# Drexel Zeiss SUPRA 50VP

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*These notes are meant to serve as an aid to assist users who have been trained and certified by MCC Staff. If ever you are unsure about the correct operation of the instrument or any of its components, please consult a MCC staff member before continuing. Never use equipment that you are not trained and approved to use.*

*Before using the MCC, please review the MCC User Handbook available through our website.*

Preliminary Checks:

1. Log book - always check logbook first for problems, sign in to logbook

2. Check compressed air
    a. Check compressed air pressure gauge at wall to left. Should be approximately 70PSI (follow Cu pipe down from ceiling, blue hose to mini regulator)

       or if house air is down....

    b. Switch to compressed air tank, with pressure 1OOPSI. (Yellow tank-left gauge)

3. Liquid nitrogen (LN2)
    a. Chamber is vented with $N_2$ gas. RED ball valve is used to OPEN and CLOSE line to vent the chamber. OPEN to vent, CLOSE once chamber is vented. Leave main LN2 tank valve open.

       DO NOT ADJUST-Contact a staff member for help

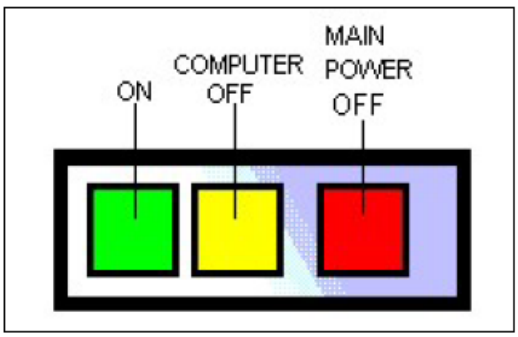
    b. Check compressed $N_2$ tank pressure 3 PSI (Liquid $N_2$ tank, Blue Hose)

       OR if LN2 tank is out for refilling....

       Switch over to compressed $N_2$ (Black tank)

       If LN2 tank is empty, you may disconnect the blue vent line from the regulator in order to vent the chamber with room air. Notify MCC staff.

4. Green light on the SEM is on. Notify Kate Vanderburgh if light is not green.



***MAKE SURE PRELIMINARY CHECKS (above) ARE COMPLETED AND SATISFIED BEFORE PROCEEDING!**

| Procedure | Notes & why this is important! |
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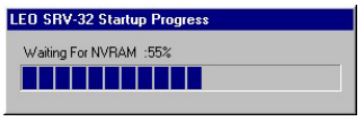
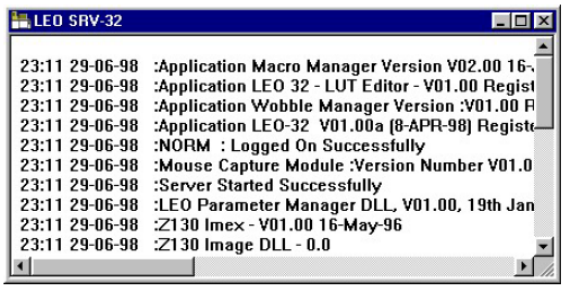
| **STARTUP** | |
| 1) Login to paper logbook. | Users are required to log in and note any issues during use. Check for any issues reported by previous users. |
| 2) Log in to iLab account access to begin session. | Users are required to log their usage of the equipment. |
| 3) Double click on the SmartSEM icon on the desktop.<br><br>The Zeiss logo will be displayed, then two information windows will appear as follows:<br><br>  <br><br><br>  <br> | Log on to the SEM software to communicate with the microscope. If the Zeiss EM Server is already running, it may be minimized and found at the bottom of the left-hand monitor status bar. |
| 4) The log-on prompt will appear. Login to the SmartSEM interface with your username and password. | Each user has a customizable interface and different levels of privileges.<br><br>If a user file has not yet been established, you will not be able to login |

