tilted sample will also certainly help in attaining this balance point). This phenomenon results in internal charge neutralization and means that a non-conductive substrate can be imaged successfully without needing surface metallization.
- b. There are now many different Al-based sample mounts available for use in the Nanofab Sirion SEM. They all utilize a standard 1/8" pin in the base that slides, via a simple press fit, into any of the 12 available pinholes on the multi-mount holder that is permanently installed on the Sirion stage. They are:
- Circular 1" diameter, 0-degree top-view mount;
 - Circular 2.5" diameter, 0-degree top-view mount;
 - Rectangular 1" x 0.5", 90-degree cross-section mount;
 - Rectangular 1" x 0.5", 20-degree tilted mount;
 - Rectangular 1" x 0.5", 45-degree tilted mount;
 - Rectangular 1" x 0.5", 70-degree tilted mount.

When handling any of the mounts, please use gloves! Choose the holder you wish to use and either clip your sample in place using the Cu-Be clips present on the active surface of each mount (best method) or use the supplied double-sided 'conducting' Cu tape to stick it to the mount (charging artifacts may be observed however when using the tape—especially if your substrate is non-conductive – so be forewarned!).

Be sure to secure your samples on the tilted mounts so that the top edge of the sample lies no higher than the top edge of the mount. It is recommended that you use pinhole 15 (at the cut edge) to secure the tilted mounts to the multi-mount holder – if your sample is large, then part of the mounted sample can lie below the base of the mount and not hit the surface of the multi-mount holder.

2. Loading your sample into the SEM chamber.

- a. Log into Windows on the SEM PC using the "generaluser" account and password (if needed).
- b. Click on the XL-Microscope Control icon located on the desktop, and enter your group's FEI system username and password to open up the Main Window of the control program, Fig. 1. This window displays status and control features for the Sirion SEM (image window, menu bar, tool bar, data bar, and control area).

