Notes on Verios Operation

IMPORTANT, Read all of this to better understand the operation of the instrument!

It is assumed that you have read this document when you are operating the Verios!

- ## Linking the stage – IMPORTANT – all users must know this

The link function mathematically translates the focal length of the objective lens to the Z-stage position. When the image is in focus, the focal length of the objective lens (WD in the data bar) is equal to the physical working distance (distance from pole tip to sample). Only when the image is in focus should the link button be pressed.

Do not put samples of greatly varying height in the instrument at the same time. If the stage is linked on a short sample, a taller sample can contact the pole tip or expensive detector and cause damage to the instrument. Nothing can be fixed or replaced for less than $30k!

In general, microscope manufacturers use "WD" or working distance to display the focal length of the objective lens. This is because they assume that the user wants sample to be in focus. When the physical working distance matches the focal length of the objective lens 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 allows the user to specify the physical working distance so that stage motion is safe.

If an insulating sample charges up, this can create the effect of an electrostatic lens. When this happens, the focal length of the objective lens is not equal to the physical working distance because of the lensing action from the sample itself.

Always to a reality check and confirm that the physical working distance appears to match the focal length of the objective lens when linking and moving the stage!

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

- ## Column alignment, i.e., direct adjustments

To align the electron-optical column, open the direct adjustments window. Confirm that the “Beam” tab is selected. First check the cross-over and then the modulator.

Cross-over: Press the cross-over button to create an image of the cross-over. Start at the upper right with the UC centering, if the UC is on. The upper corrector is centered when the projection of the beam appears to be a diffraction pattern superimposed on an American football. Once the UC is centered, place the brightest central maxima on the green cross using the Source Tilt.

Lens Alignment: Focus and correct astigmatism to make a reasonable image at a minimum of 25kX. Higher magnifications are OK and, indeed, recommended. Turn on the modulator. The system should choose the modulator (HV or Lens) that is appropriate for the selected beam voltage (low voltages use the HV modulator, voltages above 5kV use the Lens modulator). The system will then start modulating the image. If the column is not aligned, then the image will be observed to shift. Adjust the lens alignment until the image stops shifting and appears to breathe, i.e., the image will go in and out of focus but not shift. A modulator amplitude of 30% (0.3) is recommended. If the motion is very small, it is better to increase magnification than modulator amplitude. Large adjustments may require turning off the modulator and correcting focus and astigmatism and then turning the modulator back on.

- ## Upper Corrector

The upper corrector is a slit type monochromator designed to reduce the energy spread in the electron beam. Since it is highly unlikely that the energy spread is reduced to zero, this means that the beam is still polychromatic, but less so than without the corrector (measurements suggest a factor of 10 reduction). UC mode is recommended for low voltages and currents. It is recommended to leave the UC mode auto button checked. The system will automatically turn the UC on when conditions are correct for its use (5kV or less and 25pA or less). Very little current can pass through the UC, so it will turn itself off when more current is requested than it can pass. Since the energy spread is constant, the UC is not utilized in high voltage conditions where the total beam spread is very small relative to the chosen voltage (>5kV).

- ## Sample Bias

It is possible to apply a negative bias to the stage to decelerate the primary beam electrons before they strike the sample. This allows for a high beam energy in the column, which is good for reducing aberrations and increases the potential resolution, and low beam energy at the sample, which is good for observing surfaces as the interaction volume decreases with decreasing beam energy at the sample. A positive stage bias is not allowed as this would accelerate the beam electrons after they leave the column with no benefit.

Applying a sample bias has additional effects beyond decelerating beam electrons before the strike the sample. Secondary electrons, which are defined as electrons with less than 50eV, can be accelerated to energies high enough to be detected by an electron detector that would normally be blind to SEs. High energy electron detectors are typically called backscatter detectors because only backscattered electrons (BSEs) have enough energy to be detected. Since low energy SE emission is generally associated with changes in topography and BSE emission is generally associated with changes in composition, BSE detectors are usually thought of as producing compositional contrast and SE detectors showing topographical contrast. By applying enough bias to accelerate the low energy SEs to the energy range of typical BSEs, a so-called backscattered electron detector is able to observe topographical effects normally associated with low energy SEs.

An additional effect is that low energy electrons are forced up the column closer to the optic axis. This means that increasing the bias will lead to more of a topographical signal in the detectors that are farther up the column (the MD and ICD). The ICD in particular demands a biased sample.

