CHECKLIST FOR VERIOS OPERATION

1. GENERAL

- The SEM lab is used assuming "operating room" cleanliness, i.e., the SEM lab is a high visibility lab and must be kept clean and neat so clean up behind yourself
- The SEM and all associated instruments are treated as ultra-high vacuum grade equipment, i.e., use gloves when handling samples, sample holders, the sample shuttle, tweezers, tools, and any other object that is going into the SEM or will touch anything that is going in the SEM.

2. Sample Mounting

- Sample stubs should be clean and free of residual hydrocarbons. If you have magnetic samples, please see Chuck.
- Samples should be minimally volatile and free from contaminants, i.e., the sample is clean and won't outgas
- Final sample cleaning is done with methanol, isopropanol, or ethanol. If acetone is used, clean with alcohol last.
    - Methanol is preferred for final cleaning.
- The sample region of interest should be located as close to the center of the stub as possible
- Mount SEM stubs in the shuttle, tighten set screw gently (two fingers!)
- Sample height is checked with the gauge with at least 1 mm clearance between gauge and sample
- Sample holder post is checked to ensure it does not protrude beyond the bottom plane of the shuttle

3. SAMPLE LOADING

- The shuttle is properly seated on transfer arm – the three rubies are seated in the grooves on the load arm
- The shuttle has been clamped and did not move during the clamping process
- The loadlock door is closed and the door is in solid contact with the gasket (push down on the back of the door)
- Click load sample on computer (navigation tab) and watch the process making note that all steps occur without issue

4. PREPARING TO TURN ON THE BEAM

- Confirm vacuum is $5*10^{-6}$ torr or less before turning on the beam
- Double click on the sample of interest in NavCam image to place the sample under the beam
- SEM is initially set for mode 1 operation – if the sample is <1mm tall the starting working distance will be ~8.5mm

5. IMAGING

- Turn on the beam: Choose the landing energy, current, and stage bias desired
    - 2kV and 13pA are a good starting point if you do not know what to choose
- Click on quad1 to make it active, then un-pause the acquisition. Check that this is set to the ET detector in SE mode.
- Find the region of interest, focus, stigmate, and zoom. Do so until you have a reasonable image at ~10kX
    - Don't spend too much time making things perfect in mode1! Do this in mode2!
- Link stage and check that the link was successful, i.e., the stage position and WD in data bar match
- Decrease working distance to 6-4 mm. Users are encouraged to go in stages, i.e., ~2mm per step, to be safe
    - Shorter working distances make better high resolution images
    - Longer working distances make better depth of field
- Switch to Mode2, then check the focus and astigmatism correction (don't try to make it perfect yet!)
- Perform direct beam adjustments, i.e., crossover and HV modulation
- Perform the final focus and astigmatism correction – make the image as good as possible at this point
    - Check the stage link!
- If 2kV and 13pA do not provide good results, adjust the landing energy, current, and sample bias to suit
    - After changing beam conditions, check the direct adjustments as they will likely change

6. FINISHING YOUR SESSION

- Pause the active acquisition(s)
- Turn off stage bias
- Turn beam off
- Make sure that CCD camera is live (i.e., un-paused)
- Retract any movable detectors (CBS and/or EDS and/or STEM)
- Zero the stage tilt and scan rotation
- Click on sample unload from the navigation bar and watch the process
    - The system will take the stage to the sample exchange position automatically
- After transfer, open load lock and press "release" button
- Remove your sample – turn the set screw one turn only
- Re-load the empty shuttle to keep the shuttle clean and the load lock under vacuum
- Confirm that the user log has been filled out

Linking the stage and objective lens -- IMPORTANT

The link function links the Z-stage position with the focal length of the objective lens such that the software knows that the focal length of the objective lens is in focus on the sample at the current stage position.

Microscope manufacturers assume that the sample is in focus when displaying the "working distance" on the data bar. WD on the data bar is really the focal length of the objective lens. When the physical working distance matches the focal length of the objective lens, then the image is in focus. The Verios has a stage position sensor and monitors the focal length of the objective lens. What the Verios does not know is if the image is in focus or how tall the sample is. The link function handshakes the focal length of the objective lens with the stage sensor such that the Z-position displays the physical working distance.

The sample must be in focus before you link!

- Link on the highest point on the sample
- Do not put in multiple samples with radically different heights
- Link early and often, especially after big changes in the Z-stage
- Do not link when tilted! Link when tilt = 0.
- Do a reality check, i.e., look at the chamberscope to observe if the physical position is consistent with the linked position

Detectors (general)

The Everhart-Thornley secondary electron detector (ETD) is the most common detector in an SEM. The ETD is used in mode1 or non-immersion mode imaging. The magnetic field that the sample is immersed in during mode2 or immersion mode operation precludes secondary electrons from reaching the ETD, which is located inside the chamber. In mode2, secondary electrons spiral up inside the column and are reflected by the electron mirror into the through-the-lens detector (TLD). The ETD/TLD use the same electronics and amplifier but different scintillator positions, so they are mutually exclusive, i.e., the ETD is used in mode1 and the TLD in mode2. The ETD/TLD is an optical detector, i.e., the detected electron creates a photon by striking a scintillator. The photon is then amplified by a photomultiplier tube. This detector system responds very quickly and TV rate imaging is possible.

