Thermo Scientific Axia ChemiSEM Scanning Electron Microscope Operating Procedures

Specimen Preparation and Handling

⇒ For high vacuum mode, sample material should be clean: no oil, grease or dust residue and handled with powder-free clean room gloves;
  - If the sample is non-conductive, a thin, conductive layer will improve image quality (but may cover up fine structure that is <10 nm)

⇒ For low vacuum mode, non-conductive polymer and biological samples may be observed in their natural state when surrounded by the water vapor environment;

⇒ Most 10-15 mm or smaller specimens are attached with conductive carbon tape or carbon/silver paint to the SEM mount and placed into the slot of the sample holder carousel (push down into the slot, for a flush fit);

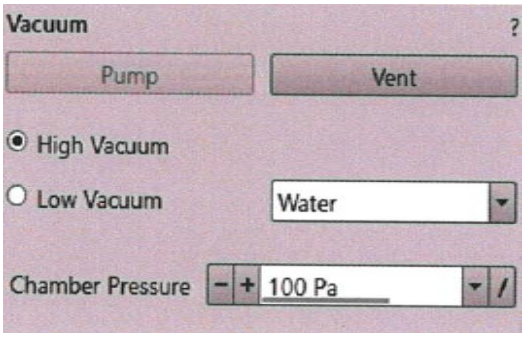
⇒ Larger specimens, such as wafers or cement samples may be attached directly to the sample holder carousel, with conductive tape – ensure it is electrically grounded to the metal holder.

Built-in safety measure: If an action is not possible under the operating conditions, a dialog box appears on the screen and a "tooltip" at the bottom of the screen gives the reason and suggestion for an action to take.

Getting Started with the SEM

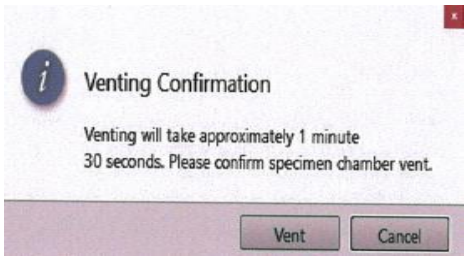
Log into SUMS with your GT credentials, choose Axia ChemiSEM and "Log In".

Loading/Exchanging the Specimen(s)



⇒ In the Vacuum module, click Vent (see diagram).

⇒ "Venting Confirmation" pops up; click "Vent" in the box to confirm.



⇒ A progress bar on the bottom of the screen will show when the chamber has reached atmospheric pressure.

⇒ After the chamber is at air, open chamber door by swinging it on its hinge, from left to right.

⇒ Wearing gloves, grab the holder on either side and pull it straight back.

⇒ Place the holder on a clean surface (lint-free wipe or Al foil), insert specimen mount into one of the holes, push down the pin, for a flush fit (no tools needed). Repeat for next specimen.

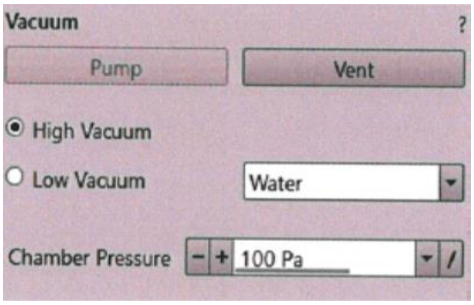
⇒ Put the holder with specimens back into the chamber, onto the stage.

⇒ Close the chamber door, click Pump in the Vacuum Module, and hold the door shut for a few seconds to ensure it seals.

⇒ Select "High Vacuum" or "Low Vacuum" in the Vacuum module, as appropriate for your work

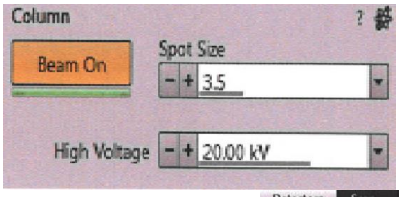
⇒ For "Low Vacuum", "Water" is pre-selected; you may set the target "Chamber Pressure"

⇒ While waiting for the proper chamber pressure, bring the specimen's highest point to the 10 mm yellow line visible in the CCD display.

