ANFF ACT FEI Verios 460 SEM with Gatan MonoCL 4 & Oxford EDS Standard Operating Procedures



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Contents

Verios Quick Start Guide
General microscope setup
Imaging using the Immersion Lens 2
MonoCL 4 Quick Start Guide
System startup
Mirror alignment
Imaging
Spectroscopy
Mapping
System shutdown
Energy-Dispersive Spectroscopy Quick Start Guide
System startup
Analyzer
Point & ID
Linescan
Map
System shutdown
Sample Exchange
Through chamber door
QuickLoader operation instructions
Troubleshooting
Useful Contacts

Verios Quick Start Guide

General microscope setup

- 1. Insert the sample in the chamber through the QuickLoader or the chamber door.
- 2. Activate the infrared CCD camera by selecting Quad 4 and clicking on the **II** icon in the menu bar. The blue border and blue Databar indicate currently selected Quad.
- 3. Collect a Nav-Cam image by either:
 - a. Stage Take Nav-Cam Photo...
 - b. Ctrl + Shift + Z
- 4. Double click on the Nav-Cam image to move to your sample. The green cross indicates the area that is directly under the electron beam and consequently centred on the screen.
- 5. Select appropriate electron beam conditions (Beam Current and High Voltage). Suggested parameters: 5 kV and 25 pA
 - Low voltage will give more surface information.
 - High current will give lower noise but also lower spatial resolution.
 - Lower voltage can improve imaging of low conductivity/insulating samples.
 - Ideal beam conditions are sample dependent.
- 6. Click Beam On. The button turns yellow to indicate the beam is active.
- 7. Make Quad 1 active by clicking on it and start the image scanning by clicking on the **II** icon in the menu bar or pressing F6. The blue border and blue Databar indicate currently selected Quad.
- 8. Activate scanning preset 1 or 2 by clicking on the number in the top right of the screen.
- Adjust the Brightness & Contrast, Focus and astigmatism. The reduced area mode ris useful when focusing and adjusting the stigmators as the scan speed is faster in this mode. Can also be activated by pressing F7.
 - Brightness & Contrast: F9 for auto BC or respective knobs on the control panel.
 - Focus: Right click and drag left and right (↔), or use the Coarse and Fine focus knobs on the control panel.
 - Astigmatism: Shift + right click and drag left & right (+) to adjust the X stigmator or up & down for Y, or use the Stigmator X and Stigmator Y knobs on the control panel.
- 10. Once the image is accurately focused and astigmatism corrected, use the Link Sample Z to Working Distance button <u>⊼</u> → <u>⊼</u> to change the sample height reference to the bottom of the pole piece. The sample Z value changes to the current WD.
- 11. In the Navigation page change the sample height (Z) to 6 mm. Check the focus again and re-link the stage height $\ge \rightarrow \boxed{\mathbb{X}}$.

Esc will stop any stage movement. Don't hesitate to press it if you are not sure that the initiated movement is safe!

- 12. Change the sample height to 4 mm, focus and relink the sample Z to the WD.
- 13. You are now ready for general imaging.
 - Always focus on the sample surface and Link Sample Z to Working Distance before changing the sample height.
 - Follow stage movements on the CCD camera in Quad 4.

Imaging using the Immersion Lens

The Immersion Lens is used to collect high resolution images of samples at high magnification. To reach the ultimate resolution of the SEM, beam and lens alignments must be performed.

- 1. Follow General Microscope Setup instructions.
- 2. Change to Mode 2: Immersion by clicking on the icon in the menu bar. \frown \rightarrow
- 3. Open the Direct Adjustments window using the icon. The Direct Adjustments window will appear.





(b) Stigmator Centering tab

- 4. In the Beam tab click on the Crossover. Adjust the Contrast until you can see a circular spot. Use the Xover Zoom slide bar to adjust the size of the image.
- 5. Using the Source Tilt box crosshair, adjust the tilt so that the circular spot aligns with the centre cross in Quad 1.