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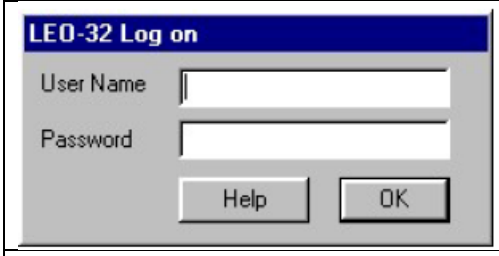
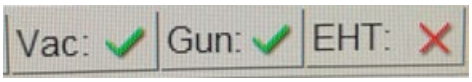
|  | to the SmartSEM software. Users and passwords are setup with the SmartSEM Administrator.<br><br>See Kate Vanderburgh for training and if you need a login and password. |
| 5) Status check: vacuum running, gun on, EHT off. In the bottom, right-hand corner of the interface, the corresponding indicators should appear as follows:<br><br>  | The vacuum should be running when the microscope is not in use. This is to protect the microscope from contamination and support successful operation. |
| **SAMPLE LOADING** | |
| 1) Vent the chamber.<br><br>a) OPEN the RED LN2 ball valve to vent.<br>b) Locate the Chamber view icon in the TOOLBAR (or select TV as detector or Camera button). Place the mouse cursor over the VAC field of the Status Bar and select. A pop-up dialog, VENT will appear.<br><br>  <br><br>c) CLOSE RED LN2 ball valve once chamber is vented. | The chamber is vented with liquid nitrogen (LN2) gas. RED ball valve is used to OPEN and CLOSE line to vent the chamber. This step is necessary to open the chamber door to load your samples.<br><br>Click on VENT. Answer the prompt 'Are you sure you want to vent'. Select "OK" After several seconds the pumps will stop and venting will occur, wait for the pressure to equalize, and then pull open the door (~2.5 min). The stage should automatically drop to the lowest Z position. |
| 2) Load your sample – please make sure your sample is dry and clean!<br><br>Use compressed air and/or plasma cleaner to clean samples prior to loading.<br><br>For training on the plasma cleaner or questions regarding sample prep, contact Kate Vanderburgh. | Since the samples will be in a vacuum, it is important to keep them and internal chamber components clean. WEAR DISPOSABLE GLOVES to handle anything that will be inside the chamber. Since you will typically use a tool in one hand, only one glove is needed to hold the part that will go into the SEM.<br><br>The keyboard, mouse, controller, computer area is a GLOVE FREE zone. |

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| | Please take gloves off before using the controls. |
| 3) Mount sample stubs into desired holder and gently tighten the set screw(s) to secure each stub.<br><br>Remember where you put your samples in the holder. | Measure the height of your samples from the top of a standard SEM stub. Use this value when raising the stage in step 7.<br><br>Inspect the bottom of the holder and adjust the height of any protruding pins. Protruding pins will prevent you from sliding the dovetail mount onto the receiver post. |
| 4) Slide the dovetail portion of holder onto the spring-loaded receiver on stage. The FLAT side of the dovetail goes on first and aligns with the flat side of the receiver. | Make sure that the holder is centered and all the way on the SEM stage otherwise the calibration will be off.<br><br>Ensure that the door seal and mating chamber surface are clean. Close the stage door and hold it closed until the vacuum keeps it closed. |
| 5) Pumping down.<br><br>Once the samples are loaded and the SEM chamber door is closed, Place the mouse cursor over the VAC field of the Status Bar and select. Click on Pump.<br>  <br><br>CLOSE the RED LN2 ball valve once pumping has begun. | The system will take several minutes to reach a working vacuum (1.5 x 10-5 Torr). The exact time is dependent on many factors such as length of time the stage door is open, relative environmental conditions (humidity), condition of sample holder, sample adhesives (if any), and the sample itself. No actions, which are dependent on vacuum, will be permitted until this level of vacuum has been reached. |
| 6) Wait until vacuum ready is reached. When Vacuum Ready is reached, the VAC field of the status bar will indicate a green check.<br><br>  | A vacuum reading can be obtained by selecting either, the TOP MENU or the SEM Control panel - VACUUM, VACUUM STATUS. System Vacuum is the sample chamber. |

