

# **ZEISS ULTRA-60 FIELD EMISSION SCANNING ELECTRON MICROSCOPE (FE-SEM) PROCEDURE**

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## **SPECIAL NOTES OR RESTRICTIONS:**

- **Must be qualified to use the tool by Nanofab staff.**
- **Must be given a Smart-SEM account by Nanofab staff.**
- **Always use the specimen exchange assembly to load and unload samples. If a sample breaks or becomes stuck in the specimen chamber, notify Nanofab staff immediately. DO NOT ATTEMPT UNASSISTED REMOVAL!**
- **If the red OFF button light or the yellow STANDBY button light is illuminated, contact Nanofab staff. DO NOT ATTEMPT TO START THE INSTRUMENT!**
- **ALWAYS TURN ON THE CHAMBER CAMERA BEFORE ATTEMPTING TO RAISE THE STAGE. Failure to observe the interior of the specimen chamber may result in damage to the SEM and the sample. Never adjust x, y, rotate, or tilt if there is a chance that the sample may touch the objective lens cap.**
- **When screwing the sample exchange rod into the sample holder, DO NOT OVERTIGHTEN.**

## **SAFETY PRECAUTIONS:**


- **This instrument may generate radiation during operation. DO NOT remove any cover panels, particularly those on the electro-optic column and the specimen chamber.**
- **The maximum acceleration voltage is 30 kV.**
- **Keep the area in front of all ventilation openings clear to prevent fire hazard and overheating of electronics.**
- **Do not bump into the specimen exchange assembly or apply pressure that may bend the specimen exchange rod.**

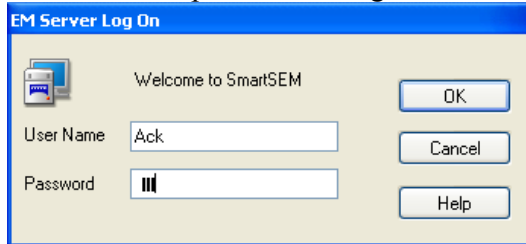
## **LOGGING ON TO THE SYSTEM:**

- **Log on to the network using your NIST network credentials.**
- **Enable the tool in Coral through the ensuing dialog window.**

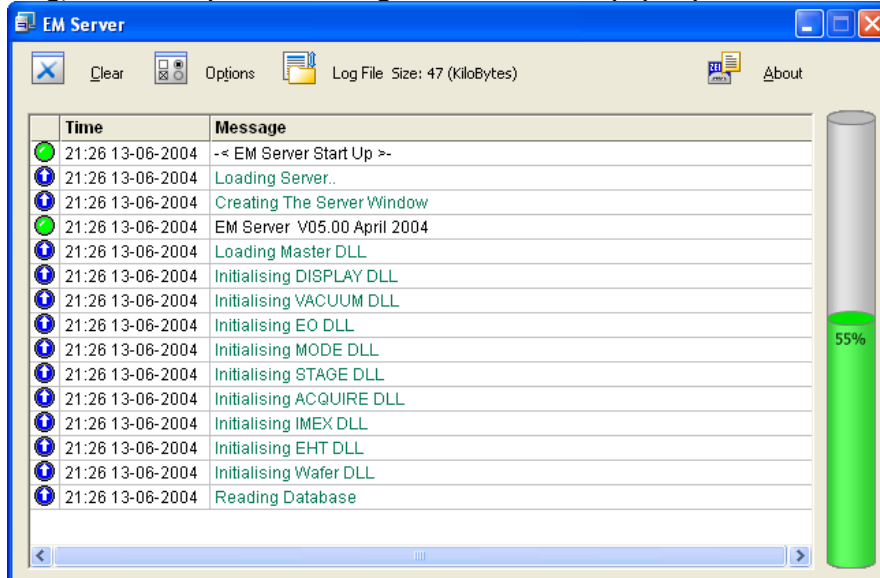
## SAMPLE LOADING:

- Check the FE-SEM vacuum/electronics status panel on the front of the column unit. If GREEN button is lit, OK to proceed. If YELLOW or RED are lit, stop immediately and contact Nanofab staff.

- Click on the Smart-SEM icon  on the left hand LCD to launch program. Enter user name and password to log on and activate the SEM interface.



