ATAMI Standard Operating Procedure

Hitachi Desktop SEM

Last saved by Randy Greb on 12/31/2019 10:37 AM

Revision	Date	Description/Change	Curator
0	11/1/2019	New document	Randy Greb
1	11/7/19	Added example images with different detectors for training, improved info on EDX WD setup, more info on user file locations.	Randy Greb
2	12/16/19	Added additional sample size and stage position information	Randy Greb
3	12/31/19	Added quick start and shutdown guide per user request.	Randy Greb
4	6/18/2021	Added procedures for using standardless quantification with a subset of elements, and for labelling secondary peaks in spectra.	Randy Greb

Contents

Scope:

Operation of the Hitachi desktop SEM for imaging, EDX and 3d surface mapping.

System Specifications:

- SEM imaging with backscattered and secondary electron detectors.
- Fixed 5, 10, 15 kV accelerating voltage.
- Sample size up to 80mm in diameter and 50mm height.
- 40 x 35 mm XY stage travel. Tilt holders available.
- 3D surface profiling using 4 quadrant backscattered dector.
- Elemental analysis (spot, line scan, 2-d maps), using Brucker EDX detector.
- See ATAMI website and supplier documentation for further details.

Sample Size and Stage Motion Specs:

Maximum samples size in X/Y: 80 mm.

Maximum height: 30 mm with camera nav stub, 50 mm with standard stub.

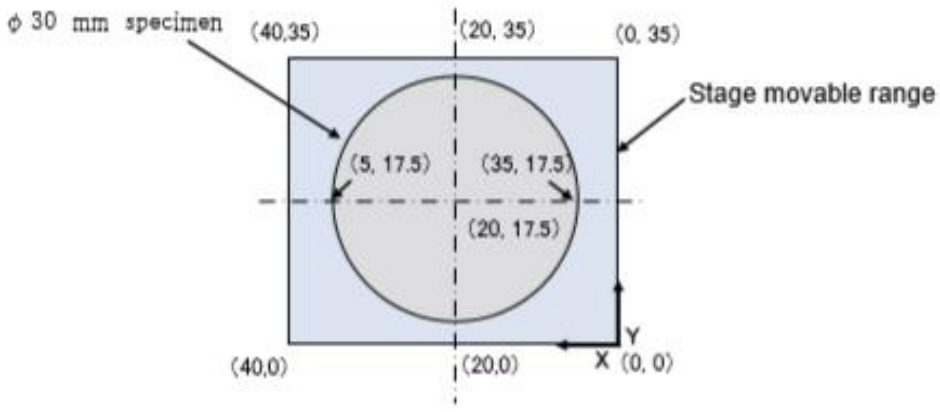
Optical image field of view: 25 mm with camera nav stub, 15 mm with standard stub.

Range of X motion: 0-40mm, **Range of Y motion:** 0-35 mm

SEM observation position for standard stub: X= 20 mm, Y = 17.5 mm

SEM observation position for camera navi stub: X = 16.5mm, Y = 17.5 mm

Optical camera position: X=40mm, Y = 17.5mm



Safety

General

Comply with all safety warnings posted on the tool.

The energy of the electron beam can cause heating and charging on a sample. Only analyze solid samples with the SEM.

Liquid samples should not be introduced into the SEM. Severe contamination and damage to the SEM can occur from liquids in the chamber.

Some solid samples may contain liquids that can explode in the chamber. If you are unsure about the suitability of your sample, contact ATAMI staff before you introduce it in to the chamber.

PPE Required

Standard ATAMI PPE for the lab you are in.

Nitrile gloves are required at all times when loading and unloading samples to prevent contamination of the chamber.

Hazardous Energies

Electrical: NA

Mechanical

Use caution with extended use. Take frequent breaks to avoid strain injury from observing the imaging screens.

Stored/Potential: NA

Thermal: NA

Materials/Consumables Hazard: NA

Interlocks

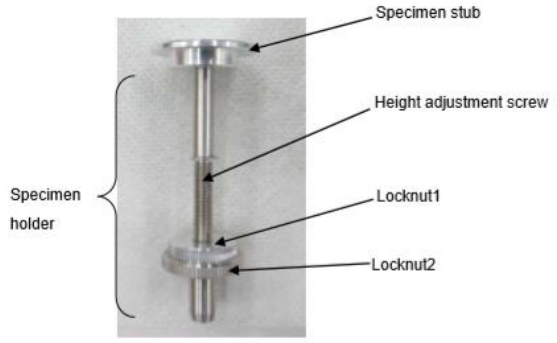
Never remove covers while equipment is in operation due to high voltage (15kV) present in the system.

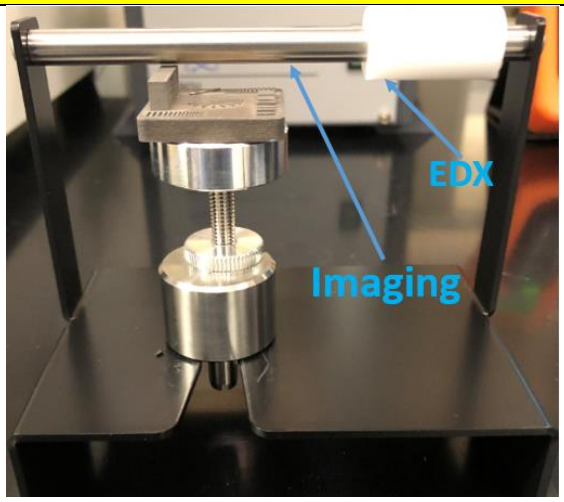
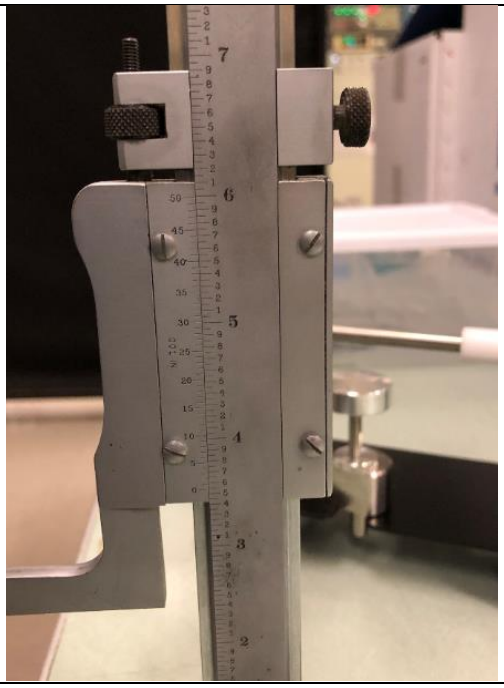
Training Requirements

1. Pass all ATAMI required safety courses
2. Finish lab tour with qualified ATAMI trainer.
3. Complete all hands on training for this system and signed off by trainer.
4. Verify access to this document for reference.