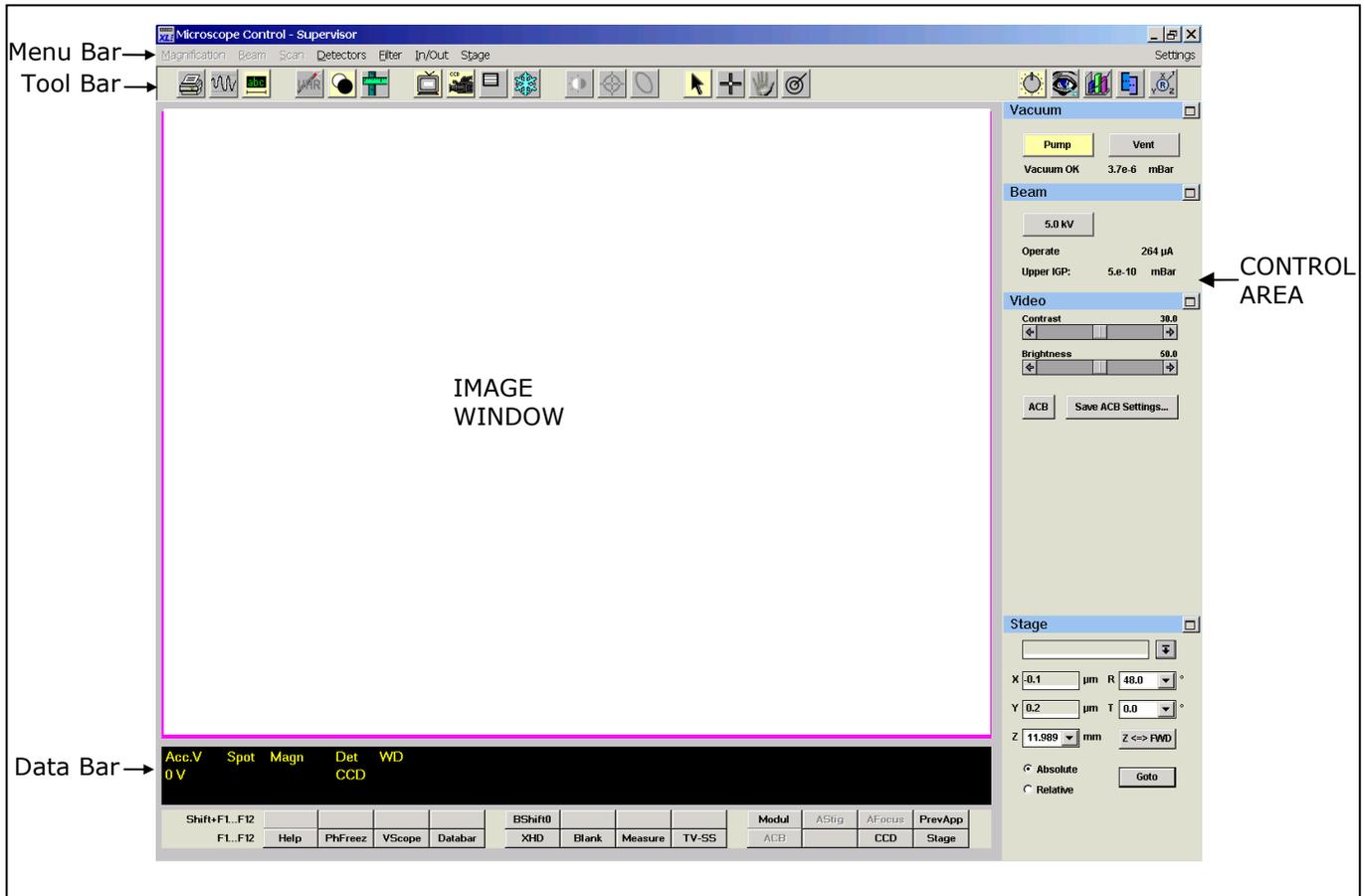


FIGURE 1: The initial Microscope Control window

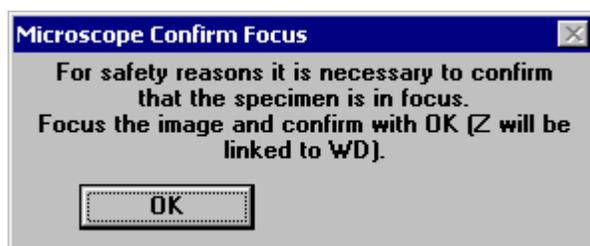
- c. If not already done, activate the CCD detector to image the interior of the sample chamber in the IMAGE WINDOW (click the CCD On/Off icon in the Tool Bar, , which will turn yellow when the CCD image is on).
- d. Click on the **Vent** button of the **Vacuum** control group located in the CONTROL AREA (Fig. 1) of the main window to bring the sample chamber up to atmosphere (button goes yellow).
- e. When the chamber is vented, slowly slide open the chamber door while watching the CCD image in the IMAGE WINDOW to ensure that the stage (with multi-mount holder) will move out freely. At this point, make sure that the stage positioning is set to $(x,y) = (0,0)$, tilt, $T = 0$ and that

rotation, $R = 48^\circ$ (refer to the **Stage** control group in the CONTROL AREA). This ensures that the stage is centered and that the cut edge of the multi-mount holder is located directly next to the secondary electron detector (SED).

- f. Carefully press the pin on the sample mount into the appropriate pinhole on the multi-mount holder until the base of the mount is sitting flush with the surface of the holder.
Utilize the metallic 'Eucentric Height Adjuster' to ensure that your sample is indeed a few mm below the 5 mm indicator on the adjuster for safe loading of your sample.
- g. As a further check (you can never have enough of these!) verify, using the CCD image, that the mounted holder will not hit the final lens cone as you slide the chamber door closed
- h. Holding the door closed, click on the **Pump** button of the **Vacuum** control group located in the CONTROL AREA of the main window (button goes yellow). Also, make sure that the "Sample Touch Alarm" cable is attached to the BNC connector on the door (cable is labeled 'Stage Door')
- i. Wait for the pressure readout below the **Vent** button to fall below $3.0e-5$ mBar before proceeding. This should take no longer than 5 minutes.