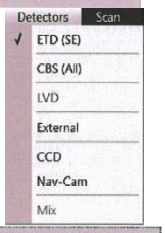


Turning on the Beam and Choosing a Detector

When the microscope icon in the status bar at the bottom of the screen is green, the chamber pressure is ready for the beam.

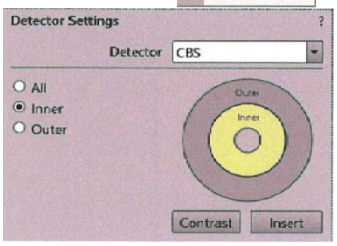


⇒ In the Column module, choose "High Voltage" value for the session (check Fast Start chart on page 6) and click "Beam On". Intermediate values for kV and spot size may be entered into the text boxes.



⇒ Select the appropriate detector [ETD (SE) or CBS (All) in "High Vacuum" mode; LVD Is automatically selected in "Low Vacuum" mode.]

- CBS detector is to be inserted prior to imaging with it.
  - CBS (All) – max. atomic number contrast is emphasized
  - CBS (Inner/Outer) – topographical contrast is added to the atomic number contrast
- Signal collected depends on working distance, lens mode and beam deceleration.



Finding the Specimen and Using Stage Controls

Stage movement is by keyboard arrows or mouse wheel-click.

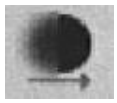
❖ To use the keyboard arrows, press Shift and the arrow key for the direction.

❖ To use mouse wheel-click, press and hold down the mouse wheel and drag the mouse in the desired direction. The graphic shown at right will be seen on the screen.

⇒ Decrease the magnification to the minimum value (~30X) using the knob on the hard panel, or "Toolbar" list box or keyboard (+/ - / *). The star (*) key rounds the mag value.

⇒ Apply "Auto Contrast & Brightness", using the icon at the top of the screen (or F9).

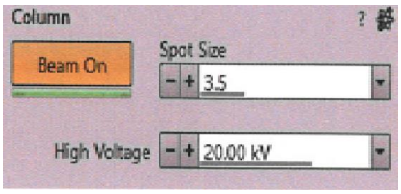
⇒ Focus the image with either the knob on the hard panel, or apply “Auto Focusing” using the icon at the top of the screen (or F11).



⇒ Apply “Link Z to FWD”.

⇒ Apply auto “Astigmatism Correction” or use knobs on the hard panel (or Control + F11).

Optimizing the Beam for Imaging



⇒ Accelerating voltage and beam currents may be quickly adjusted for the active display from the toolbar dropdown list boxes. Intermediate values may be entered into the text boxes in the Column module.

⇒ Adjust spot size for the analysis conditions desired: spot sizes 1- 1.5 -> charging samples and high resolution; spot sizes 2-5 -> standard imaging; spot sizes 4-7 -> high current for X-ray analysis

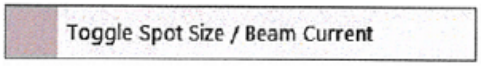
⇒ Select a feature on the sample that is either round or has well-defined edges (avoid straight lines).

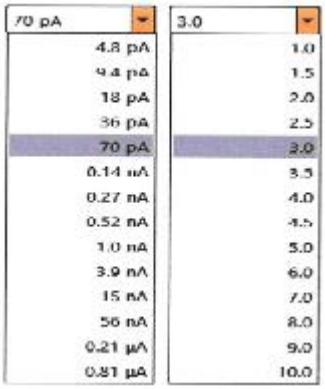
⇒ Adjust the magnification to a value about twice the value wanted for the recorded image; if image is to be recorded at 5Kx, adjust the focus at 10Kx.