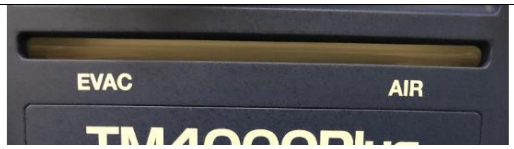
Procedures

How to mount a specimen on the specimen stub:

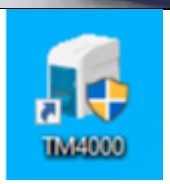
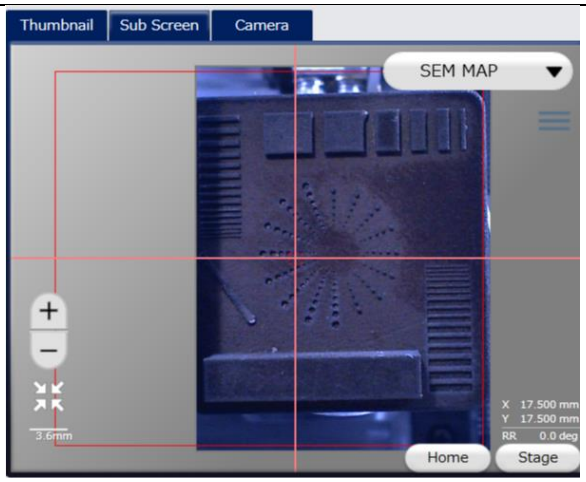
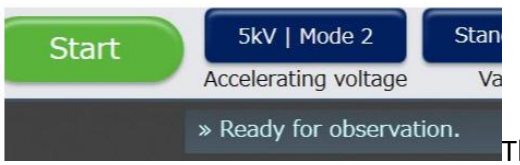
Step	Action	Notes
1	In general, samples will be attached to the top of the specimen stub assembly.	
2	Most samples can be attached to the top of the specimen stub using double sticky, conductive tabs or tape. **The maximum size of a specimen is 80mm across and 30mm high for the camera navigation holder, and 50mm high for the standard holder.** **Do not exceed that size.**	 
3	Attach the specimen stub to the specimen holder.	

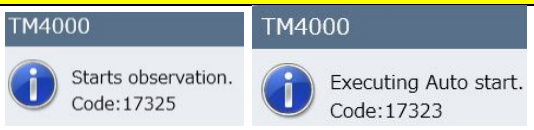
Step	Action	Notes
4	Adjust the specimen height using the height gauge (shown here). Short and long specimen height screws are available. Best image resolution and signal-to-noise is achieved at minimum allowed working distance, which is just at the bottom of the stainless steel bar as shown to the right. For EDX, use the specimen height gauge specified for EDX.	
5	Optimum EDX is around 10.4 mm working distance. This is roughly 5.539 inches with the Vernier shown to the right.	

How to start the system and load a sample into the chamber:

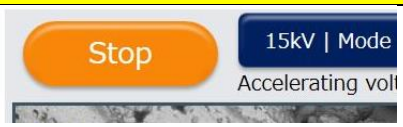
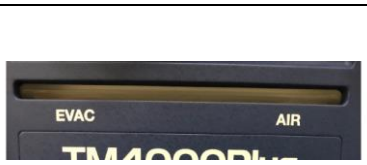
Step	Action	Notes
1	Turn on the power switch at the right side of the main unit.	 You may see some stage initialization messages during startup.
2	Be sure to mount the specimen to the correct height using the procedures shown above. Also be sure that the specimen size is within system limits (< 80 mm across, < 40mm height).	
3	If the EVAC LED (blue) is on, press the EVAC/Air switch and wait until the chamber is at atmosphere.	 
4	Wait approximately thirty seconds after AIR LED (white) is solid on (not blinking), hold the handle and slowly pull out the specimen stage.	

Step	Action	Notes
5	Insert the specimen stub which has already been adjusted to the correct height for either imaging only or imaging and EDX.	
6	Tighten the hexagon screw to hold the sample. In case of high magnification imaging or a heavy sample….????	
7	Insert the specimen stage. Make sure the specimen doesn't rub against the top plate shown to the right. If it does, be sure to recheck specimen height.	
8	Evacuate air from the specimen chamber by slightly holding the specimen stage and pressing the EVAC/AIR switch.	When the evacuation is completed, the EVAC LED (blue) will be turned on solid. It will blink when it is pumping down the system.

Step	Action	Notes
		
9	Log in to the PC and if the SEM software is not already started, go ahead and start it with the TM4000 icon from the desktop.	
10	The system will move the sample to the optical image camera and collect an image. If you use the specimen holder for Camera Navigation, the image field of view will be 25mm radius, with the standard holder, the field of view is 15mm.	 
11	When the system is fully evacuated, you can push the start button to start collecting the SEM image.	 The stage will move to the SEM imaging position and you will see a couple of status messages.

Step	Action	Notes
		
12	Refer to the procedures below for hints about how to use the various imaging conditions of the SEM.	

How to unload a sample and shutdown the system:

Step	Action	Notes
1	Press the Stop button to shut turn of the electron beam.	
2	Press the EVAC/AIR button to evacuate the chamber.	
3	Wait approximately thirty seconds after AIR LED (white) is solid on (not blinking), hold the handle and slowly pull out the specimen stage.	
4	After removing the sample, press the door closed again and start evac with the EVAC/AIR button.	The EVAC LED will start blinking blue.
5	Close the Hitachi and Bruker software applications.	
6	Put the PC to sleep and then logout of the access control box.	
7	Turn off the Bruker EDX controller.	
8	After the EVAC light has turned to solid blue, you can turn off the SEM with the power button on the right.	

Quick How to:

Start the System:

1. Press Power button on the side of the chamber.
2. Press EVAC/AIR button and wait until the AIR LED is solid (not blinking).
3. Load the sample.
4. Start the Hitachi SEM application.
5. Choose which specimen holder you are using.
6. Once the EVAC LED is solid blue, you can press the Start button in the Hitachi app to start imaging.
7. Turn on the EDX processor box and start the EDX application if you are doing EDX.

Shutdown the System:

1. Press Stop to stop imaging.
2. Shutdown the EDX software and turn off the EDX processor.
3. Shutdown the SEM software.
4. Press the EVAC/AIR button.
5. Wait until the air LED is solid, and then wait an additional 20 seconds.
6. Unload your sample.
7. Close the chamber door.
8. Press the EVAC/AIR button.
9. Wait until the EVAC LED is solid blue.
10. Power off the SEM.
11. Put the computer into sleep.
12. Log off of the card reader.

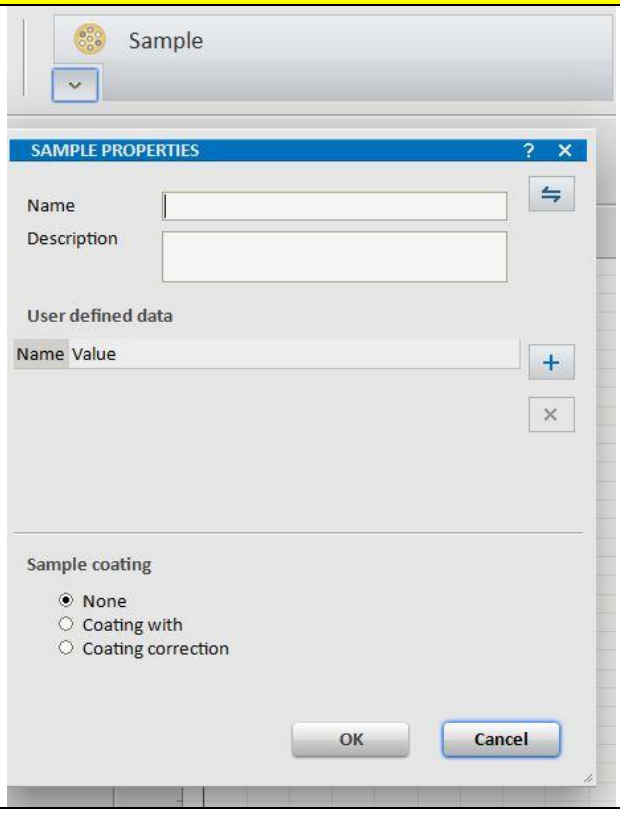
Image Collection Procedures:

After initial training, please refer to sections 2.0 and 3.0 of the Hitachi manual for directions for using the SEM.

