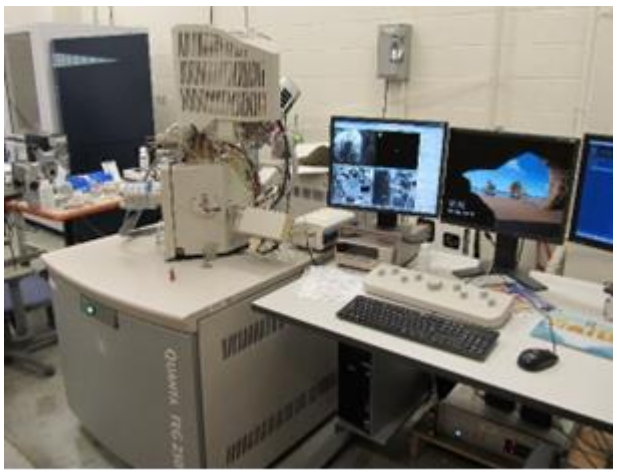
College of Engineering Electron Microscopy Center	Operational SOP	Date	May 30, 2023
	FEI Quanta 250 E-SEM/Helios NanoLab 660/G3	Revision	1.0
		Primary Trainer	Nicolas Briot

Purpose: Basic energy dispersive spectroscopy operation (EDS) on the FEI Quanta 250 E-SEM & Helios NanoLab 660/G3

Required PPE:

Nitrile or latex gloves



Quanta FEG 250 E-SEM

Helios NanoLab 660/G3

Potential Hazards:

This instrument generates x-ray radiation when the electron and/or ion beam are ON.

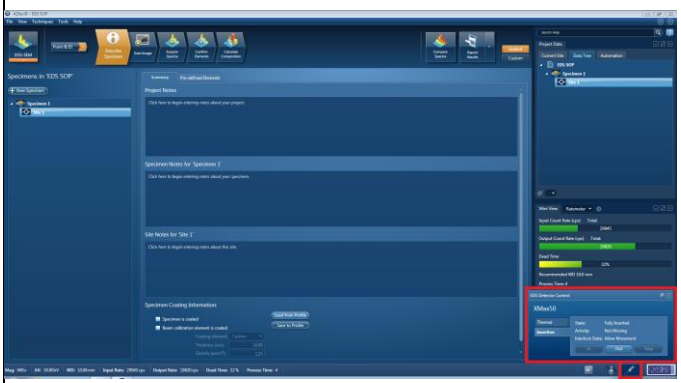
Some parts of this instrument create strong magnetic fields, although not extending more than 15 cm (6 in).

Reference Documents:

- Scanning Electron Microscopy and X-Ray Microanalysis, 4th Edition, Goldstein et al.

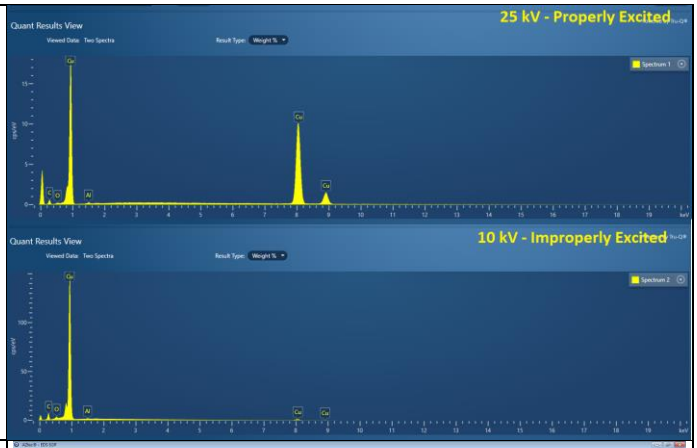
Required Equipment & Materials:

- SEM holders
- SEM mounting accessories

Steps	Key Points
1. Detector insertion (Helios) • Mount and load any samples as normal, with the addition of a small piece of copper tape (or silicon if you will be using an accelerating voltage too low to fully excite copper x-rays) in a blank spot on your mount. Bring the surface of the sample to the analytical working distance. • Insert the EDS detector using the "In" button. Be prepared to stop the detector in case of collision using the "Stop" button. • Pause the chamber view, it interferes with the detector.	