3. Start your imaging session.

- a. It is necessary to first ensure that all column alignments get loaded into the software correctly after logging into Microscope Control. To do this, make sure that the **HT** button on the SYSTEM CONTROL PANEL is OFF (the button is located in front of the SEM chamber on the console) and click on the **5 kV** button of the **Beam** control group. This will generate a "HT Off" error window on the screen. Clicking OK will reset the software and/or alignment bug that sometimes appears and ensure that your imaging session will work correctly.
- b. Now turn the **HT** button ON to activate the high voltage to the filament (the button will be lit when active) and then click on the **5 kV** button (the button will turn yellow) which opens the gate valve (isolating the column from the chamber) and sends the electron beam down to your sample.
- c. A "Microscope Confirm Focus" window will appear on the screen.



Do not click on OK! Drag it to the bottom of the main window (away from the IMAGE WINDOW) for now.

- d. Initialize the imaging conditions:
- i. switch the imaging detector to the standard secondary electron detector (click the yellowed CCD On/Off icon, , in the Tool Bar to turn it off and switch to the SED image),
 - ii. unblank the beam by clicking on the yellowed Blank On/Off icon, , in the Tool Bar,
 - iii. select the desired beam conditions (see procedure **4.b.** below), and set magnification to the lowest possible value (see procedure **4.a.** below).
- e. Correct the (rough) focus of the image (see procedure **4.c.** below).
- f. Click on the Track Mode icon, , on the TOOL BAR which allows continuous stage movement with variable speed in any direction. A green circular overlay appears on the IMAGE WINDOW. Click outside the outer green circle for fastest stage motion. To slow the stage speed, move the cursor inside the outer circle to the inner circle. The maximum speed at which the stage moves in Track Mode scales inversely with magnification...low mag implies higher speeds.
- g. Move the stage around to first find your sample. Once you have done that, then find the highest point on the sample mount (i.e., closest to the final lens cone). [Note that if you are using a Cu-Be clip to hold your sample on the flat (0-degree) mounts, look for the scratches on the clip which indicate the high point.] Zoom in and focus till you get a focused image at > 2000X magnification (use the + key on the keyboard). Drag the "Microscope Confirm Focus" window back into the IMAGE WINDOW and click OK. The red NC (Not Calibrated) next to the Z readout in the **Stage** control group will disappear and the stage Z position will now be linked to the Free Working Distance of the microscope (WD value on the bottom of the IMAGE WINDOW). This Z readout now represents the distance between the highest point of your sample holder and the bottom of the final lens cone...a very important thing to know if you don't want to drive your sample into the cone!
- h. In order to obtain good high resolution images (especially for UHR mode), WD values should be reduced to 5 mm.

If using a flat mount, then first decrease the (linked) Z value to 10mm by clicking on the Z readout box of the **Stage** control group and selecting 10mm from the pull-down menu, and hitting return. Refocus the image and click on the **Z<->FWD** button in the **Stage** control group. The "Microscope Confirm Focus" window will reappear. Clicking on OK updates the Z value to the new WD (the smaller the value of WD, the more accurate the linking will be!). If using a tilted mount, then your highest point will already be around the 10mm range so you can ignore the steps above!

Now move the highest point to 5 mm separation by clicking on the Z readout pull-down menu, selecting 5 mm and hitting return.

4. The imaging session procedures

Below are details of all the basic procedures you will need to perform during a standard imaging session in order to collect good SEM images. You should refer to these procedures to assist you with your session.

a. Magnification changes:

- i. Use the + or – key on the keyboard to increase and decrease magnification, OR,
- ii. use the magnification knob on the Manual User Interface panel below the monitor to change the magnification to preset values, OR,
- iii. click on *Magn.* in the MENU BAR and select one of the preset values from the pulldown menu.

b. Beam voltage and spotsize:

- i. You can change the beam voltage and spotsize by clicking on *Beam* in the MENU BAR to select one of the preset values in the pulldown menu.
- ii. Recommended beam voltages: 0.5-10 kV for non-conducting samples, > 10 kV for conducting samples. 30 kV maximum!
- iii. Spotsizes are numbered from 1 to 7 (only 1-5 are used at UCSB though), with the beam current and diameter at the sample increasing with the spotsize number.
Optimum spotsizes:
Spot 1 => ultra high resolution but poor signal-to-noise (S/N) since the current is so low;
Spots 2, 3 => standard high resolution imaging and good S/N (spot 3 is the usual setting at UCSB)
Spot 4, 5 => poorer imaging resolution but good for EDS analysis.