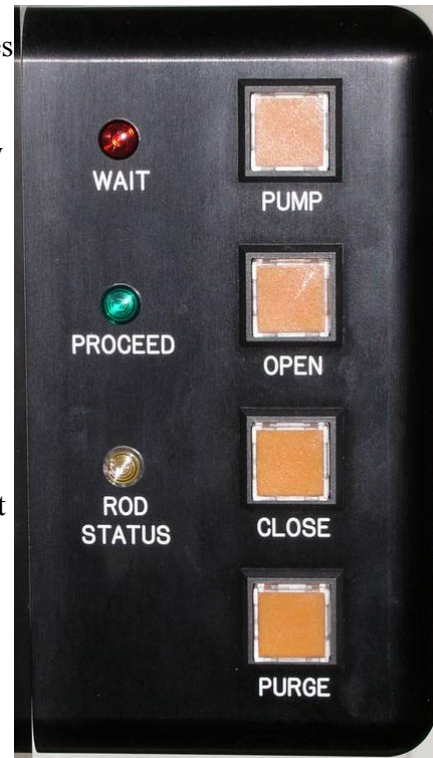
- Verify that EM server is running. If not, contact staff support. You can move (click & drag) the server panel to the right hand LCD if it pops up on the left one.



- Choose sample holder and mount sample with carbon tape, paint, or clips (carbon tape is not suitable for non-conductive specimens. Carbon paint is best for conductivity, but requires several minutes to dry). Sample holders are available for 4-inch wafers, wafer pieces, cross-sections, and mounting samples at an angle. The small orange-handled screwdriver is used to mount the pin-type mounts in the multi-angle holder.
- Press the **CAMERA** button on the keyboard. The interior of the specimen chamber will appear.
- Press the **EXCHANGE** button (15 [refer to control panel graphic on page 4]) on the keyboard. Wait for the stage to move to the exchange position and stop. Position

yourself in front of the sample exchange assembly and wait for the green **PROCEED** light to illuminate.

- Check that the **CLOSE** and **PUMP** buttons are illuminated and **OPEN** button is dark. This indicates that the sample exchange area is under vacuum and the door separating the specimen chamber from the sample exchange chamber is closed. You can verify that the door is closed by looking through the sample exchange window.
- Press **PUMP** button (light will go out).
- Press **PURGE** button (light will illuminate). You will hear N<sub>2</sub> flow into the sample exchange chamber and the door will be released.
- Pull the door back and hook the door latch to hold it open.
- Press **PURGE** button to stop N<sub>2</sub> (light will go out).
- Slide the sample holder onto one of the two mounts and gently screw the sample exchange rod into the sample holder. **DO NOT OVER-TIGHTEN.**



- Release the door latch and carefully guide the door back to the closed position.
- Press **PUMP** button (light will illuminate) and the sample exchange chamber will pump down.

\*\*\* Wait for the green **PROCEED** light to illuminate. This means the sample exchange chamber is pumped down.

- Press **CLOSE** button (light will go out).
- Press **OPEN** button (light will illuminate). The door between the sample exchange chamber and the specimen chamber will open.
- Gently release the specimen rod latch.
- Carefully, without applying any bending pressure to the rod, slide the sample exchange rod into the specimen chamber and slide the sample holder onto the stage in the specimen chamber. If you feel too much resistance, the rod may be slightly off center. Adjust the rod position with the black knob on the front of the sample exchange chamber until the sample slides onto the stage.

- Unscrew the rod from the sample mount, fully retract the rod, and lock the rod into place with the latch. Be careful not to push down on the end of the rod while engaging the latch.
- Press **OPEN** button (light will go out).
- Press **CLOSE** button (light will illuminate) and the door between the sample exchange chamber and the specimen chamber will close.

### Return to the left hand LCD, and click **RESUME EXCHANGE** and **OK**. Wait until Smart SEM moves the stage from the load position to the viewing position and stops.

### OPERATION:

- If the chamber camera is off, turn it ON by pressing the **CAMERA** button (17) .
- If not already displayed, press Ctrl-G to bring up the SEM control panel, and pin it to the empty panel to the right of the image window.
- Click on the **STAGE** tab of the SEM control panel. Using the left hand joystick (Z) carefully raise the stage (+y joystick deflection) without tilting it (+/-x joystick deflection). Make sure tilt remains at 0° on the SEM control panel's stage tab. Stop when the sample is about half way between initial position and the bottom of the objective lens cap.
- Press the **CAMERA** button (17) on the panel to turn the camera OFF.