(b) Good Crossover

- 6. Exit crossover mode by clicking on the Crossover.
- 7. Adjust the brightness, contrast, focus, and stigmators again.
- 8. Adjust the lens alignment by activating either HV Modulator or Lens Modulator. Use the recommended setting. Minimise the image wobble by using the Lens Alignment box crosshair. You may wish to use the reduced area mode different while adjusting the Lens alignment.
- 9. Once the Lens Alignment is adjusted, stop the lens wobble and check the focus and astigmatism.
- 10. Open the Stigmator Centering tab in the Direct Adjustments window.
- 11. Activate Modulator X and minimise the image wobble using the Stigmator Center X box. Repeat for Modulator Y.
- 12. It is recommended to check the lens alignment again after adjusting the stigmator centering.
- 13. Adjust the focus and stigmators once more. The beam should now be properly aligned.
 - If you change the beam conditions (current/voltage), you will need to repeat these alignments.

MonoCL 4 Quick Start Guide

System startup

- 1. Ensure that the HT knob on the MRU is wound all the way anti-clockwise and the HT switch is set to Off/Reset.
- 2. Ensure that the multi position filter housing is set to an empty position.
- 3. Power on the recirculators for both the PMT and CCD by connecting the black DA-15 cables to the towers. Ensure there is air flowing from the vents.



4. Turn on the PMT controller from the power switch at the back and start cooling the PMT. If you hear a beeping sound, the recirculator is not powered on. Power on the recirculator and power cycle the PMT controller. Then turn the cooling on.



5. Turn on the CCD, DigiScan, and PA4 controller. The power switch for the CCD is on the inline power supply while that of the PA4 controller is at the back.



(a) CCD power supply



(b) PA4 controller and Digiscan

- 6. Leave the system to warm-up and stabilise for around 10 min before turning on the PMT HT. The PMT temperature should stabilise to around -30 °C.
- Note: If you want to use the InGaAs detector or perform cryo CL, the detector or chamber needs extra preparations. Please contact ANFF staff beforehand.

Mirror alignment

Mirror alignment is important to achieve maximum collection efficiency on the system. Alignment is best performed on a flat, uniform sample which emits high intensity CL.

- 1. Load samples in the chamber, focus and set the working distance to 7.5 mm on a bright flat sample.
- 2. Set beam conditions to obtain adequate CL. For a silicon substrate, a beam energy of 30 keV and beam current of 1.6 nA will give enough CL emission to allow for alignment. Lower beam power may be more appropriate for bright samples.
- 3. Open Digital Micrograph on the left most computer. You may need to change the input to DVI-D if the screen is black.



4. Set the scan rotation to 60°. The mirror is mounted to the microscope chamber at an angle of 60°. Changing the scan rotation to 60° makes alignment easier.



5. Insert mirror in chamber using the mirror insertion knob until the mirror aperture is roughly centred on the crosshairs in Quad 1. The mirror is a made from a machined piece of aluminium and is quite delicate. Be careful not to crash the mirror into the sample/stage.



(a) Mirror insertion knob



(b) Mirror position

- 6. **Pause CCD chamberscope in Quad 4.** This turns off the IR LEDs in the SEM chamber which can flood and trip the PMT.
- 7. Set the light path to panchromatic in the MonoCL Control window. The button should show Pan when the light path is set to panchromatic mode. If it's showing Mono, click on it to switch to Pan.



Panchromatic mode on left and Monochromatic mode on right. Click on button to toggle lightpath.

8. Turn the PMT HT on and slowly increase the PMT HT to about -1000 V from the MRU. Make sure the overload light is not blinking red. It the PMT is tripped, cycle the HT supply switch on the MRU.



Manual Remote Unit (MRU)

- 9. Click on Search in the DigiScan window. This should open up 3 windows SEM, CL, and Pulse 0.
- 10. On the CL or Pulse 0 Windows, use the Line annotation tool to draw 2 diagonal lines from opposing corners to mark the centre of the scan window.



- A V V O S
- 11. Slowly move the stage up manually using the Stage Z control on the Navigation page in the xT Microscope Control software. Keep an eye on the current Z value. You may have to adjust the Pixel Time to get decent CL signal and also the Brightness/Contrast.