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| 7) While the vacuum is pumping down, raise the stage to a working position. | In the SEM control panel, select the detector tab and make sure you have selected the TV detector for this step so you can visually see the stage moving.<br><br>Expand the toolbar to the left of the panel of tabs and select Stage Navigation. Enter the height of your specimen (relative to the top of a standard SEM stub) in the height under specimen. This will change the height of the specimen in the z-axis pictogram of the stage relative to the objective lens. This is an aid to prevent hitting the objective lens. If you are using a wider or taller SEM stub, please take this into consideration before raising the stage.<br><br>Using the JOYSTICK position your sample for initial viewing while in the Chamberscope view by (Camera button on keyboard) raising your sample to the black line on the back of the chamber.<br><br>**DO NOT drive the stage into the electron emitter (also referred to as the pole piece) in the center.**<br><br>If you do happen to hit the electron emitter pole piece, please immediately contact Kate Vanderburgh. |
| 8) When vacuum is ready, turn EHT on.<br><br>  <br><br><br>  <br> | Select a value for target EHT (kV) either with the SEM Control panel Gun tab or the accelerating Voltage Icon (2nd from left in toolbar), or Double click on EHT in the DATAZONE field with the left mouse button. Enter the desired value. The EHT is switched on by selecting the EHT field of the Status Bar and then selecting EHT ON. (If the gun is fully off then this will indicate GUN ON.) |
| 9) Take a breath. You are now ready to begin imaging. | Look over the software and hardware interface to make sure all is set. |

| **IMAGING & ALIGNMENTS** | |
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| 1) Select detector. | a) Secondary electron SE1 Inlens detector - surface structure<br>b) Secondary electron SE2 detector - topography and surface structure<br>c) Backscatter detector (BSD) - Topography and compositional contrast |
| 2) Zoom all the way out and focus. | This is to get your bearings and locate where your samples are. This will be a very rough focus; the fine tuning is coming.<br><br>To change between coarse and fine focus (TAB button). Stage navigation can be performed using the controller or CTRL+tab. |
| 3) Locate your samples in the holder and match sample labels on carousel holder with those of your loaded samples. | Go to stage navigation. For the 9-stub carousel holder navigate to the smaller hole (relative to holes for SEM stubs) between spots labeled 6 & 7. From stage navigation, select settings and go to holder rotation offset calibration. Adjust the holder rotation offset calibration until your selected location matches that of the carousel label. |
| 4) Make sure you are fully zoomed out and select your sample of interest. | The sample location and carousel label should match now. |
| 5) Zoom into the sample to your selected region of interest (ROI). | Make sure you are in focus. A coarse focus may be useful to begin. |
| 6) Collecting high-quality images:<br><br>a) Focus<br>b) Astigmatism correction (x & y) → focus<br>c) aperture alignment (x & y) → focus<br>d) Repeat.<br>e) Brightness contrast (B/C) | Focus at higher magnification than you want to take the image.<br><br>Stay at this higher magnification for focus and astigmatism corrections. Select a faster scan speed (1 or 2) with line averaging (N) = 3.<br><br>For aperture alignment, adjust line averaging to 1 (N = 1) to perform the wobble and x and y aperture adjustments. |

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| | Return line averaging back to 3 (N=3) after aperture adjustments. |
| 7) Focus | Find something on sample to focus on (i.e. feature on sample itself, edge of sample, carbon tape) Coarse and fine focus knobs. Adjust scan speed. |
| 8) Astigmatism correction | Use COARSE to adjust X knob, then focus. Then adjust Y stigmators, then focus to obtain the sharpest image possible. You may need to re-adjust focus. Fine tune the astigmatism adjustments using the FINE mode and re-adjust focus (x knob → focus → y knob → focus). |
| 9) Aperture alignment | A shifting image while adjusting focus indicates an out of alignment aperture. Activate the FOCUS WOBBLE, either from the APERTURES menu in the CONTROL PANEL or the WOBBLE BUTTON on the keyboard. Go to the fastest scanning speed with line averaging set to N=1. Adjust WOBBLE AMPLITUDE according to the current Magnification. Large Amplitude for lower magnifications, Small Amplitude for higher magnifications. Using the APERTURE ALIGNMENT knobs at the top left of the keyboard, adjust the aperture position until the lateral image motion ceases (image looks like it's blinking in place). Select either COARSE or FINE mode depending upon the magnification. Aperture is aligned when there is no image shift while adjusting focus.<br>**Note:**<br>The astigmatism may require re-adjustment after aperture adjustment and Z changes. |
| 10) B/C | Auto B/C is an option. Typically, contrast should be between 25-30% and |