- Click on **EHT** on the bottom-right portion of the left hand LCD and click **EHT ON**. Use the **GUN** tab on the SEM control panel to change the **EHT**. 5 kV is usually a good starting point, unless the sample is exceedingly thin or has charging problems. The screen should brighten, but the image will probably be out of focus. If image signal is poor (too bright or dark) use brightness and contrast (9 & 10) to adjust
- Record the listed gun and vacuum parameters on the sheet on the table top. EHT is Accelerating voltage. Units in the vacuum display windows can be toggled by

single-clicking the LMB in the parameter display window.

- If not already displayed, Ctrl-D brings up the “data zone” at the bottom of the image. This will be part of your saved images when it is ON. Ctrl-D toggles this feature off and on.
- Select the **APERTURE** tab on the SEM control panel to choose an aperture. The 30 um aperture is a general purpose aperture and a good place to start. Seven apertures are available ranging in size from 7.5 um to 120 um. You may change the aperture at any time, but adjustments for astigmatism and aperture centering may be necessary to achieve an optimum image.
- Select the **DETECTOR** tab on the SEM control panel and choose the secondary detector **SE2**.
- Toggle the coarse/fine bar to coarse (if not already so) with the mouse and focus the sample. Increase magnification, toggle to fine, and focus again. With the image in focus, navigate the stage in X and Y to find the area of interest on the sample.
- Check the working distance (WD) on the data zone. LMB double-click on the data zone WD to open a dialog window, and input desired WD. Before moving stage to match the new focal point, turn the camera on and verify specimen position & movement response of the stage with the Z joystick.
- Turn camera off and continue moving the stage until the SEM image comes back into focus. It is good practice to toggle the camera on frequently to check stage location & movement progress. However, **DO NOT** move the stage with the camera on when close to the objective lens cap. Stage motion speed is greatly accelerated in this mode and risk of collision with the cap increases substantially.
- The right hand joystick controls lateral motion (X/Y) and rotation (twist). All stage motion may also be controlled from within the **STAGE** tab of the SEM control panel. Find the desired features and manipulate the stage to position the sample.
- The **SE2** detector is usually satisfactory for moderate to long working distances and the entire range of accelerating voltages. Using the **SE2** at short WD yields very poor S/N.
- For superior SE image quality at low accelerating voltages (3 kV or lower) use short working distances (2 to 5 mm), and the **IN-LENS** detector. The **IN-LENS** detector may be used **UP TO 20 kV**, but image quality may degrade as working distance increases. Do not use the **IN-LENS** detector above 20 kV; use the **SE2** detector instead.
- The **EsB** is a high-resolution enhanced backscatter and secondary detector that may be useful for working distances of 5 mm or less. **QBSD** is a 15 mm 4-quadrant backscatter detector that must be manually engaged. See Nanofab staff for using the **QBSD**.

- The **IN-LENS detector** at short WD provides the highest resolving power. However, the minimum achievable WD is limited by the kV, and more critically by the physical relationship between the sample and the lens cap. Making contact with the lens cap will not only damage the sample, but also **SERIOUSLY** damage the SEM.
- With the chosen detector and aperture in place, the accelerating voltage selected, and the feature of interest on the screen, adjust contrast (10) and brightness (9).
- If there is a large amount of astigmatism present, perform a preliminary correction with the x/y astigmatism correction knobs on the keyboard (2 and 3). Center the aperture by pressing the **WOBBLE** button (13) on the panel. You may adjust the wobble amplitude in the **APERTURE** tab of the SEM control panel (30 is typical). Use the aperture centering knobs on the keyboard (4 and 5) to stop the image from shifting in x and y directions. Focus and correct again for astigmatism, using either the keyboard or mouse controls.
- Shift-F2 activates lens clear (degauss). Use this if you are unable to correct the astigmatism or have an otherwise unsatisfactory image. Focus again. Repeat lens clear/focus two or three times if necessary until you can obtain a satisfactory image. If there is still a problem, contact Nanofab staff.

#### **SAVE IMAGE:**

- When the image is optimized, you may choose to save it. Within the SEM control panel, you can choose from several types of scans (line/frame average, line/frame integration, pixel average) and speeds.
  - Pixel Average: This gives each pixel the longest continuous exposure to the electron beam
  - Line Average: This gives each pixel an exposure to the electron beam shorter than Pixel Average but longer than Frame Average
  - Frame Average: This gives each pixel the shortest continuous exposure to the electron beam
  - “N” is the number of lines or frames that are averaged together to produce the image
- With averaging, choose a speed and click on **FREEZE** in the SEM control panel when you are ready to save. On the left hand LCD, click on **FILE** and **SAVE IMAGE**. Click on **CHANGE DIRECTORY** then choose drive D and open your image folder. Create a sub-folder if you wish, type in a file name and press **SAVE** or **ENTER**. The micron bar appears on the data zone, but the magnification does not. If you want to save the magnification with your image, append it to your filename (such as “image1\_120kx”). In the SEM control panel, click on