Stage Z	?
Down	Up
<	>

12. Move the stage up until the light bundle in the CL images is focused into a bright spot. This should happen at a WD of \sim 6.5 mm.

Keep an eye on the counts and the overload light to make sure the PMT is not being saturated. Do not go higher than 6 mm or you risk hitting and the bending mirror, and/or damaging your sample.



(a) Under focal point

(b) At focal point

(c) Above focal point

13. If the hot spot is not in the centre of the screen, under the intersection of the 2 diagonal lines, physically move the mirror using the insertion knob and the thumbscrews at the front of the MonoCL4 box to bring the hot spot in the centre.

The mirror aperture does not need to be centred, only the hot spot has to be.



If scan rotation is set to 60°, the mirror knob will move the mirror up and down while the thumbscrews move it left and right on the screen.

You may have to readjust the sample height to get the hot spot in focus again if the mirror is moved significantly.





- 14. The system should now be set-up for CL imaging. It is worthwhile adjusting the image focus again and noting down the working distance.
 - When navigating long distances over the sample move the stage down ~0.5 mm first. Then move to the area of interest in the x-y plane and finally move the stage back to the correct height. The sample and/or stage are never really flat and moving long distances can cause the sample to hit the mirror.

Settings on the MonoCL Control window are detailed below:



- 1. Displays the name of the grating in use (as defined by the Profile Description in the Gratings Properties dialogue) or if no grating is seated.
- 2. Indicates whether in panchromatic or monochromatic mode and, when in monochromatic mode displays the central wavelength (in nm).
- 3. Free control over the rotation of the grating (i.e. wavelength) left pointing arrow drives the monochromator to a shorter wavelength, square stops driving the grating (backlash automatically removed) and the right pointing arrow drives the monochromator to a longer wavelength.
- 4. Opens wavelength dialogue box to set the central wavelength.
- 5. Choice of stepper motor speed (up to maximum defined in the PA4 Setup dialogue recommended maximum 333 steps).
- 6. Select detector input channel 1, 2 or 3. PMT is 1, InGaAs detector is 3. 2 is not used.
- 7. (Sensing) Mode
 - a. PMT Photon counts per second.
 - b. PSD (Phase sensitive detection) Output of lock-in amplifier. Note that the chopper must be turned on from the MRU to activate PSD. Software does not sense whether this has been done or not.
 - c. Linear Analogue signal level after MRU, i.e. influenced by brightness and contrast levels.
- 8. HT Status switches between PMT HT on/off (when MRU in computer control only) and indicates when an HT trip has occurred. Pressing this radio button when HT trip is indicated resets the HT to a PMT.
- 9. Indicates the current chosen light path. Clicking on this radio button switches between the two modes. When mirrors are being moved between the two positions the status box turns red.
 - a. Pan panchromatic mode (light bypasses the spectrometer).
 - b. Mono monochromatic (light enters the monochromator).
- 10. Indicates whether the light is going through the exit slit to a single channel detector (Serial) or to the Parallel detector. Clicking on this radio button switches between Serial and Parallel mode. When mirrors are being moved between the two positions the status box turns red.
- 11. Tools icon to open the PA4 Setup dialogue.
- 12. Count rate signal is counts per second and depends on sensing mode.

Imaging

To collect an image:

- Bring the desired area under the electron beam and set the correct scan rotation. Move the stage down before moving long distances over the sample.
- 2. Press Search on the DigiScan window.
- 3. Set appropriate HT, Contrast, and Brightness from the MRU to get decent contrast.
- 4. Set acquisition conditions from the DigiScan Set-up window, e.g. image size, pixel time, integrations etc.
- 5. Set appropriate filters in the lightpath from the filter housing, if needed.
- 6. Press Record.
- 7. A monochromatic image is collected as above, but with the light path set to Mono and Serial and selecting the appropriate central wavelength.