⇒ Manually correct astigmatism:

- Focus the image, then move through focus, to the left and right (using hard panel knob) to check for stretching or smearing distortions
- Adjust the hard panel Stigmator X and Y knobs, then adjust the focus again to check for stretching and smearing; if stretching and smearing is still visible, adjust Stigmator X and Y knobs again until the image appears clear and sharp when moving through the focus setting.

If focus and a clear image cannot be achieved, the spot size or beam current may be too large. Try a different combination. Beam current and Spot size may be toggled by right-clicking on the toolbar text box.





Measurement and Annotation

The measure features for distances, angles, diameters and areas are found in the Measure/Annotation toolbar. Each tool shows its description as a tooltip.

⇒ Click on the down arrow to open the list of available tools.
⇒ To deactivate the drawing mode tool, press ESC on the keyboard.
⇒ Selecting T from the Annotations menu will place text on the image.

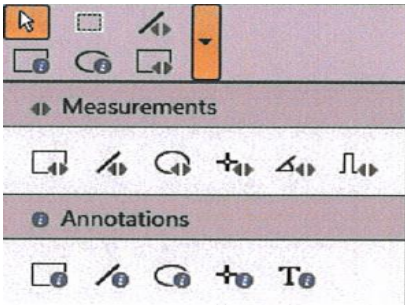
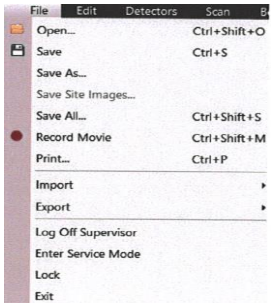


Image Capture (single)

⇒ Adjust the scan speed to suit the specimen: slow scan for conductive samples, faster scan with averaging or integration for poorly conducting samples.
⇒ Select the pixel resolution from the dropdown list.
⇒ Optimize the image for magnification, focus, astigmatism.
⇒ Check the videoscope to set contrast & brightness manually between the top and bottom yellow lines, or apply “Auto Contrast & Brightness”.
⇒ Pause the image display from the toolbar or F6.
⇒ Click the camera icon in the toolbar. Save the image: File -> Save
The software will add an incremental number to the last filename used.
File -> Save As will allow change in folder and/or file name.
Indicate folder, type of file, then “OK”.



Recording Movies (multiple images)

⇒ File -> Record Movie menu item starts and stops recording of all active displays at the same time.
Ask for a demonstration of this feature if using it for your work.

ChemiSEM Elemental Analysis

<u>High Vacuum operation:</u> yields the most accurate results for the areas of interest, if the specimen is electrically conductive.

<u>Low Vacuum operation:</u> chamber gas molecules deflect some of the electrons from the area of interest and these electrons generate X-rays that may be unrelated to the area of interest. Using the lowest possible gas pressure will help reduce this effect.

⇒ The ChemiSEM EDS detector is always inserted and X-ray elemental information is being collected, except when CCD is selected. Collection is refreshed after any imaging parameter is changed.

To view the elemental maps, click the color wheel icon in the toolbar:

To view the spectrum, click the spectrum icon in the toolbar:

⇒ Additional tools will appear, including point, region, line analysis, and mapping.