All images will be saved in the "C:\Users\Public\Pictures\SemImage" folder by default. These images can be deleted by the next user. Please copy all image data to your own user folder in "C:\SEM_EDX_data\Atami" when done. See below for folder information and naming conventions.

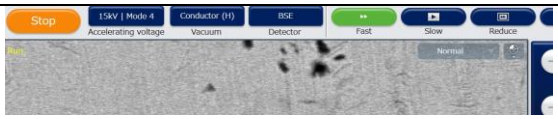
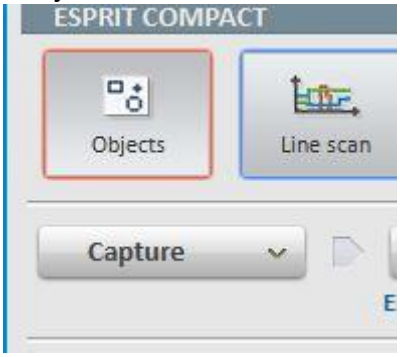
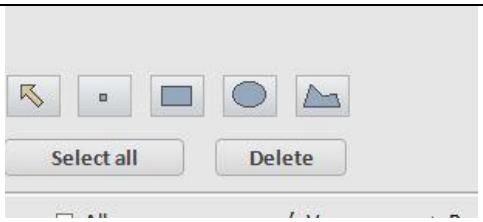
Applications guides which describe how specific settings can be used to collect different types of information from the SEM are shown below.

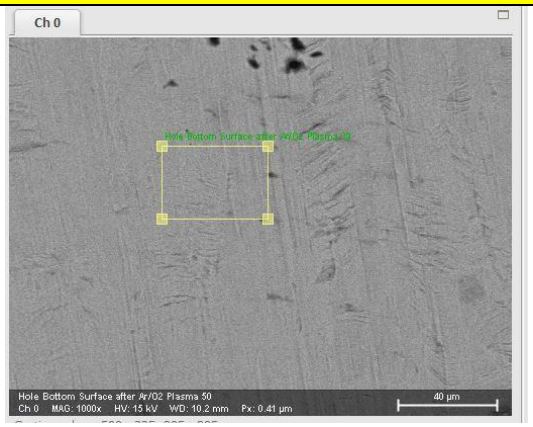
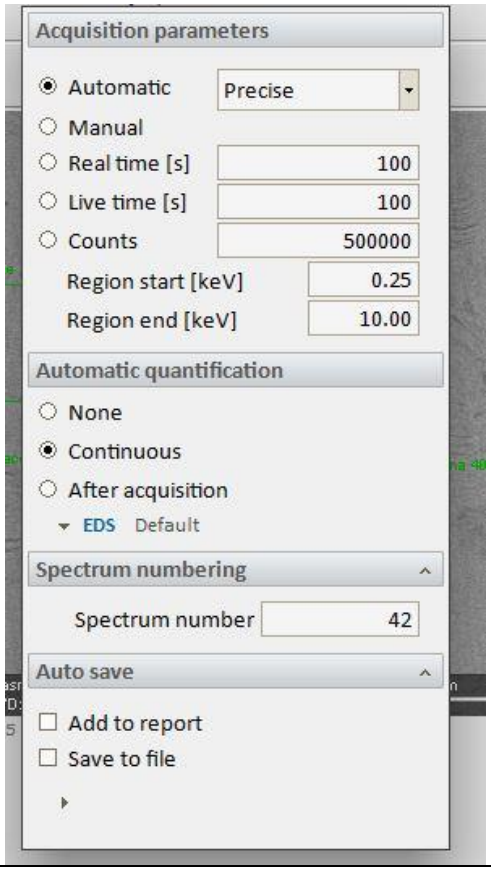
Set up your sample information before starting EDX data collection:

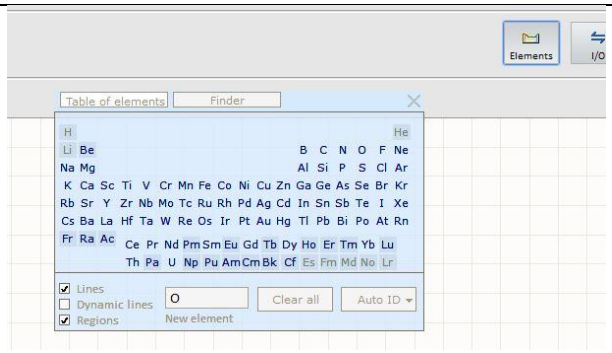
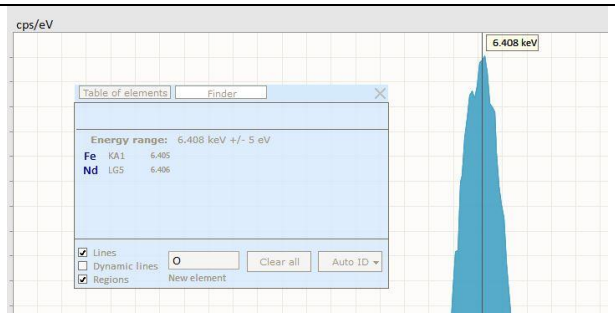
Step	Action	Notes
1	Press the Sample drop-down. You add sample Name and descriptions here that will carry through to reports. Most of the time sample coating should be none. If you coat with Au, use the coating with button.	
2		

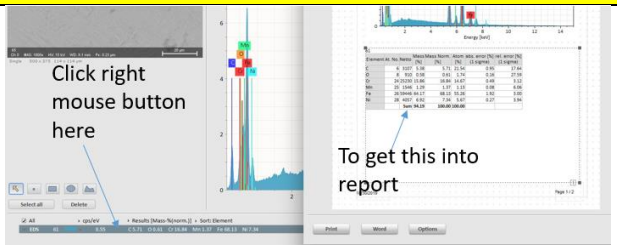
How to collect an EDX Spectrum:

Step	Action	Notes
3	Be sure that the sample is set to the EDX working distance, not optimum imaging distance	Count rates will be too low if the sample is not at the optimum imaging distance.