The various options on the DigiScan window are detailed below:



- 1. Search keeps on scanning frame by frame until stopped.
- 2. Preview is similar to search, but with different configured scan parameters.
- 3. Record initiates a new scan based on the preset parameters as defined in the Set-up Dialogue window and will terminate the scan at the end of the defined acquisition.
- 4. Restart restarts the Search, Preview or Record acquisition.
- 5. When a scan is active the button is green.
- 6. Stops an acquisition immediately. Note: the Esc key performs the same function.
- 7. Zoom indicates the value of the DigiScan zoom.
- 8. Displays the pixel size of the front most image.
- 9. Pixel dwell time can be altered during an acquisition.
- 10. Scan rotate can be altered during an acquisition.
- 11. Waveform monitor signal intensity during scanning is superimposed on top of the image as a red horizontal Z-trace.
- 12. Control beam places the beam in spot mode as defined by a cross hair in each of the appropriate live images.
- 13. Scan reduced area enables the user to scan a selected area within the field of view. This is done by drawing a rectangular ROI on an image which is live and then clicking the icon.
- 14. Increase the pixel density doubles the number of lines and pixels/line in the live acquisition.
- 15. Brings up the DigiScan Set-up Dialogue to configure scan parameters e.g. image size in pixels, time per pixel, number of integrations.

Spectroscopy

To obtain ParaCL spectra from the CCD:

- 1. Make sure the mirror is properly aligned.
- Navigate to the area of interest on your sample.
 Move the stage down before moving long distances over the sample.
- 3. Select the central wavelength from the MonoCL Control window to set the collected wavelength range appropriately.
- 4. Ensure the light path is set to Mono Parallel to direct the light to the CCD.
- 5. Start a live spectrum acquisition by clicking on View in the ParaCL Acquire window.
- 6. Move the sample up and down to achieve maximum recorded intensity on the live spectrum. It may be helpful to set spectrum binning to 8x to maximise signal at lower integration times while adjusting the sample height.
- 7. Adjust acquisition conditions e.g. exposure time, spectrum binning, etc. Then press Acquire and choose the number of spectra to collect.

 1
 ParaCL Acquire
 2

 4
 Exposure (s):
 1.5
 5

 3
 View
 Acquire
 6

The options on the ParaCL Acquire window are shown below:

- 1. Exposure time (s) defines the integration time used to acquire a spectrum.
- 2. Up and down arrows doubles or halves the exposure time respectively. Note: keyboard up and down arrows have similar functionality if the ParaCL Acquire window is foremost.
- 3. Status button turns green to indicate detector is in use.
- 4. View press for live update of single Parallel CL spectrum according to set integration period.
- 5. Acquire button press acquire for single acquisition of Parallel CL spectrum in a new window. Note: multiple frame integration is possible, but with this function, the dark noise (if requested) is only recorded once.
- 6. Tools button press to open the ParaCL Acquire Setup window. More details below.



- 1. Corrections can be either Unprocessed or Dark Subtracted. Dark subtracted subtracts the saved dark reference from every spectrum recorded.
- 2. Spectrum binning The number of pixels in the dispersive plane averaged together to form a single data point in the spectrum. Increasing spectrum binning increases the signal to noise ratio in the spectrum and increases the camera readout speed, however, this is at the expense of spectral resolution.
- 3. Vertical binning The number of vertical (non-dispersive plane) pixels averaged together to form a single pixel. Increasing vertical binning increases the spectrum readout speed and also increases the signal to noise ratio in the spectrum. However this is at the expense of dynamic range.
- 4. The tools button opens another window that controls which pixels of the CCD are used. Default conditions are full area.