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| | then brightness can be adjusted accordingly. To check black/white/gray scales, select the histogram and your entire ROI. The histogram should look like a bell curve. The majority of the image will be different gray scales but black and white should also be present.<br><br>To manually adjust the B/C, in the scanning tab, check line scan. This should bring up a linescan/rastering of the B/C. Using the B/C knobs on the keyboard, turn both brightness and contrast all the way down. Slowly, increase the brightness until you see a flat line ~ 1 cm above the bottom of the line scan box. Then begin to increase the contrast. Adjust B/C until the linescan is in the middle of the screen with some lines hitting the top and bottom of the line scan box. Close out of the line scan box by hitting the x and your changes will be applied. |
| 11) These steps may need to be repeated until you are satisfied with the image quality. | Take your time and be patient! |
| 12) Adjust scan speed | Select a scan speed that gives a high Signal to Noise ratio (SNR). The image should appear sharp and clear.<br>a) Select image resolution (pixel size). Default is 1024x768.<br>b) Set Noise Reduction upper panel to Freeze on = End Frame<br>c) Set Noise Reduction = Line Avg<br>d) Set N=3 - 10. There is never any reason to average more than 10 frames.<br>e) Freeze the image by pressing the FREEZE button on the Keyboard or selecting FREEZE in the drop down menu.<br>f) A colored indicator will appear in the lower right corner of the image.<br>Orange = Pre Frozen, Red = Frozen, Blue= Frozen & Saved. |

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| 13) Save image | Images can be saved to your directory in C:/USER IMAGES/"your directory" by selecting FILE – SAVE IMAGE ( or keyboard shortcut ctrl+E). It is recommended that users save their images to the C:/USER IMAGES folder.first, then transfer them after your session to the desired storage device (see more info below). Images may also be directly saved to the Network Drive-Shared Folder. Enter a file name. 30 character maximum.<br><br>If desired, select auto numbering so files of a specified name will be auto indexed as you save. To do so, enter a starting value in 'Nest' and then select the number of digits in the dropdown below. Be sure that the Annotation box is checked or you will not have saved the DATAZONE or ANNOTATION objects. Close the Save Images window. |
| **SHUTDOWN** | |
| 1) When you have completed your imaging session, return parameters to the starting conditions. | Zoom out, scan speed 1, set to line averaging N = 3, aperture set to 30 µm, store resolution 1024x768 pixels, imaging mode to SE2 detector. |
| 2) Shut down EHT only. | Leave the GUN on, the current SEM conditions will be saved automatically when you log off. |
| 3) Make sure BSD and EDS detectors are withdrawn | All retractable detectors should be fully withdrawn before venting. |
| 4) Vent the chamber<br><br>OPEN the RED LN2 ball valve to vent. | Before venting, select TV as the detector (or Chamberview icon or camera button) so that you are viewing the Chamberscope.<br><br>The chamber is vented with liquid nitrogen (LN2) gas. RED ball valve is used to OPEN and CLOSE line to vent the chamber. |

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| | The stage should automatically drop to the lowest Z position. |
| 5) Remove sample holder from stage. | Place in sample prep area while closing the chamber door. |
| 6) Close the chamber door, check the door seal, hold the door closed and re-pump the chamber. | CLOSE the RED LN2 ball valve once pumping has begun.<br><br>Please wait until pump down is complete and system is under vacuum before closing the software and logging off. |
| 7) Log Off the SmartSEM software. | Close the program. Select “Yes”, as you wish to log off of the software. |
| 8) Log out on the paper record sheet. | Record any problems in the Comments section. For critical issues/machine damage contact Kate Vanderburgh immediately. |
| 9) Clean up your area. Remove your samples from holder. | Put all tools and miscellaneous items away where they belong.<br><br>Take your samples with you. Please DO NOT leave in sample prep area |
| 10) Transfer any images you want to keep. | Use either a USB stick (please scan for viruses BEFORE bringing to the MCC) or access the networked server accessed via crffiles.coe.drexel.edu |
| 11) Logout of iLab. | End your session in the iLab kiosk. |