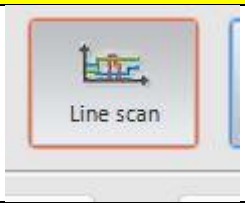
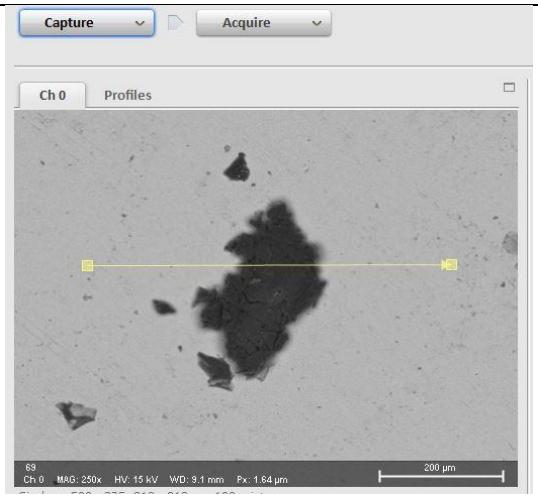
Step	Action	Notes
4	Open the EDX application with the Bruker ESPRIT Compact icon.	Username and Password are both "edx" 
5	Make sure the Hitachi SEM software is running and the beam conditions are suitable for EDX. See the table in the attachments below for characterization of beam energies vs count rates on a stainless steel sample.	
6	The beam needs to be turned on for EDX. It's typically easiest to leave it on "Fast".	
7	Press the Objects box to get EDX spectra.	
8	In the ESPRIT COMPACT EDX application, press "Capture to acquire an image. You can adjust imaging conditions with the down arrow.	The EDX system will now take over scanning, and the image on the SEM application screen will just look like random noise. 
9	Use one of the selection tools to determine the region to collect a spectrum from and draw it on the captured image.	

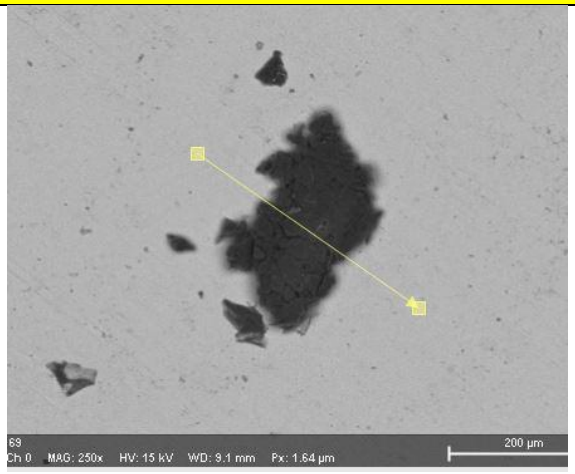
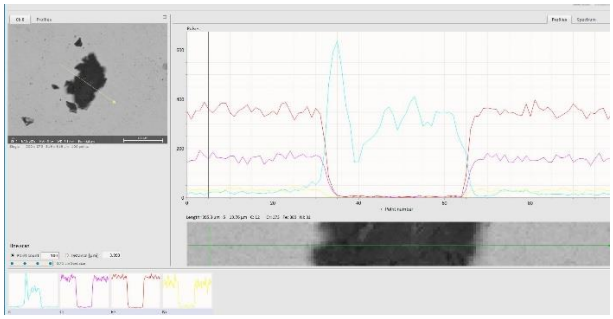
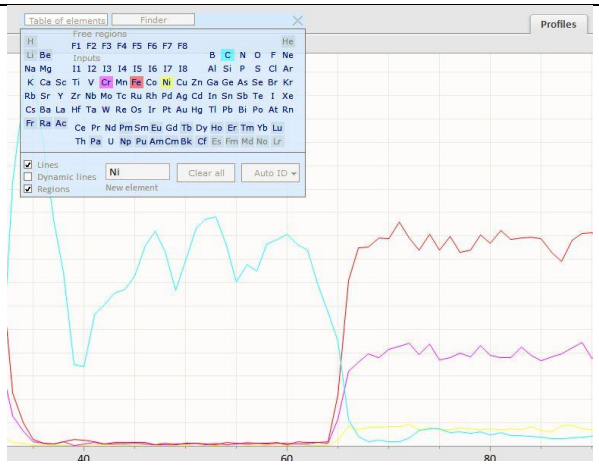
Step	Action	Notes
		
10	Press Acquire to get an EDX spectrum from the region identified in the image.	The Acquire button will change to stop.
11	You can turn automated element labelling and quantification on and off in the drop down menu.	Generally "precise" is a good choice for acquisition parameters. But you can also customize the time for the acquisition. 
12	Once a spectrum is collected you can zoom in and out of the X-axis with the mouse scroll button. You can also move the X-axis right and left, and expand and contract	

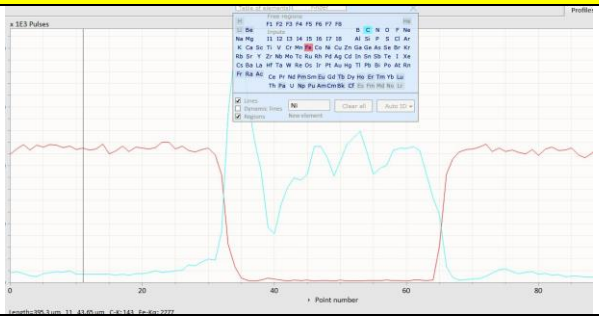
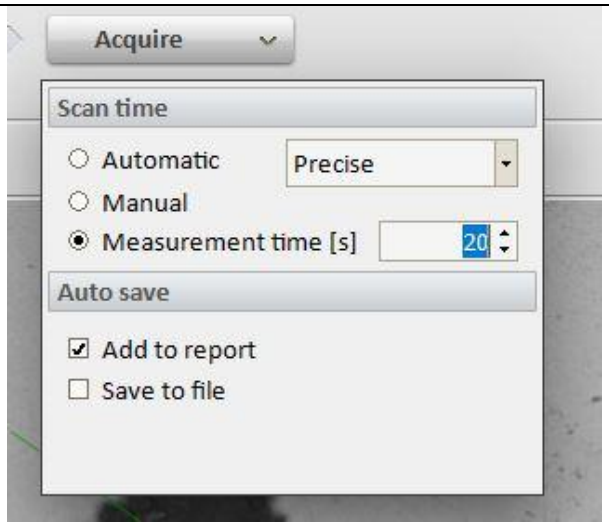
Step	Action	Notes
	the Y-axis with the mouse and left button. If you want to go back to the start, right click and select "automatic scale".	
13	If you have continuous quantification turned on, then you will see element labels for the peaks in the spectrum. If not, you press the Elements button to do labelling, or press the Quantify button.	
14	When you press the elements button, you can manually label peaks, clear peaks, and use Finder to locate element labels that are closest to the peaks of interest.	
15	To use Finder, place the cursor over the peak of interest and press the Finder button. This will list x-ray lines with the highest probability of matching the peak of interest. You can use the left/right keys to move by small increments	
16	When you click on the element you believe matches the peak, the label will be added to the spectrum and the list of labelled elements in the Elements pop-up window.	
17	Click right to save the spectra. Navigate to the User directory (C:\SEM_EDX_data\Atami) and save the report to your	
18	To build a report from the various objects, click right in the spectrum, image or quantification table to add to the report.	The I/O button opens and closes the report writer.

Step	Action	Notes
		
19	Press Print to print to a PDF file. Press Word to send to a Microsoft Word file.	Save reports to your user directory in C:\SEM_EDX_data\Atami.