The concentric backscatter detector (CBS), mirror detector (MD), and in-column detector (ICD) are all solid state, high energy electron detectors. As such, they react much more slowly than the ETD/TLD. High energy detectors also require more landing energy and/or current and/or sample bias than the ETD/TLD. Imaging rates of >1us/pixel are typically required. If an image can be observed with the ETD/TLD but not with a solid state detector, increase the landing energy and/or current and/or bias and confirm that the scan rates is on the order of 10us/pixel or more. In a conventional SEM with no stage bias capability, the CBS, MD, and ICD would only detect backscattered electrons. Since the stage can be biased, low energy secondary electrons can be accelerated away from the sample to high enough energies to be detected by a solid state detector, so more topography will likely be observed with these detectors than otherwise would be expected when a stage bias is applied.

If data from one of the solid state detectors is desired, then is smart to collect simultaneous images from the ETD/TLD simultaneously. This way, it is possible to focus with the TLD or ETD (fast detectors) and then perform a slow scan to collect high quality data with the slow solid state detector.

Due to geometric considerations, the ETD will show direction in a sample while the TLD may not. The ETD is mounted on the side of the chamber so parts of the sample that face it will be bright and parts of the sample that face away will be dark. Directionality can make images aesthetically pleasing since humans are used to a point source of light (the sun) creating bright areas where sunlight is reflected toward the viewer and dark areas where light is absorbed or reflected away from the viewer. The TLD is inside the column and electrons that strike it spiral back up into the column so directionality of the sample is lost, i.e., all parts of the sample face the detector.

Due to geometric considerations, the CBS will show more topography than the MD which will show more topography than the ICD. The ICD will show mostly compositional contrast. The MD and CBS will show some combination of topography and compositional contrast.

Since the CBS and MD and ICD are all high energy electron detectors, they will be less subjected to charging than the ETD/TLD. This allows for outside of the box thinking and allows conditions that would not otherwise work in a conventional SEM to be applied. That is, a high landing energy and current may produce beautiful images of an insulating sample with one of the solid state detectors while the ETD/TLD image is unusable.

When the MD or ICD or CBS detectors are collecting data, the chamberscope (quad4) is automatically paused. Never adjust working distance when the CCD is paused.

CBS detector

Increase the working distance to at least 6mm when inserting the CBS detector. Do not use the CBS detector with a WD < 3mm. Low voltages and currents will require stage bias to get a signal from the CBS detector. The direct adjustments will change after CBS insertion, so recheck! If you are going to use the CBS detector, it is generally smart to insert it prior to final alignment. Increase the working distance to 6 when retracting the CBS detector.

The CBS detector requires either significant current (>50pA) and/or significant landing energy (5kV or more) and/or a significant sample bias (>1kV). If you can get images with the TLD or ETD and not with the CBS, then you do not have sufficient signal and need to increase the current and/or the landing energy and/or the stage bias.

STEM detector

There is a transmission detector available. To use this detector, samples must be electron transparent, i.e., prepared as they would be for TEM including mounting on a TEM grid. There is a special holder for TEM grids. Up to six TEM grids can be mounted at one time. The grid holder fits into a special sample shuttle. Once the grid holder shuttle is in place on the stage, get an image of the grid with the ETD and link. Then, choose an unused quad and assign the STEM III detector to it. There will be a pop up window that asks if the correct shuttle is in place. Assuming that it is, click on OK. Then the system will

very likely pop up another window which will state that the stage position is out of the limit for STEM III detector insertion and will ask if the operator wants the instrument to move the stage to a safe position. Answer OK and the system will put the stage in the correct limit range and insert the detector.

Data save recommendations

It is recommended that data be saved in the TIFF (16-bit) format.

- 8-bit TIFFs have 256 grey scales.
- 16-bit TIFFs have 65,536 grey scales.
- 24-bit TIFFs are really 8-bits (256 grey scales) in three colors (red, blue, green) for color images. This is great for color images, but not so good for SEM images, unless you want color graphics superimposed on the grey image.
- JPEGs are not recommended due to compression.

It is important to confirm that the "save image with data bar" check box is checked. If not, the data bar will not be saved and the information contained in the data bar will be lost.

Recommendations on when to use sample bias (from FEI)

If the landing energy is <1keV, always use stage bias
If the landing energy is 1-2keV, maybe use stage bias, i.e., it might help or it might not
If the landing energy is >2keV, never use stage bias

To boost contrast: 50-100V
To reduce drift due to sample charging: 100-500V
For enhanced SE imaging: 500-1700V
For CBS detector imaging: 2kV-4kV

If stage bias is being used and the astigmatism cannot be corrected (the stigmator control box shows it at the edge of the range), then the bias must be reduced. The stigmator correction coils can only push so much on the beam and the astigmatism induced by applying the stage bias can overwhelm the stigmator coils.

