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

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SPECIAL NOTES OR RESTRICTIONS:

- **Must first be qualified to use the tool by NanoFab staff.**
- **Must be issued a SmartSEM user 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 OVER-TIGHTEN.**

SAFETY PRECAUTIONS:

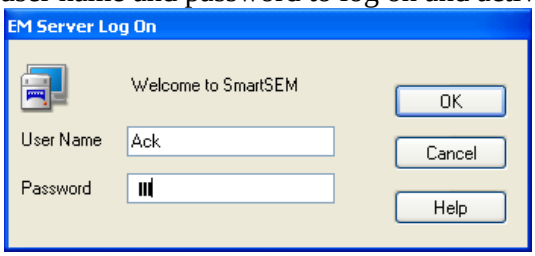
- **This instrument may generate radiation during operation. DO NOT remove any cover panels, particularly those on the electro-optic column and the specimen chamber.**
- **The maximum acceleration voltage is 30 kV.**
- **Keep the area in front of all ventilation openings clear to prevent fire hazard 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:

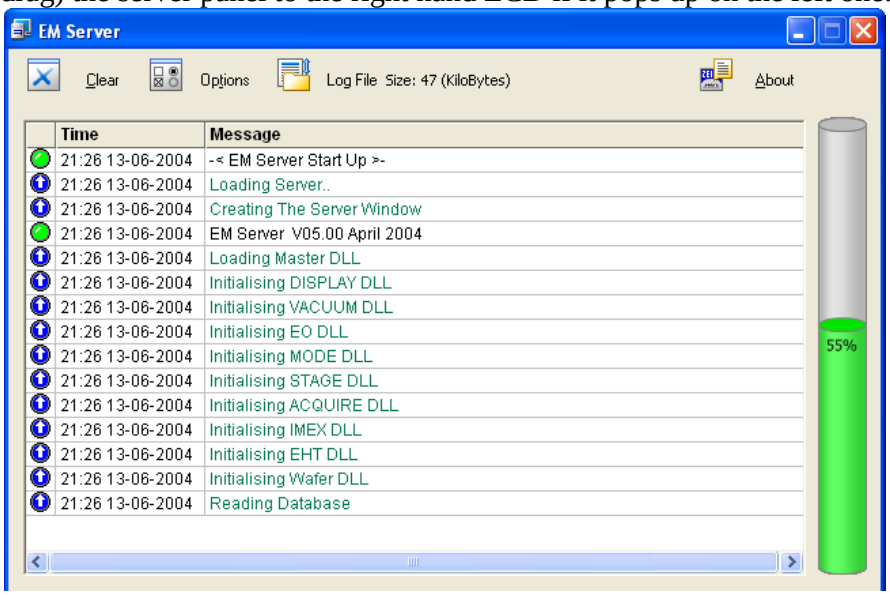
- ✓ Log on to Windows™ using your NIST network credentials.
- ✓ Launch a Coral session from any Sun Ray client (or the Oxford INCA PC) and enable ZEISS FESEM.

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 a NanoFab staff member.
- ✓ 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.



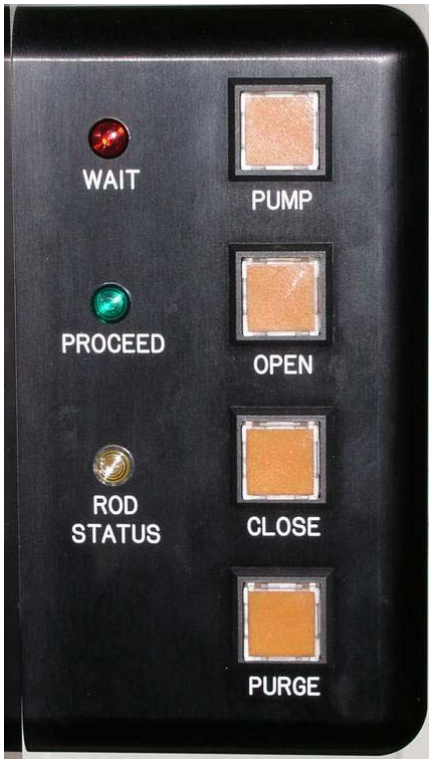
- ✓ Verify that EM server is running. If not, contact staff support. You can move (click & drag) the server panel to the right hand LCD if it pops up on the left one.



- ✓ 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 6 and 4-inch wafers, wafer pieces, cross-sections, and mounting samples at pre-defined angles.