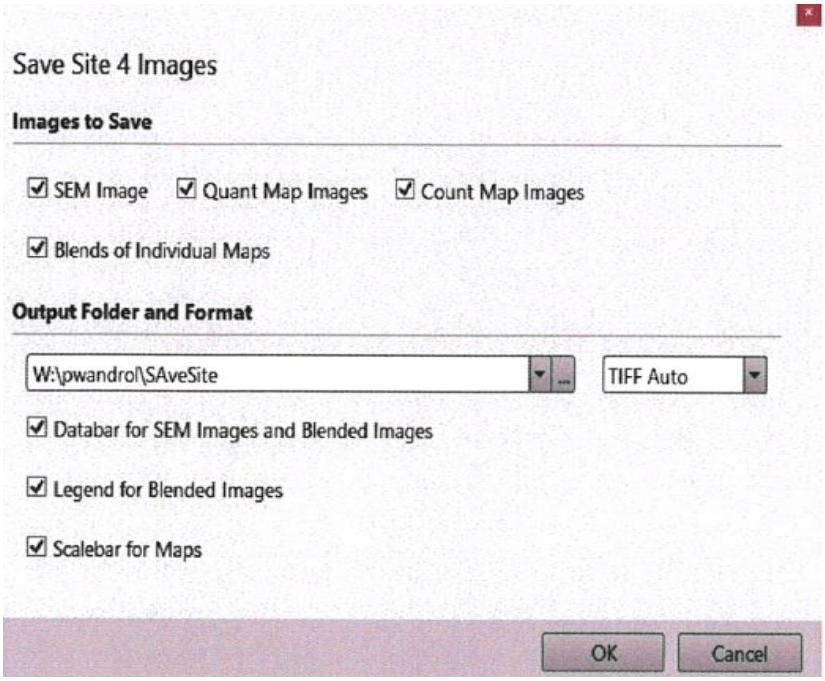
⇒ To select or deselect elements, click the element in the displayed periodic chart.

- Right-clicking on an element in the periodic chart allows the displayed color for that element to be edited.

Creating a Report

⇒ Under "Analysis History" is the list of maps and analyses collected during the session and a button to "Create Report". The report will be saved as .rtf file.

⇒ To save only the maps and images, use File-> Save All:



End of Session Specimen Removal and Instrument Conditions

⇒ In the Column module, click "Beam On" to turn off the beam.

⇒ In the Vacuum module, click Vent.

⇒ "Venting Confirmation" pops up; click "Vent" in the confirmation box.

⇒ A progress bar on the bottom of the screen will show when the chamber has reached atmospheric pressure.

⇒ After the chamber is at air, open chamber door by swinging it on its hinge, from left to right.

⇒ Wearing gloves, grab the holder on either side and pull it straight back.

⇒ Place the holder on a clean surface (lint-free wipe or Al foil), remove specimen mount(s) from the holes. Repeat for each specimen.

⇒ Put the empty holder back into the chamber, onto the stage.

⇒ Close the chamber door, click Pump in the Vacuum Module, and hold the door shut for a few seconds to ensure it seals.

⇒ Record your session in the instrument logbook.

Log out of the Axia ChemiSEM and then log out of SUMS.

Axia ChemiSEM Suggested Conditions, for fast start

Adjustment	Electron Beam Setting
Vacuum mode	*High Vacuum*: conductive samples *Low Vacuum*: nonconductive, mixed or contaminating samples
Accelerating Voltage	Select voltage relative to the specimen type: - low kV for surface imaging, beam-sensitive samples and slightly charging samples - high voltage for conductors, high resolution, composite info (BSE, X-ray) Examples: - biological samples HV = (1 - 10) kV - metal samples HV = (1 - 30) kV
Beam Current ***Spot size***	100 pA at 30 kV 3 - 5
Scan rate	*High Vacuum*: fast scan (dwell time 0.1 - 0.3 µs) *Low Vacuum*: slow scan (dwell time about 3 µs)
Working Distance (FWD)	Set the highest specimen point to approximately 10 mm (yellow mark in an optical imaging display), tilt to 0° and press Ctrl + F (set FWD to 10 mm function).
Magnification	Set to lowest, from 20× to 200×
Standard Detector	*High Vacuum*: ETD (SE) *Low Vacuum*: LVD
Filtering	*High Vacuum*: Live or Average (2-4 frames for fast scans) *Low Vacuum*: Live
Contrast and Brightness	Start with the *Auto Contrast Brightness* functionality, otherwise try following procedure: With contrast at the minimum value, adjust the brightness to just show a change in intensity on the screen. Increase the contrast to produce reasonable imaging. Increasing brightness and decreasing contrast produce softer imaging and vice versa.