Mapping

To acquire a CL map:

- 1. Make sure the mirror is aligned properly.
- 2. Navigate to the area of interest on your sample and maximise the CL signal. Move the stage down before moving long distances over the sample.
- 3. Acquire a ParaCL spectrum to judge suitability of spectrum wavelength range, exposure time and spectral resolution.
- 4. Using the DigiScan with any analogue or pulse signal, record a survey image that contains the area of interest.
- 5. With the most recently recorded image front most, click Assign Image from the Spectrum Imaging window.
- 6. Draw a square/rectangle or linescan on the survey image using the region of interest tool. This appears as a red and white dashed square or line on the survey image.
- 7. In the Spectrum Imaging window press Assign ROI and select Spectrum image from the dialogue. The dotted line turns green and is labelled as a spectrum image region.
- 8. Choose the number of pixels in the X or Y directions and/or the pixel size, taking into account the likely experimental duration and file size. SI pixels are square so the software automatically calculates the orthogonal coordinate based on the aspect ratio of the ROI.
- 9. You can apply drift correction by drawing a square on the survey image using the region of interest tool. Press Assign ROI and select Spatial Drift to set the area as a reference for spatial drift correction. You can customise the drift correction parameters in the Spectrum Imaging Setup window.
- 10. Assess the acquisition time required per pixel based on a spectrum acquired using ParaCL acquisition and enter the pixel dwell time.
- 11. Ensure that the light path is set to parallel mode and that the beam conditions have not altered.
- 12. Press Start on the Spectrum Imaging window.
- 13. You can stop the map acquisition at any time and save the data collected so far, no need to wait for the map to be finished.

The options on the Spectrum Imaging window are shown below:



1. Assign Image – assigns an image to be used for spectrum image acquisition. Turns to Deassign Image after assignation.

- 2. Assign ROI assigns a sub-area or for spectrum image acquisition (only selectable after Assign image has been performed).
- 3. Define the acquisition area (or line) using number of pixels, step size and/or range. Spectrum-imaging pixels are square so the software automatically calculates the orthogonal coordinate based on the aspect ratio of the ROI.
- 4. Choose pixel dwell time (taking into account the experiment duration) using a ParaCL Acquire spectrum for guidance.
- 5. Estimate of total acquisition time updates during acquisition to show remaining time.
- 6. Show live data i.e. spectrum at each pixel during acquisition.
- 7. Turns green to indicate that spectrum image acquisition is in progress.
- 8. Start the spectrum image acquisition.
- 9. Pause spectrum image acquisition in case dark reference requires repeating.
- 10. Estimate of total spectrum image file size.
- 11. Tools button to open Spectrum Imaging Setup dialogue. You can modify drift correction settings there.

The Spectrum Imaging Setup window is shown below and drift correction settings, e.g. frequency, can be set by clicking on the Tools button.

Spectrum Imaging Setup
Scanning device: DigiScan (software-sync) 🔻
Number of passes: 1
SI Signals
🔽 CL 🔀
Scan Corrections
V Spatial Drift
Scan Options
🔽 Acquire DigiScan signal(s)
Apply sub-pixel scanning
Data Management
🗖 Auto-Save data 🛛 🕅
OK Cancel

SI Spatial Drift Correction	x
Frequency Display Advanced	
Apply correction after every 1 row(s) Apply correction on resume from Pa pixel(s) row(s) frame(s)	
ОКСС	ancel

System shutdown

After your CL experiments, please shut the system down as follow:

- 1. Wind the PMT voltage all the way down and switch the HT off.
- 2. Close the Gatan Digital Micrograph software.
- 3. Lower your sample to a WD higher than 7.5 mm.
- 4. Retract the mirror fully until the indicator light on the MonoCL box turns green.



- 5. Stop the cooling on the PMT and turn off the controller.
- 6. Turn off the CCD, DigiScan and PA4 controller.
- 7. Let the coolant flow for at least 1 h after shutting down the system to prevent damage to the CCD or PMT. You can leave the recirculators on overnight and power them off the next day if needed.

Energy-Dispersive Spectroscopy Quick Start Guide

System startup

1. Check the LED lights on the detector. The left one should be green and the right one blue. If any of the LEDs is off or red, please contact ANFF staff.



- 2. Load samples in the chamber, navigate to the area of interest, focus and set the working distance to **4 mm**. The detector is set up for maximum collection at a working distance of 4 mm.
- 3. Pause CCD chamberscope in Quad 4. This turns off the IR LEDs in the SEM chamber which can oversaturate the EDS detector.
- 4. Open AZtec on the left-most PC. You may have to swap the input if it's showing the CL PC.