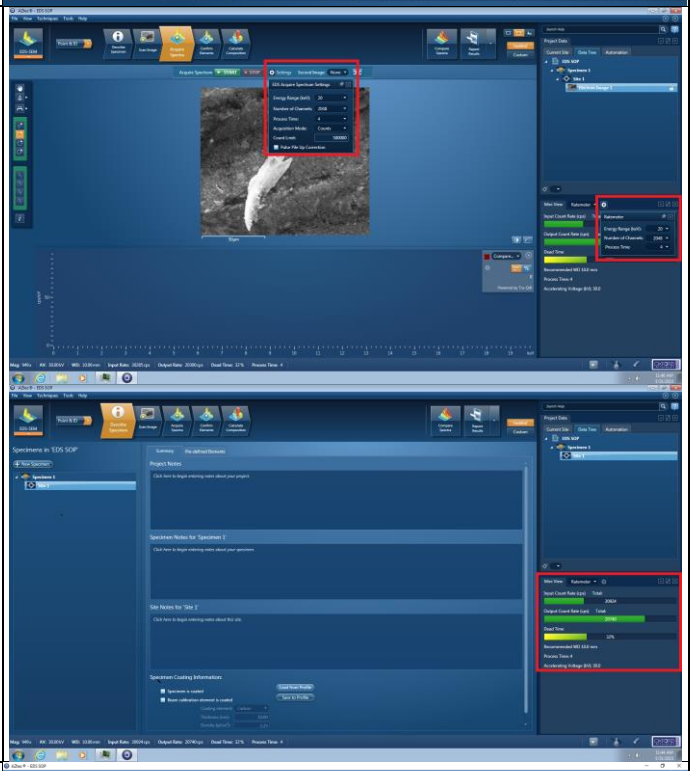
2. Accelerating voltage selection

   - The accelerating voltage of the electron beam should be at least 2.5x the energy of the peak(s) of interest in eV
   - The accelerating voltage should not be so high that the interaction volume is increased needlessly or that significant charging occurs.

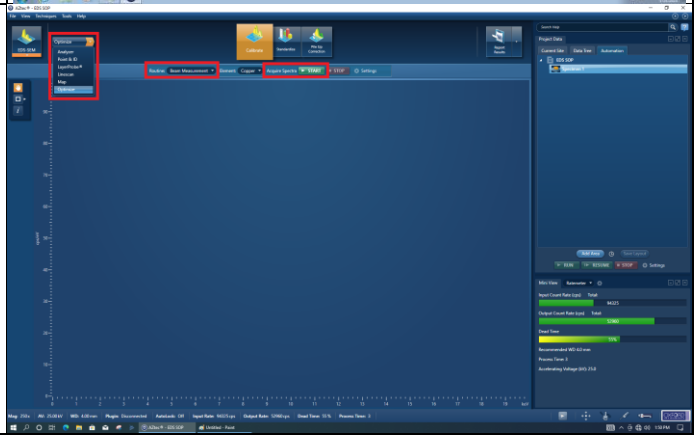


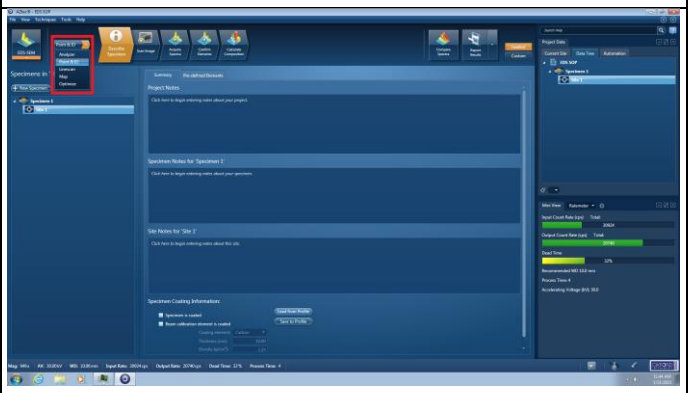
3. Process time & spot size/beam current selection

   - Increased process time creates high quality data, at the expense of lower count rates and higher dead time (& thus longer acquisition times to gather sufficient counts).
   - Increased spot size/beam current creates higher count rates but increases dead time (& thus lowers data quality).
   - Process time & spot size/beam current should be adjusted to provide optimal count rates and dead time for each sample (refer to individual training).



4. Beam Measurement Calibration
   - Open "Optimization" in the indicated dropdown menu.
   - Select "Beam Measurement" if it is not already selected.
   - Set your beam current/spot size and accelerating voltage to the settings you will be using for your sample.
   - Move the stage so that you are focused on the pure copper or silicon you have mounted with your sample.
   - Select the correct element based on



whether you are using copper or silicon to calibrate, then start the calibration by clicking "Start."	
5. Point scans, line scans, & maps • Point scans can be used to cover a specific point or region as a whole & are useful for gathering an average composition of a feature or region. • Line scans can be used to cover a line and are useful for examining composition changes with position. • Maps can be used to cover the points within a region & are useful for examining composition changes in two dimensions of position. However, maps can be significantly more time consuming.	
6. Detector Retraction • Do **NOT** vent the chamber before retracting the detector. Retract the detector using the "Out" button. Be prepared to stop the detector in case of collision.	

History of Revisions			
Revision Number	**Revision Date**	**Revised By**	**Reason for Revision**