The rate of success with biased samples increases if the sample is symmetric and is symmetrically located on the stub, increasing the chance of a uniform field at the region of interest. Large stubs should generally work better since the E-field will change less across the stub. If the astigmatism is indicated to be all the way to the edge of the adjustment range and more astigmatism correction is needed, the only solution is to decrease the bias.

- ## 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 (for a low voltage or current beam): 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 and CBS even when the landing energy is above 2keV.
-If you want data from the ICD, no matter what landing energy, apply 2kV or more of stage bias
-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. In practice, ???
-For highly insulating samples, a good starting place is a 500V landing energy and a 500V stage bias and a current of 6.3pA

- **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 (primarily) in mode2. Note that data from the TLD can be obtained in mode1 but the signal will be low. 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 and MD would only detect backscattered electrons and the ICD would not detect much of anything due to its position in the column. 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. In general, the image from an ETD will appear to be from the perspective of the column with the light source being at the detector’s position with respect to directionality. 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. The MD and CBS will show some combination of topography and compositional contrast. As sample bias is increased, more secondary electrons are forced back up the column and the signal from the MD will start showing more topographical features and the ICD will begin to show signal.

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, aka the CCD camera, (quad4) is automatically paused. Never adjust the Z-stage or tilt when the CCD is paused.

- ## ICD detector

The ICD is a special case detector that works unlike other high energy electron detectors due to its position in the column. The ICD is far enough up inside the column that electrons are not likely to reach it unless they are forced to do so. Geometrically, it is not likely that many BSEs will strike the ICD. If one chooses a high voltage and current with no stage bias, very little signal is observed on the ICD.

To get reasonable images from the ICD requires a relatively low landing energy (~5keV or less) and significant sample bias. Under these conditions, the sample bias forces secondary electrons (defined as electrons with less than 50eV when the exit the sample) up the column and simultaneously accelerates them to enough energy to be detected by the ICD. This makes ICD images both have significant topographical features (from the SEs) and also appear relatively flat (from the geometry). The ICD is also the most sensitive to sample contamination, again due to the low electron emission coefficient of the deposited C-contamination, the suppression of SEs from the surface below the contamination, and SEs being accelerated to enough energy to be detected by the ICD. Therefore, despite the fact that the ICD is a solid state, high energy electron detector the image formed is almost purely secondary in nature with very few BSEs reaching the detector.

Take care in interpreting ICD images.

- ## CBS detector

Increase the working distance to at least 6mm when inserting the CBS detector. Do not use the CBS detector with a WD < 4mm. Stage tilt is not possible with the CBS inserted. 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 insert the CBS detector before you align the column.

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.

- ## X-ray Analysis

Run TidyUp before you start AZtec. Remember to insert the X-ray detector. The optimum working distance for EDS is 6.0 – 6.5 mm. Signal can be collected over a wide range of working distances. The signal drops off dramatically below 5 mm and above 8 mm. 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.

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 (if you don't know what is in your sample, start with 20kV 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 can be 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. If the X-ray detector is inserted and the sample is at 5.2mm, then this should not be a problem.

- **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

NOTES FOR VERIOS OPERATION

- The physics of the beam mean that lower energy beams will interact closer to the surface generating images that are more topographical in nature. As the energy of the beam dips below 1keV, topographical contrast will change as the interaction volume for low and high Z (and density) materials starts to converge. At extreme low energies (100 eV), the interaction volume becomes extremely small and the contrast between high and low Z materials is greatly reduced.
- For traditional looking SEM images, choose beam energies that are 2keV or higher.
- For most conductive or semi-conducting samples try starting here:
    - Topography: 2kV, 13pA
    - EDS: 20kV, 3.2nA
    - Conventional SEM settings work well, examples of which would include a beam energy in the 5 – 20 keV range with currents in the 13 – 200 pA range.

- For insulating samples, a good place to start is
  - Topography: 500V and a stage bias of 500eV with a low current (13pA or less, if possible)
- For very insulating samples (amorphous insulators such as glass), 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 – use the snapshot or photo function to integrate with drift correction.
- Good, bad, or indifferent, some samples look much better with a thin coating of a conductor. 5 – 10 A of Au/Pd or a thin coating of amorphous carbon can make a very big difference in image quality
  - No matter how thin the coating, at or above 50kX magnification, Au/Pd will be observed as texture on the surface.
- For topography on insulating samples, the mirror detector and the CBS seem to work well. Both of these detectors are sold stage detectors that will require stage bias or significant current or beam energy (or some combination).
- 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)
- For extreme magnifications, you may need WD < 4 mm
  - See Chuck for authorization for using working distances less than 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