5. Create a new project, or open a saved one from the Welcome Screen.



- Click on the EDS Detector Control at the bottom right of the window. Go to the Thermal tab and check that the Operating Status is Cold. If not click on Operate. Wait for the flashing blue LED on the detector to go solid (~10 min).
- 7. Go to the Position tab, click on In, and wait for the detector to be fully inserted in the chamber.

EDS Detector Control: X-Max Detector	EDS Det	ector Control: X-Max Detector 🔶 [
Thermal Operating Status: Cold Position Operate Standby	Ther	tion State: Fully Retracted Activity: Not Moving In Out Stop

(a) Thermal tab

(b) Position tab

8. Choose your required module from the drop down menu.



Analyzer – Integrate all incoming X-ray photons from the sample into a single overall spectrum.
Point & ID – Quickly analyse composition at a specific point or across a region of interest.
Linescan – Determines the concentration of elements across a user-defined line.
Map – Determines the spatial distribution of all elements in the specimen.

9. After choosing an option you will be presented with the Describe Specimen window. There you can write notes about the sample and the parameters used which will be included in any saved reports. If the sample is coated, make sure to enable the option and put in any coating details and its thickness, e.g. C, Au, Pt, etc.

O Image: Confirm Specimen Image: Confirm Elements Image: Confirm Spectra Image: Confirm Spectra	Guided Custom
Summary Pre-defined Elements Project Notes	
Click here to begin entering notes about your project.	
Specimen Notes for 'Specimen 1'	
Click here to begin entering notes about your specimen.	
Specimen Coating Information:	
Specimen is coated Beam calibration element is coated Coating element: Carbon	
Cooling element Carbon Thickness (nm): 2.00 Density (g/cm ³): 2.25	

- 10. In the Pre-defined Elements tab you can select any element of interest.
- 11. Set the beam conditions appropriately. The beam energy should be at least 2 to 3 times the energy of the lines to be investigated.
- 12. Verify that you have a good Output Count Rate (green bar, about half-width of the screen) and a reasonable Dead Time (between 30% to 50%). If not adjust the beam conditions accordingly.

Analyzer



- 1. Go to the region of interest on your sample.
- 2. Start scanning with a quick scan rate or set to spot mode from the xT Microscope Control software on the main PC.

In this mode only, the beam scan is controlled by the microscope and AZtec is just detecting any X-rays that are produced. AZtec has full control of the beam scan in other modes.

- 3. Go back to AZtec.
- 4. Switch to the Acquire Spectra tab, set the acquisition conditions, and then click on ▶ Start. The spectrum collection will run until the preset time is reached or until you stop the acquisition by clicking Stop.
- 5. If you move over the sample during acquisition, the collected spectrum will not reset and will add X-rays from the new region to the one in memory.
- 6. In the Confirm Elements step (that you can access even while the spectrum is still being collected) you can confirm all elements that you want to consider in your analysis and Include or Exclude them as you please.
- 7. Go to Calculate Composition where you can deconvolve some elements and export the results as reports.

Point & ID



- 1. Go to the region of interest on your sample.
- 2. Go to Scan Image and scan an SEM image first. The detector which is currently selected in Quad 1 of the xT Microscope Control software will be used.
- 3. In Acquire Spectra set the relevant acquisition conditions.
- From the left side menu choose to scan from a point or an area then click on ▶ Start. If no selection is made, the spectrum will be collected from the entire scanned area. The spectrum collection will run until the preset time is reached or until you stop the acquisition by clicking Stop.
- 5. In the Confirm Elements step (that you can access even while the spectrum is still being collected) you can confirm all elements that you want to consider in your analysis and Include or Exclude them as you please.
- 6. Go to Calculate Composition where you can deconvolve some elements and export the results as reports.