**UNFREEZE**, then return to **PIXEL AVERAGE** and fast speed (perhaps 3) to return to “live” image.

- With integration, choose the number of frames and wait until SEM control panel indicates the integration is finished. On the left hand LCD, click on **FILE** and **SAVE IMAGE**. Click on **CHANGE DIRECTORY** then choose drive D and open your image folder. Create a sub-folder if you wish, type in a file name and press **SAVE** or **ENTER**. The micron bar appears on the data zone, but the magnification does not. If you want to save the magnification with your image, append it to your filename (such as “image1\_120kx”). In the SEM control panel, click on **UNFREEZE**, then return to **PIXEL AVERAGE** and fast speed (perhaps 3) to return to live image.
- To load a previous image, perhaps in order to make and store a measurement on the image, click on **FILE** and **LOAD IMAGE**. Press Ctrl-A to bring up the annotation menu. Move the menu to the right hand LCD, choose the annotation type, and perform the measurement. To save the annotated image, follow the save image procedure described above.

#### **EXCHANGE / UNLOAD SAMPLE:**

- Click on **All:** on the lower-right of the left hand LCD and click **EHT OFF**.
- Press the **CAMERA** button on the keyboard. The interior of the specimen chamber will appear. If sample is very close to the lens cap, move it down with Z to at least midway between the cap and the bottom of the camera image.
- Press the **EXCHANGE** (15) button on the panel.
- Wait for the green **PROCEED** light on the airlock to illuminate. This means the sample exchange chamber is pumped down.
- Verify that the **PUMP** and **CLOSE** buttons are illuminated.
- Press **CLOSE** button (light will go out).
- Press **OPEN** button (light will illuminate). The door between the sample exchange chamber and the specimen chamber will open.
- Gently release the specimen rod latch.
- Carefully, without applying any bending pressure to the rod, slide the sample exchange rod into the specimen chamber and gently screw the specimen rod into the sample holder. Do not over-tighten. Gently slide the sample off the stage, fully retract the rod, and lock the rod into place with the latch. Be careful not to push down on the end of the rod while engaging the latch.
- Press the **OPEN** button (light will go out).

- Press the **CLOSE** button (light will illuminate) and the door between the sample exchange chamber and the specimen chamber will close.
- Press **PUMP** button (light will go out).
- Press **PURGE** button (light will illuminate). You will hear nitrogen flow into the sample exchange chamber and the door will be released.
- Pull the door back and hook the door latch to hold it open.
- Press **PURGE** button to stop nitrogen flow (light will go out).
- Unscrew the rod from the sample holder and remove the sample holder from the mount.

**You may load another sample at this time if desired.**

- Release the door latch and carefully close the door closed.
  - Press **PUMP** button (light will illuminate) and the sample exchange chamber will pump down.
- *If another sample has been loaded, return to page 3, and proceed according to the **SAMPLE LOADING** instructions, starting with step \*\*\* and continuing through step ###.*
- *If another sample is **NOT** to be loaded, wait for the green **PROCEED** light to illuminate to verify that the sample exchange chamber is pumped down; then continue with the next steps.*
- When finished, verify that all samples have been removed from the specimen and sample exchange chambers, and the **PUMP** and **CLOSE** buttons on the sample exchange chamber are illuminated.
  - Return to the left hand LCD, and click **RESUME EXCHANGE**. Wait until Smart SEM moves the stage from the load position to the viewing position.

Log off Smart SEM to end your session (answer yes to “Close UIF?” Popup) and disable the tool in Coral. **PLEASE MAKE SURE YOU LOG OFF THE PC. IF YOU DO NOT LOG OFF THE COMPUTER, THE NEXT USER WILL NOT BE ABLE TO LOG ON.**

- The SEM’s personal user images folders are network-shared. They can be accessed through any NIST networked PC by mapping ‘My Network Places’ > ‘Entire Network’ > ‘Microsoft Windows Network’ > ‘Nist’ > ‘Ultra60sem’ > ‘Ultra60SEM Images’ > ‘images’ > and then selecting a user folder. Alternatively, use ‘Add a Network place’ under ‘My Network Places’ and type in ‘\\Ultra60sem\Ultra60SEM Images\users’ to create a network shortcut folder.