Chuck's recommendations: Pretty much the same as FEI except:
-Stage bias can improve images collected with the MD, CBS, and ICD even when the landing energy is above 2keV. -Don't hesitate to try a larger range of biases to reduce stage drift due to charging
-The effect of stage bias on EDS results is unclear. In theory, it shouldn't make a difference as long as there is enough landing energy to create an X-ray.
-For highly insulating samples, a good starting place is a 500V landing energy and a 500V stage bias and a current of 6.3pA

X-ray Analysis

Run TidyUp before you start AZtec. Remember to insert the X-ray detector. The optimum working distance for EDS is 5.2mm, but signal can be collected from 4mm – 6.5mm. Be sure to check the direct

adjustments after you put in the detector, set the working distance, and change to X-ray data collection conditions. If your sample is not coated, uncheck the box in the specimen coating information or the system will ignore the elements selected (the default is carbon coating).

EDS requires sufficient landing energy to excite X-rays and sufficient current for statistics. A good place to start is 2.5 times the X-ray energy for the landing energy and enough beam current for >1500cps (typically on the order of 10keV and 1.6nA). The beam current can be high enough for up to 30% dead time with no issues. If the dead time is very high and/or an odd low energy peak is observed, confirm that the chamberscope is paused. Process time 4 is recommended.

If you collect X-rays in mode2, there will likely be an extra Al peak. Mode1 is recommended for X-ray data collection. Recall that due to the interaction volume, EDS spatial resolution is a function of beam energy and not spot size. This means that if you need to do EDS with extreme resolution, you may need to make a thin section and do EDS in a TEM (e.g., the AIF Titan) at high energy. EDS at high energy on a thin section will mean that the beam does not spread much and nm scale spatial resolution is possible.

EDS in STEM mode is not likely to work. The amount of material in the sample is very small, which will produce little signal and unlike a dedicated STEM or TEM, the detector is located just below the sample inside the same chamber as the EDS detector. The end result is that the EDS detector is bombarded by X-rays from the STEM detector and the few X-rays from the sample are typically at a very low level relative to the Si X-rays from the STEM detector. This generally does not lead to satisfying results.

Magnetic materials

If you have magnetic materials, please see Chuck before inserting them into the instrument. If any magnetic material gets into the lens, the instrument can be rendered inoperable and the subsequent repair will be very, very expensive. In general, magnetic materials are very difficult to image with electrons. The magnetic nature of the sample will push the beam into an odd shape, often one that cannot be corrected with the astigmatism correction coils.

Magnetic materials that are not rigidly fixed on a substrate that is itself rigidly fixed to the sample holder are not allowed. Pieces of magnetic material that are too large to get pulled up into the column are allowed, but they must be rigidly fixed to the sample holder. Immersion mode is strongly discouraged when observing magnetic materials. In immersion mode (mode 2 or high resolution mode), the sample is immersed in a strong magnetic field from the objective lens. In this mode, magnetic materials are subject to a strong force trying to pull them into the objective lens. To repeat, if any magnetic material gets into the lens, the instrument will become inoperable and the repair will be very expensive and very time consuming.

In general:

- Magnetized particles are not allowed unless they are embedded in a rigid material
    - Even then, the surface of the polished sample must be rinsed with alcohol and then vigorously blown off with dry N2.
    - Loose particles (even when stuck down with carbon tape) can get sucked up inside the column
- Thin sections made of magnetic particles in epoxy for STEM are not allowed
    - Epoxy thin sections are easily destroyed by current density allowing the magnetized particle to get sucked up into the column

USERNOTES FOR VERIOS OPERATION

(As of June 1, 2015)

- For most conductive or semi-conducting samples try starting here:
  - Topography: 2kV, 13pA
  - EDS: 20kV, 3.2nA
- For insulating samples, a good place to start is
  - Topography: 1kV or 500V, 6.3pA, and a stage bias of 500eV
- For very insulating samples, drop the current (and try a solid state detector)
  - High quality images from insulating samples require minimal current
  - High quality images from insulating samples may require a short dwell time and line integration and/or image integration
- For topography on insulating samples, the mirror detector and the CBS are unbeatable
- Remember that when you switch between beam settings at high magnification you will likely need to check the direct adjustments (cross-over shape, position, and HV modulator)
- If you have a good link and you know that all of your samples are the same height, you can operate the microscope safely. Most of the things (not all) that can damage the scope are excluded by the software
- For extreme magnifications, you do need WD < 4 mm
  - Use extra care with extremely short working distances (<4mm)
  - Do not use the CBS detector with a working distance < 3mm
  - Do not insert the CBS detector unless the working distance is 6mm or more
  - Do not use working distances <2mm
- If you need ridiculous resolution and/or think you want to use a WD < 2mm, see Chuck