Linescan



- 1. Go to the region of interest on your sample.
- 2. Go to Scan Image and scan an SEM image first. The detector which is currently selected in Quad 1 of the xT Microscope Control software will be used.
- 3. In Acquire Line Data set the relevant acquisition conditions.
- 4. Click and draw a line on the SEM image to collect X-rays from that area, then click on ► Start. The acquisition will run until the preset time is reached or until you stop the acquisition by clicking Stop.
- 5. Go to Contruct Linescans to choose the elements to be included in the lines or to be excluded and other linescan parameters such as smoothing.

Мар



- 1. Go to the region of interest on your sample. Make sure the sample is stable and does not drift over the map acquisition time period.
- 2. Go to Scan Image and scan an SEM image first. The detector which is currently selected in Quad 1 of the xT Microscope Control software will be used.
- 3. In Acquire Map Data set the relevant acquisition conditions.
- From the left side menu choose the area to scan then click on ► Start. If no area is selected the map will be collected from the entire scanned area. The acquisition will run until the preset time is reached or until you stop the acquisition by clicking Stop.
- 5. Go to Contruct Maps to choose the elements to be included in the map or to be excluded.

System shutdown

1. Retract the detector from the chamber by clicking on Out from the Detector Control. You can leave the cooling on.



- 2. Close AZtec.
- 3. Copy required reports and files to the SharedData folder.

Sample Exchange

Through chamber door

If the sample is too big to fit through the QuickLoader, you will have to use the chamber door.

- 1. Move the stage all the way down.
- 2. Move the stage all the way near the door by changing setting the X coordinate to 81 mm.
- 3. Click on Vent. A dialogue box will appear to confirm the vent procedure. Venting the chamber will take around 2 minutes.



- 4. Once the chamber is vented, carefully pull open the chamber door.
- 5. With the sample on a stub and the stub in a shuttle, carefully slide the assembly in the shuttle holder. Since the shuttle goes in the holder only way, you may have to rotate the stage for access.





- 6. Double check the sample height so that it will not hit the pole piece or anything in the chamber.
- 7. Carefully push the chamber door closed.
- 8. Click on Pump. Evacuation of the chamber will take around 5 minutes.
- 9. You can centre the stage under the column by pressing Ctrl + 0. This only move the stage in the X-Y plane will not change the sample Z.
- 10. Once a good vacuum level is reached (<6 \times 10⁻⁵ Torr), the microscope icon will turn fully green. Wait a couple of minutes before turning the beam on and start imaging.



QuickLoader operation instructions

Loading samples

1. Place the shuttle on the shuttle holder with the grub screw facing the pin.



Shuttle for holding sample and shuttle holder with size gauge

- 2. Place the pin stub in the small hole in the shuttle with the **smaller side of the shuttle wedge at the top**. Tighten the grub screw enough to hold your sample in place making sure to not over-tighten it.
- 3. Make sure your sample will fit through the QuickLoader gate valve by placing the sample size gauge on the shuttle holder. If any part of your sample/holder is touching the gauge, you will have to cut down the sample or use the main chamber door.
- 4. Open the lid of the QuickLoader by pulling the handle directly up and then twisting it anti-clockwise out of the way.
- 5. Place the shuttle in the QuickLoader with the large opening facing the loading arm.
- 6. Turn the handle of the loading arm down, to the unlocked position.
- 7. Push the shuttle onto the loading arm to compress the spring fully, and then twist the loading arm handle up into the locked position. The shuttle should be secured onto the loading arm.
- 8. Close the lid and press (P) to pump the quick loader, while applying a small amount of pressure in the center of the lid to aid in sealing.
- 9. Once the QuickLoader has stopped pumping, wait until the chamber vacuum icon on the FEI software turns orange and you hear a click from the gate valve.
- 10. Open the gate valve by twisting the gate valve handle to unlock and pulling the handle out. Once the handle is all the way out twist to the locked position to hold the gate in place.



Gate valve opening procedure

11. Using the loading arm handle, fully insert the shuttle and then twist the handle into the unlock position, pull back and then twist up.



Sample loading procedure

- 12. Fully retract the loading arm and twist it to the locked position, then close the gate valve.
- 13. The microscope will automatically pump once the gate valve has been closed.
- 14. Follow the Verios Quick Start Guide to begin imaging.