How to collect an EDX Line Scan:

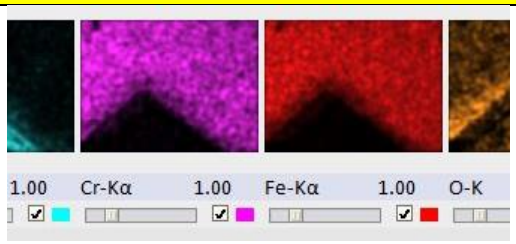
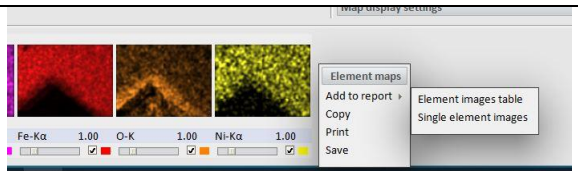
Step	Action	Notes
1	Press the Line scan box.	
2	Use the SEM software to get a good image of the object of interest. Leave the SEM in Fast mode.	
3	Press Capture to get an image in the Bruker EDX software.	

Step	Action	Notes
4	Then you can move the line around to cover the object of interest.	
5	Press Acquire.	The software will start acquiring a spectrum across the whole line, and once elements are labelled it will start producing the line scan, as shown. 
6	Press Acquire again if you want to stop the line scan collection.	
7	If you only want a subset of the elements, you can select and un-select highlighted elements in the Elements box.	

Step	Action	Notes
		
8	You can drag the cursor to see the specific counts for each element at each pixel on the line.	
9	You can right-click to save components of this display to the report. You can also select "Add to report" in from the acquire drop down menu to automatically save it.	

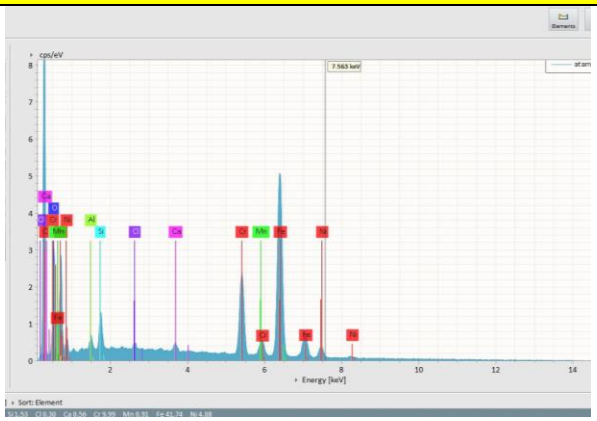
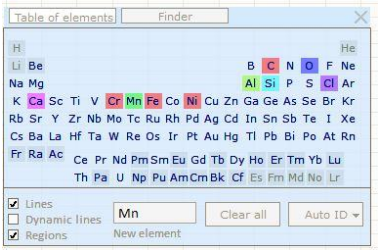
How to collect an EDX Dot Map:

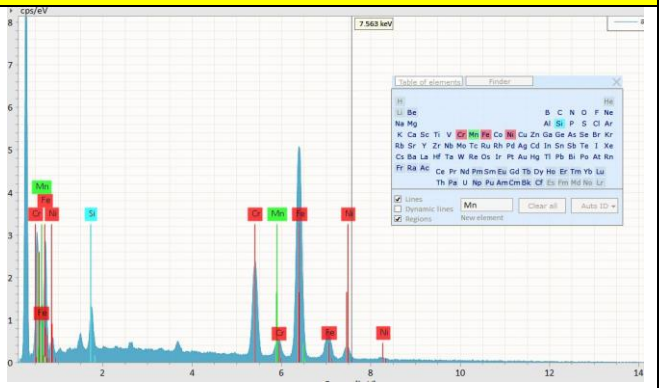
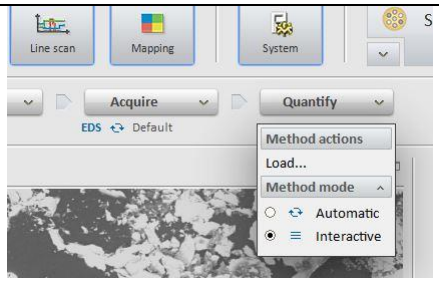
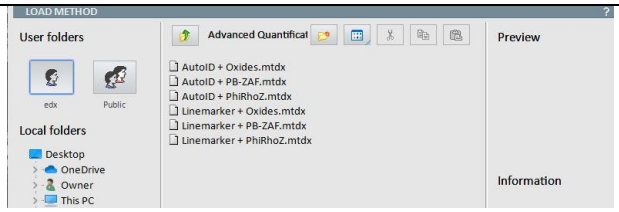
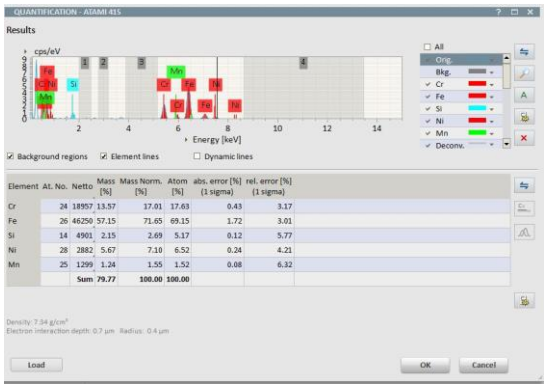
Step	Action	Notes
1	Press the Mapping box.	
2	Use the SEM software to get a good image of the object of interest. Leave the SEM in Fast mode.	
3	Press Capture to get an image in the Bruker EDX software.	
4	Draw a box around the area of interest.	
5	Press acquire.	Auto-ID and selecting elements works the same as with line scans.

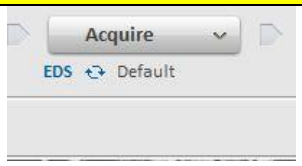
Step	Action	Notes
6	You can also select and un-select elements to be displayed in the maps with these checkboxes.	
7	To save an array of the elements maps to the table, you can right click in the image array and choose "Add to report -> Elements images table"	

How to limit quantitative results to a subset of elements in the spectrum:

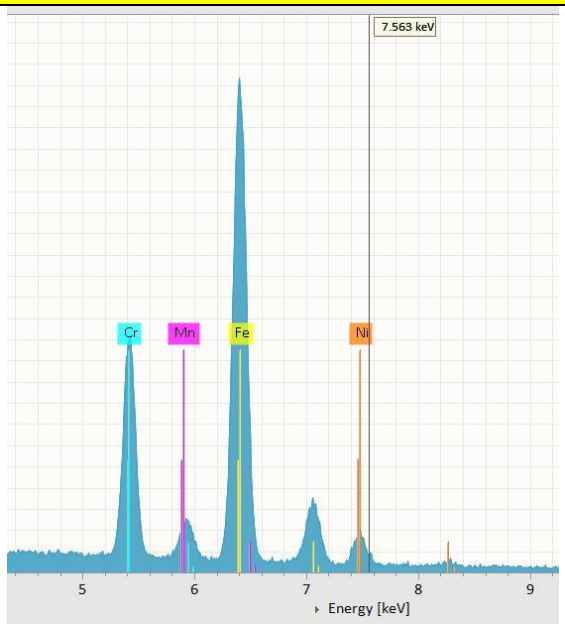
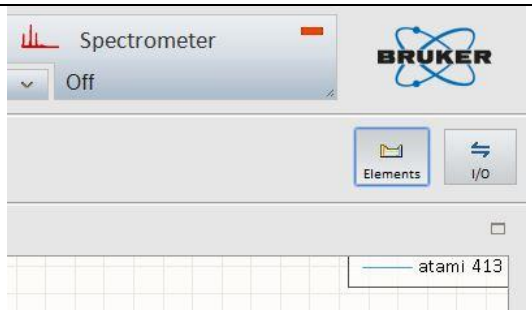
If you have spectrum from particles or other small features which may have background elements, you may want to quantify with a subset of elements that you believe are only present in the particles.