- ✓ 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 confirmation that the stage is at the exchange position (reply OK). Position yourself in front of the sample exchange airlock and wait for the green **PROCEED** light to illuminate.



- ✓ Check that the **CLOSE** and **PUMP** buttons are illuminated and the **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 N2 flowing into the sample exchange airlock and the vacuum seal will subsequently release.
- ✓ Pull the airlock back and hook the door latch to hold it open.
- ✓ Press **PURGE** button to stop N2 (light will go out).
- ✓ Slide the sample holder onto the Teflon airlock stage aligned with the exchange rod, and carefully screw the rod tip into the sample holder. **DO NOT OVER-TIGHTEN**.
- ✓ Release the airlock latch and carefully guide the door back to the closed position.
- ✓ Press **PUMP** button (light will illuminate) and the sample exchange airlock will pump down.

******* Wait for the green **PROCEED** light to illuminate. This means the sample exchange airlock 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.
- ✓ Carefully release the specimen rod latch mechanism.
- ✓ 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. Without applying too much lateral pressure on the rod, guide it so that it safely slides onto and fully engages the stage.

- ✓ Unscrew the rod from the sample mount, look inside the main chamber through the airlock's glass window and confirm that the rod is completely release from the sample holder, and only then fully retract the rod.

- ✓ 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 display panel, and click **RESUME EXCHANGE** and reply yes to the prompt to move the stage to the center of the chamber. Wait until Smart SEM moves the stage from the load position to the viewing position and stops. **IF YOU DO NOT EXECUTE THE RESUME EXCHANGE MACRO, THE COLUMN AIRLOCK VALVE WILL NOT OPEN AND YOU WILL NOT BE ABLE TO TURN THE EHT ON!**

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 0º on the SEM control panel's stage tab. Stop when the sample is about 2cm (on the camera image) from 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 tabletop. EHT is Accelerating voltage. Units in the vacuum display windows can be toggled by single-clicking the LMB (left mouse button) 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 µm 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 **SE2** detector for Signal A, if not already selected.

- ✓ Toggle the coarse/fine bar to coarse (if not already so) with the mouse and focus the sample. Coarse/fine control can also be toggled by pressing the Tab key on the keyboard. 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 (4 to 5mm for best resolution). 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 (3 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 inserted into the chamber. 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 the 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 display, click on **FILE** and **SAVE IMAGE.** Click on **CHANGE DIRECTORY** then choose your target NIST network folder. You may also create a new sub-folder if you wish, type in a file name and press **SAVE** or **ENTER.** The target image storage directory selected will become the default directory for your next session.

- ✓ Close the save dialog window and click **UNFREEZE** on the control panel, and set scanning back to **PIXEL AVERAGE** and fast speed (perhaps 3) to return to a "live" image.

- ✓ With integration, choose the number of frames and wait until SEM control panel indicates the integration is finished. To save the "frozen" integrated image, follow the same steps for saving an averaged image and returning to a "live" image above.

- ✓ To load a previously saved image (perhaps in order to make and store a measurement on the image) click on **FILE** and **LOAD IMAGE**. You may use any of the annotation/measurement tools in the annotation bar at the bottom of the GUI window. 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 display 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, and wait for confirmation that the stage is at the exchange position (reply OK).

- ✓ Wait for the green **PROCEED** light on the airlock to illuminate. This means the sample exchange airlock 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.

- ✓ Carefully release the specimen rod latch.

- ✓ Carefully, without applying any bending pressure to the rod, slide the sample exchange rod into the specimen chamber and carefully 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 flowing into the sample exchange airlock, and the vacuum seal will subsequently release.
- ✓ Pull the door back and hook the door latch to hold it open.
- ✓ Press the **PURGE** button to stop nitrogen flow (light will go out).
- ✓ Unscrew the rod from the sample holder and slide the sample holder off the Teflon airlock stage.

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, then continue with the next steps.*

- ✓ When finished, verify that all samples have been removed from the specimen and airlock chambers, and the **PUMP** and **CLOSE** buttons on the sample exchange chamber are illuminated.
- ✓ Return to the left hand LCD, and click **RESUME EXCHANGE.** If this is the end of your session, reply **NO** to the prompt to move the stage to the center of the chamber. This action will leave the stage at the exchange position for the next user, and set all the vacuum conditions to their proper settings.

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 IT MAY LOCK OUT THE NEXT USER!!**