Unloading samples

- 1. Before using the QuickLoader to remove your sample, ensure the stage tilt is set to 0°, the final lens mode is set to Mode 1: Field-Free, and the beam is turned off.
- 2. Check that the lid is closed and press (P) to pump the QuickLoader. The stage will move to the loading position automatically.
- 3. If the vacuum is still OK in the QuickLoader chamber the pump may not come on. Wait until the chamber vacuum icon on the FEI software turns orange and you hear a click form the gate valve.
- 4. Open the gate valve by twisting the gate valve handle to unlock and pulling the handle out. Once the handle is all the way out twist to the locked position to hold the gate in place.
- 5. Using the loading arm handle, insert the loading arm. Before fully inserting the loading arm twist the handle down into the unlock position, fully insert the loading arm and then twist up into the locked position.



Sample unloading procedure

- 6. Fully retract the loading arm and twist it up into the locked position, then close the gate valve.
- 7. Press (v) to vent the QuickLoader.
- 8. Open the lid of the QuickLoader by pulling the handle directly up and then twisting it anticlockwise out of the way.
- 9. Hold on to the shuttle and twist the loading arm down into the unlocked position to release the shuttle.
- 10. Twist the loading arm back to the locked position and close the lid.
- 11. Pump the QuickLoader again by pressing (P).

Troubleshooting

I can't see any image.

- Check that the beam is on.
- Check that scanning is not paused.
- Check that the beam is not blanked.
- Adjust the contrast and brightness or press F9.
- If the image is still black make sure the crossover is centred.

I can't adjust the astigmatism.

- Right click on the Stigmator 2D Control in the beam tab and select Zero.
- Focus and fix the stigmators again.

The image is still blurry when I focus.

- Fix the astigmatism as above.
- Do the lens alignments.

I can't switch to Mode 2: Immersion.

• Double check the focus and working distance and change the working distance to 4 mm.

The stage is stuck of a "Motion needs to be recovered. Please try your action again." or a "The stage hardware error." message pops up.

- Retract all detectors and run the Home Stage procedure either from Stage Home Stage or Shift + F3.
- If the error persists restart the microscope server.

Start	Stop	Show UI	Stop UI	×	I
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- Click on Stop and wait until the blue status bar ramps down.
- Then click on Start, wait until the xT microscope Control starts.
- Log in and home the stage.

The main microscope monitor doesn't turn on.

• Change the computer from 2 to 1 on the black box under the two monitors. The computer button is on the left.

There is no CL signal.

- Check that the mirror is correctly inserted.
- Check that light is going through the correct path Pan, Mono, Parallel, etc.
- Remove any filters in the light path.
- Increase the PMT HT.
- Increase the beam current to generate more emission.
- Increase acquisition time.

I get a "Failure Opening Camera: 'PIXIS'' message on Digital MicroGraph.

- Close Digital MicroGraph.
- Turn the power to the CCD off and disconnect the USB cable.
- Wait for some 15 s.
- Plug the USB cable back in and power the CCD back on.
- Open Digital MicroGraph again.

For other issues contact ANFF staff.

ANFF Sta	Ĩ	ANFF Labs			ANU Security	52249
Olivior	21002	SEM-CL	56239	JCSMR 1.147		
	01000	AI D/XRD/Hall	51597	W2 04		
			01007	VV 2.04	Graeme Cornish	53063
Kausnai	51594	ICP/PECVD	57272	W2.05		20002
	50380		50100		Mobile:	0404857302
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Gayaui	00060	EBL/NIL	57273	W2.06		
Mykhaylo	51590	Snutter	51730	Huxlev Bridge	David Anderson	61269
Chrie	50745				Dennis Gibson (EU)	51573
		MOVELTZ	00800	ו הד החום וארים	Dot I oper (Electricion)	50005
Sue	52495	MOVPE 3	58887	Accel Blda 107	ר מי במווץ (בופטווטומוו)	
			F1817	(next door insist)	Mobile:	0416931718

Jodle Bradby	<u>EME</u>
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Useful Contacts