c. Correcting focus:

- i. Center an area of interest in the IMAGE WINDOW. You may need to adjust the image brightness and contrast (see procedure **4.d** below)
- ii. Click on the Control Area: Imaging icon, , on the TOOL BAR to activate the **Imaging**, **Video**, and **Stage** control groups in the CONTROL AREA of the main window. Click on the **Window** button of the **Imaging** group and a reduced imaging window will appear (which is variable in size and can be moved to anywhere in the IMAGE WINDOW). This window utilizes frame averaging and will give an excellent resolution picture from which to do focusing.
- iii. Press the right mouse button and move the mouse from side-to-side to obtain optimum focus in the window, OR, Use the focus knob on the "Manual User Interface" panel below the monitor.
- iv. If there is significant image shift when focusing, then an electronic Final Lens Align should be performed. Do *not*, under any circumstances, touch or adjust the objective aperture to do this!!!
 1. Set mag to > 10kX (with WD ≈ 5 mm).
 2. Maximize the **Beam** control group in the CONTROL AREA of the window and click in the **Final Lens Modulator** check box-

- a cross will appear in the center of the screen and the image center will be seen to move if the Final Lens is misaligned.
3. Press and hold the left mouse button in the **Lens Align** X-Y control window to generate crosshairs in the IMAGE WINDOW. Drag these crosshairs around the IMAGE WINDOW in order to minimize movement at the image center.
 4. When corrected, turn off the **Final Lens Modulator**. There should now be no image shift observed when the focus control is used.

d. Adjusting image contrast/brightness:

- i. For most applications, simply click on the **ACB** button (auto contract/brightness) of the **Video** control group (in the CONTROL AREA) to see the image.
- ii. For more refined control for a final, publication-quality picture, the adjustment can be made manually. First switch the scan to Line mode by clicking on *Scan* in the MENU BAR and selecting Hor. Line. The image freezes, a horizontal line appears on the screen, along with the SED signal intensity as the beam is scanned along the line. Select the slowest scan speed (since that is used for best picture resolution, see procedure **4.h**) and adjust brightness (dc level) and contrast (ac level) so that the observed signal intensity lies entirely within the 20-80% range of the maximum level (as indicated by the dashed lines on the screen).

e. Moving the stage/image:

- i. Large movements - Click on the Track Mode icon, , on the TOOL BAR which allows continuous stage movement with variable speed in any direction. A green circular overlay appears on the IMAGE WINDOW. Click outside the outer green circle for fastest stage motion. To slow the stage speed, move the cursor from the outer toward the inner circle. Note that the speed is inversely proportional to the magnification.
- ii. Intermediate movements - Selecting the Get mode (click on the Get icon, , on the TOOL BAR) allows you to bring any detail to the center of the IMAGE WINDOW simply by double clicking on it. At magnifications below 6000x, the stage will be driven whereas at higher magnification, the electrostatic beam shift is used.
- iii. Small movements - Selecting the Shift mode icon, , allows you to arbitrarily move the image by clicking and holding down the left mouse button (the cursor will change to a hand) and dragging the image as you need it. There is a beam shift limit of $\pm 15 \mu\text{m}$ in both the X and Y directions in this mode.

f. Correcting astigmatism:

- i. Open the reduced area window as for step "c) Correcting focus" above. Increase magnification to greater than 30000x (the higher the better). Focus the image as well as possible.