Report any problems to Kate Vanderburgh kv434@drexel.edu (908-303-9142)

**Powering the System**

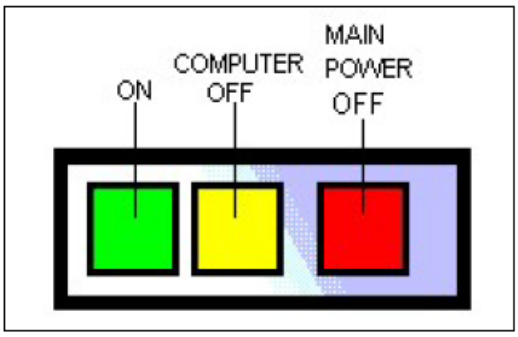
The Zeiss series of Field Emission SEM’s can be divided into two sections:

1.Gun, Column electronics and vacuum. (Left side)
2.Computer (Right side)

Normally, the gun, column and vacuum are left on and running.

The computer and some of the electronics can be switched off when not in use (STANDBY MODE). This is normally not done unless the system needs to be reset.

Three power push buttons are located on the front-top of the Plinth.




The RED (Right) button turns off main power to both sections of the instrument.
The YELLOW (Middle) button turns the computer off.
The GREEN (Left) button will turn on both the vacuum system and computer if the instrument is fully off, OR, just the computer – electronics if in a standby state.

START- UP (The system is normally left on with the chamber under vacuum.) Restarting may be necessary after a power failure.

**To Reboot PC Only (eg.: if software locks up)**

1. Shut off the EHT, if possible. (Try Alt-Ctrl-Del)
2. If possible, log out of the SmartSEM software, LOG OFF, save the conditions.
3. Close the Smart SEM server.
4. Shutdown Windows to get the "It is now safe to turn off your computer" message
5. Press the center yellow "standby" button on the front power panel
6. Wait 10 secs and press the green "on" button
7. "Yes" to continue rebooting
8. Activate CRF Login Icon, Login to CRF server.
9. Login to SmartSEM when the desktop environment is restored.
10. Restart EHT.

**To Reboot Entire System (eg.: if joystick stage control stops working)**

1. Shut off the EHT and shutdown the Gun, if possible.
2. Follow above procedure steps 1 through 5.
3. Press the Yellow "Standby" button.
4. Press the Red "Shutdown" button to power down the system.

5. Go to the back of the Column console and turn the rotary switch from ON to OFF.
6. Shut off Breaker switch located above the rotary ON/OFF knob.
7. Wait 20-30 seconds and then turn the breaker switch on and rotate the knob below it to "ON".
8. Press the yellow center "standby" button and finally the green "on" button. (WARNING: If you wait too long to re-start the system, the ion pump will shut off and it will take several hours for the vacuum to recover.)
9. Continue steps 7 through 9.
10. Once you are logged on, restart the Vacuum system (PUMP) turn on IGP.
11. Once Gun vacuum is in the 10e-10 range restart the gun filament and restart EHT.
12. It will take 10-15 minutes for the gun to become stabilized for optimum performance.

**Other troubleshooting**

Stage movement problems:

1. If the stage does not move or moves and does not stop.
    a. Open the Control Panel and select the Stage tab – Stop Stage Motion.
    b. OR - Select Stage Navigation and Select Stage Stop.

2. Software hung up, will not respond. Try closing the SmartSEM software first. (alt-ctrl-del - task manager)
3. If your sample keeps moving even when you stoop moving the joystick, press “BREAK” on Joystick.

Report any problems to Kate Vanderburgh kv434@drexel.edu (908-303-9142)