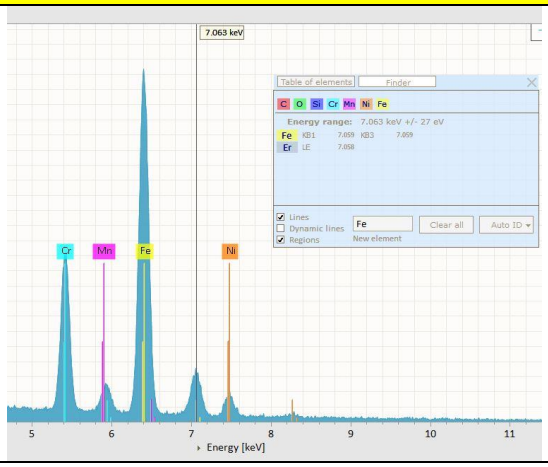
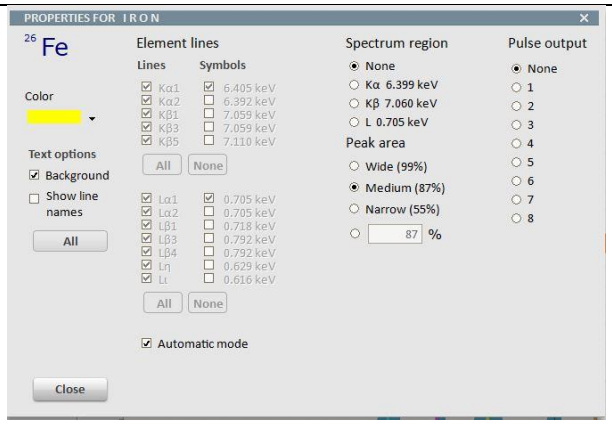
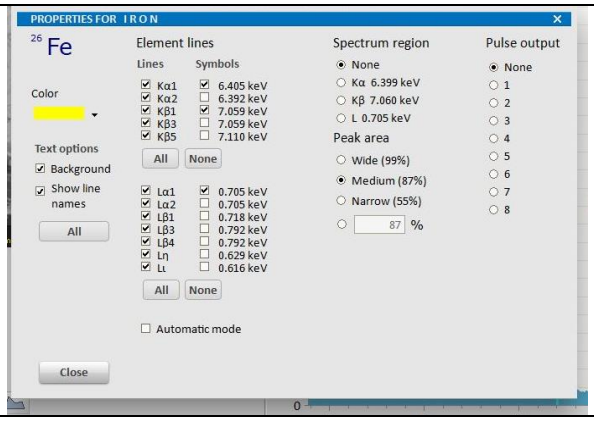
Step	Action	Notes
1	In this spectrum we see some elements such as O, Al, Cl, Ca, and a very large C peak that we don't want to include in the quantification.	
2	Open the Elements menu and click highlighted elements that you do not want to include.	This will eliminate the labels and peak line from the spectrum. 

Step	Action	Notes
		
3	Click the down arrow on the "Quantify" button and choose "Load..." under "Method actions".	
4	Double click on "Advanced Quantification Methods" and choose either "**Linemark + PB-ZAF.mtdx**" or "**Linemark + PhiRhoZ.mtdx**". The PB-ZAF method is most commonly used for standardless quantification.	
5	Press quantify, and the quantification will be re-done with the new method and normalize for only the elements included.	 
6	You can then save the quantification results as normal.	

Step	Action	Notes
7	For initial peak labelling, it's recommended to use "EDS - default"	

How label secondary peaks that are not labelled as part of AutoID:

Step	Action	Notes
1	In the example shown to the right, the un-labelled peak shows and Fe line, is Fe Kb peak, but it is not labelled. How do I label this?	
2	Move the cursor to the peak.	
3	Press “Elements” to get the Peak ID window.	

Step	Action	Notes
4	Click "Finder". Then move the cursor back and forth until the best choice of peaks is shown. Choosing the peak is based on experience and which element best matches the location and the sample materials.	
5	If the element is not highlighted with the color that matches the lines in the spectrum, left click on it, and it will assign an ID to that peak.	
6	If the peak is has and element line associated with it, but not a label, right click on the element of interest and it will bring up the "properties for ..." window.	
7	Uncheck automatic mode, and then check the box under "Symbols" that matches the peak you have identified. You can also select "Show line names" if you want the line names to show up in the spectrum as shown	
8	After you press close, the element properties will stay this way for subsequent spectra collected.	

Standard or Example Recipes

N/A:

Basic Troubleshooting

Various Issues that might come up:

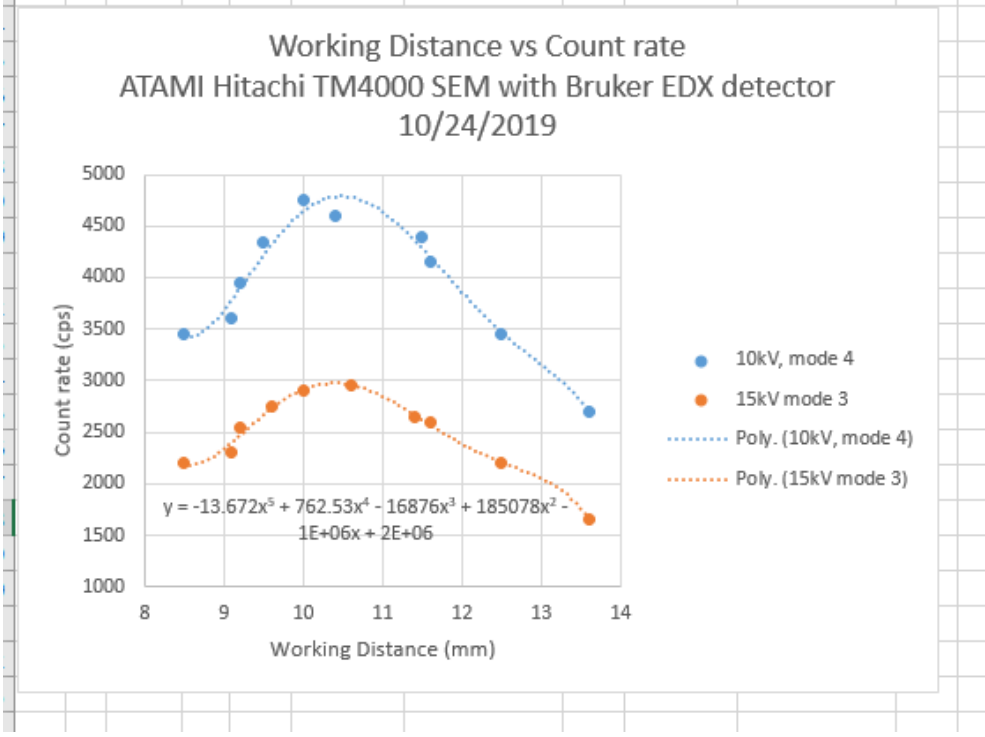
Step	If	Then	Notes
1	Have excessive charging on your sample.	1. Try changing the vacuum mode to M or L. 2. Try changing to a faster scan rate. 3. Try lower beam energy and current (for example go from 15kV-4 to 10kV-2). 4. Try coating your sample using the Cressington gold coater.	
2	Can't find your image files.	Refer to the standard image locations noted below.	
3	Get low EDX counts.	1. Make sure you are at the right working distance. 2. Make sure you have enough beam energy (kV) and beam current (1-4) for the elements of interest.	As a general rule of thumb, you should ideally use >/=2X the energy of the element of interest. For example, to look for the Fe K-alpha peak at 6.4eV use a beam energy of 15kV on this SEM.
4	You need to get better resolution	1. Go to a lower beam current (range is 1-4) 2. Try a closer working distance. 3. Try a higher beam voltage 4. If none of the above works you can try more advanced methods, see notes to the left.	Depending on the sample, "resolution" can be a function of spot size, contrast, beam voltage, charging, material contrast, surface roughness …. Sometimes a slightly larger beam, with better S/N will give better resolution. You'll need to experiment.
5			