- ii. Bring the image out of focus in one direction and monitor the image distortion direction. Repeat by defocusing the beam in the other direction. If astigmatism is present, then the two out-of-focus distortions will be rotated 90 degrees with respect to each other. Bring the focus to the midpoint of these two distortions.
- iii. Press the SHIFT key and right mouse button together to bring up the stigmator crosshairs on the image. Move the crosshair first left to right (x-stigmation) to improve image focus and sharpness and release the mouse when the optimum image is observed. Repeat for the y-stigmation by moving the crosshairs top to bottom and releasing the mouse when the best image is observed.

g. Switching to Ultra-High Resolution (UHR) mode:

- i. Note that if your sample is magnetic you cannot use the UHR mode. Your sample may be sucked up into/onto the Final Lens Cone by the strong magnetic fields needed to setup the virtual Objective Lens for UHR mode.
- ii. Only under certain combinations of WD, beam voltage, and magnification can the machine be switched to the UHR mode from the standard HR imaging mode.
- iii. If the current imaging conditions are not suitable for UHR mode, then the UHR button on the TOOL BAR will be grayed out with a 'magic' red wand (?) through it, . However, clicking on this inactive button will open a "UHR Magnification Setup" window that presents a choice of processes to obtain the desired working conditions for UHR mode (usually magnification is too low). *Never* press the Apply button (which implements the changes automatically). Click Cancel, and do the required changes *manually* only if they are safe to do so. The button will now be highlighted and become active, . Click on it to switch to UHR mode (button will turn yellow).

h. Taking pictures/storing images:

- i. For best quality images, a single pass scan at the lowest speed should be used for your picture. Either manually switch to Slow Scan 4 (*Scan* -> *SlowScan4*) and then hit the Freeze icon on the TOOL BAR, , when the scan is complete, or press F2 on the keyboard, which automatically freezes the image after a single scan at *SlowScan4* speed is completed. If image distortions from sample charging are observed (which will be accentuated when a slow scan is performed), then a faster scan mode will have to be used. Integration of a fixed number of these fast consecutive scans will improve S/N in your image - click on *Filter* in the MENU BAR and select the 'Integrate N' option (the image will automatically freeze after the Nth scan of the integration step is complete).
- ii. Click on *In/Out* in the MENU BAR and select Image from the pulldown menu. The "Save or Restore Image" window will appear.

The default directory in this window should read your subdirectory as: c:\xl\User\xxxxxxx (where xxxxxxx is the first 8 characters of your FEI system username). Note that the 'Databar on image' and 'TIFF file' options should be selected.

- iii. Type in the filename (< 8 characters!) and click on the SAVE button to store the frozen image on the screen to a TIFF file in your subdirectory.
- iv. Measurement display: click on the Measurement button on the TOOL BAR, , and the "Measurement Dialog Box" will appear. Using this box, x and y dimensions can be selected and displayed on the image and subsequently saved to an image file.

5. End of imaging session.

- a. If in the UHR mode, first exit it by clicking on the yellowed UHR button on the TOOL BAR. If the **Beam** and **Vacuum** control groups are not present, click on the Control Area: Settings icon, , in the TOOL BAR to reactivate them in the CONTROL AREA. Turn off the beam and isolate the upper column from the sample chamber by clicking on the yellowed **kV** button in the **Beam** control group.
- b. Activate the CCD detector to image the interior of the sample chamber in the IMAGE WINDOW of the main window (click the CCD On/Off icon in the Tool Bar which will turn yellow when on). Click on the **Vent** button of the **Vacuum** control group. This will first move the sample downwards to the minimum z position and then bring the sample chamber up to atmosphere (button goes yellow).
- c. Reset the stage to its initial position: (x,y) = (0,0), Tilt = 0, Rot = 48 deg.
- d. When the chamber is vented, slowly slide open the chamber door while watching the CCD image to confirm that your sample will not contact the final lens cone. Remove your sample mount from the multi-mount holder on the stage.
- e. Recheck the CCD image as you slide closed the chamber door. Holding the door closed, click on the **Pump** button of the **Vacuum** control group (button goes yellow).
- f. Press the lit **HT** button on the SYSTEM CONTROL PANEL (on the chamber console) to deactivate the high voltage to the filament (button will go dark).
- g. Fill out the logbook (noting emission current (Operate) and the gun pressure (Upper IGP) from the Settings CONTROL AREA, as well as the beam voltage, spotsize, WD used for your imaging session). Be sure to include details on the sample imaged (especially if resists were imaged) and record any issues you may have had with the system.
- h. Exit the XL control software (click on the X button in the top right corner of the Microscope Control window).