Attachments

Count Rate vs Working Distance and beam conditions for EDX :

- Optimum working distance for EDX with flat samples is roughly 10.2-10.4 mm.
- H vacuum mode generates roughly 200-300 more cps for equivalent conditions vs M vacuum mode.
- The table below shows some count rates for different conditions.
- This is on a flat piece of stainless steel.
- One full turn (360 deg) on the sample holder equals roughly 1mm of working distance change.

						10kV, mode 4			15kV, mode 3	
WD	kV	mode	vac	cps		WD	cps		WD	cps
9.1	5	3	M	500		8.5	3450		8.5	2200
9.1	5	4	H	1950		9.1	3600		9.1	2300
9.1	5	4	M	1550		9.2	3950		9.2	2550
9.1	10	3	M	1100		9.5	4350		9.6	2750
9.1	10	4	H	3600		10	4750		10	2900
9.1	10	4	M	3100		10.4	4600		10.6	2950
9.1	15	2	M	550		11.5	4400		11.4	2650
9.1	15	3	H	2300		11.6	4150		11.6	2600
9.1	15	3	M	2030		12.5	3450		12.5	2200
9.1	15	4	M	6400		13.6	2700		13.6	1650



Characteristics of beam condition settings:

Observation setting dialog is displayed by right clicking the mouse at the observation mode button. Detail setting of accelerating voltage or current can be carried out.

☞ 2.2.3.3 Observation mode setting

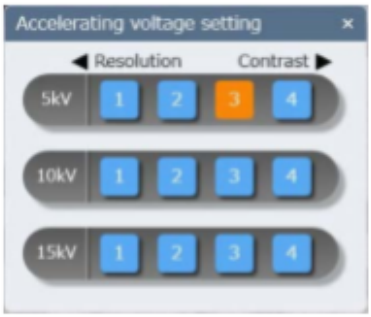


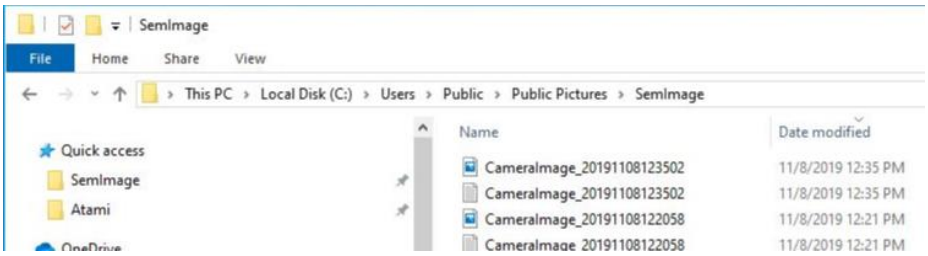
Figure 3.3—2 Observation mode setting dialog

Accelerating voltage can be selected from 5kV, 10kV, or 15kV. Each accelerating voltage can be selected by clicking a setting button from 1 to 4 which are displayed at each frame of accelerating voltage value. Feature of current setting is described as follows.

Current setting	1 High resolution	4 High brightness
Electron beam size	Small ⟷	Large
Contrast	Low ⟷	High
Noise	Low ⟷	High
Charging phenomenon	Low ⟷	High
Damaging a specimen	Low ⟷	High

Location of User Files and Naming Conventions:

Short cuts to the two main folders used in this system are in the Windows Quick Access menu:



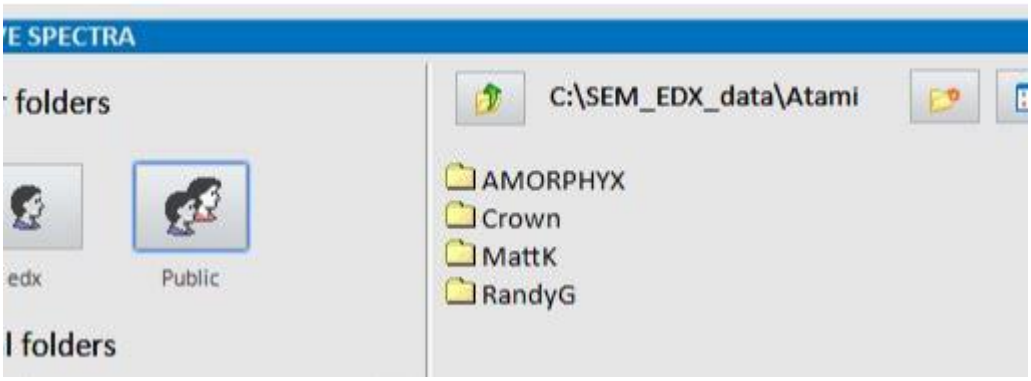
Default location for SEM to save image file: C:\Users\Public\Pictures\SemImage

Location for user SEM image files and EDX reports: C:\SEM_EDX_data\Atami

Naming convetion for user folders in the SEM_EDS_data\Atami folder is:

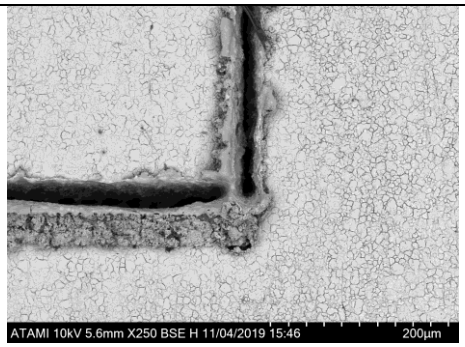
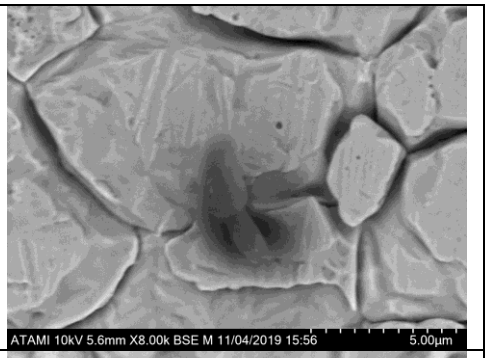
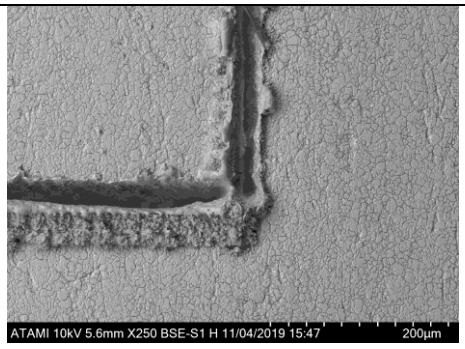
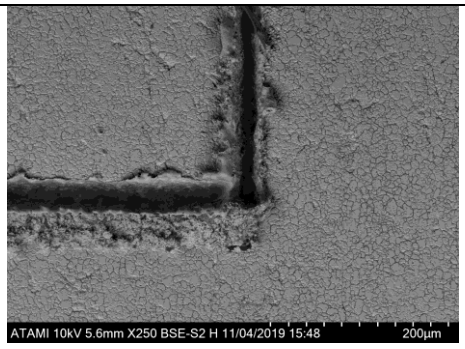
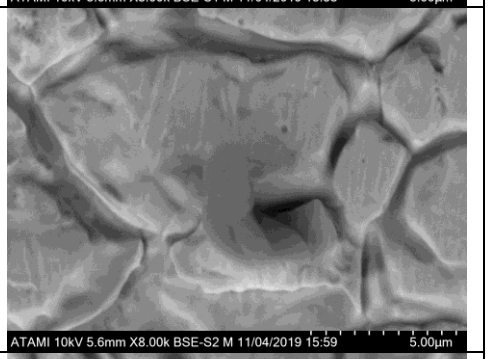
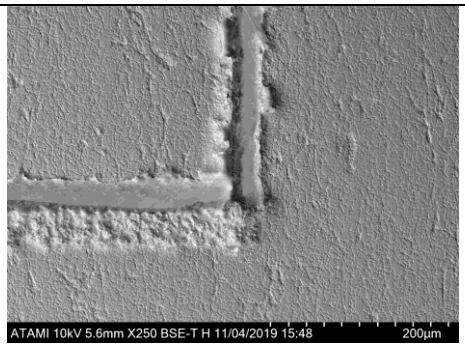
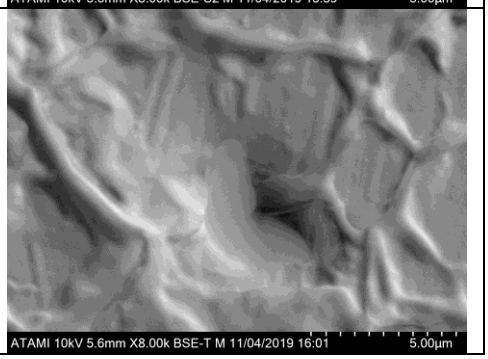
For personal folders, use Firstname and first initial of last name like – GeorgeW or PaulM

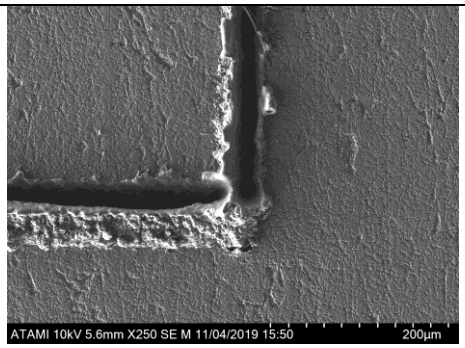
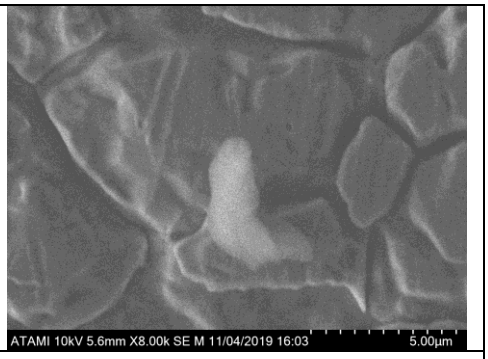
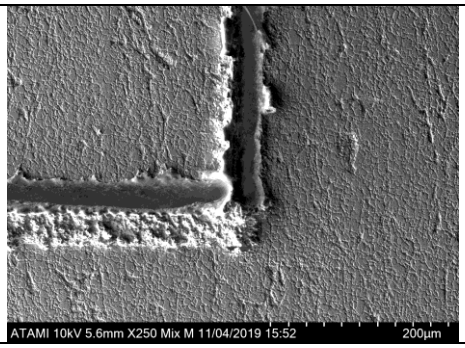
For company folders, use the company name, and then organize folders as needed within there.

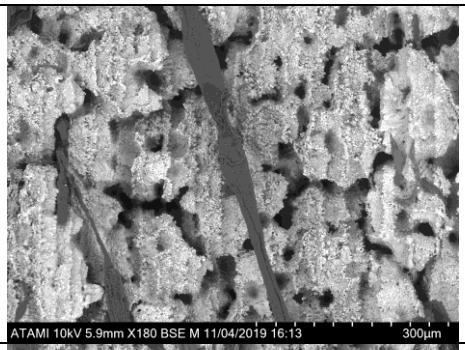
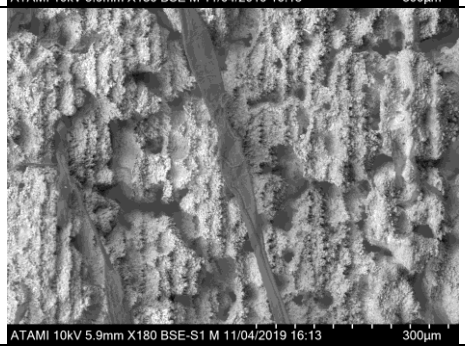


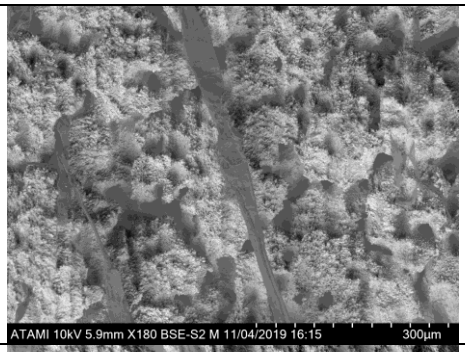
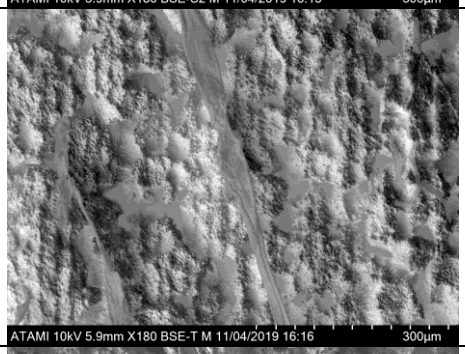
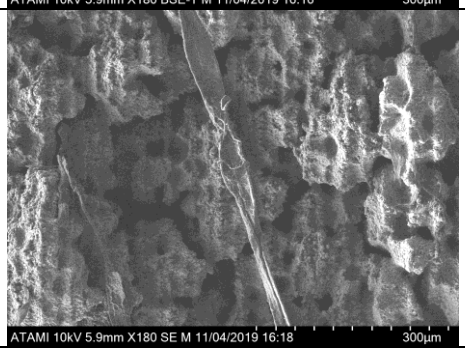
Images showing different effect of different detector settings:

These images give examples of how the image changes with different detector setups. BSE normal is best for high res imaging.

BSE normal		
BSE shadow 1		
BSE shadow 2		
BSE topo		

SE		
Mix		

BSE normal		
BSE shadow 1		

BSE shadow 2		
BSE topo		
SE		
Mix		