Phenom XL G2 SEM SOP

Located in Main Lab (176)

TO: Gerald Harris (gharris75@gatech.edu) and Victoria Pozzi (vpozzi3@gatech.edu)// MILL Wiki Article

Last updated May 21st, 2025

Primary Use

The Phenom XL G2 Scanning Electron Microscope (SEM) provides users with high resolution electron microscopy images in just a few minutes. Users can load many sample stubs into the sample holder and navigate around each sample in the Phenom User Interface. Features include mixed SED + BSD imaging, automated image mapping/stitching, returning to saved positions on your sample, and EDS/EDX (Energy-dispersive X-ray Spectroscopy), allowing for quick elemental identification and EDS mapping. Data may be retrieved using a USB-A stick.

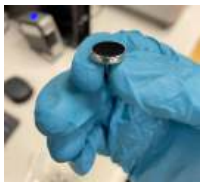
Operating Principle

SEMs use an electron beam to image materials and resolve features at great magnifications. The Phenom XL can use a variable accelerating voltage between 4.8kV and 20.5kV, has a magnification range from 160x to 200,000x, and contains both a secondary electron detector (SED) and a 4-quadrant backscattered electron detector (BSD). These two detectors provide topographical features and Z-contrast (atomic number/mass contrast) respectively, and the signal ratio from each detector can be mixed in a custom ratio to suit your samples.

Safety Procedures

The SEM poses little risk to the user, as it has many built-in safety features, such as automatically closing the chamber door if left open for too long. When the door is closed and the SEM is in use, the locked symbol (above the chamber door) will turn orange. When it is open, the unlocked symbol will turn green. Samples must be free of dust and moisture, powders must be firmly attached using colloidal graphite (in the flammables cabinet), and magnetic samples are NOT allowed. The highest point on any sample must also be 6 notches below the rim of the sample holder before loading into the SEM (or more if using tilted sample stubs). The vacuum of the system needs maintenance every 2 years (next is due June 1, 2023). An appointment needs to be scheduled with Phenom support once 1200 source hours are reached. Source hours can be viewed in Settings>Phenom>Status in advanced mode. Password for advanced mode is “expert”.

Sample Prep



1. **WEAR GLOVES!**
2. For non-conducting samples, consider using our sputter coater, unless you are also performing extensive EDS.
3. Samples such as concrete or rocks must be thoroughly degassed.
4. Retrieve a fresh aluminum SEM stub (Ted Pella Sample Mounts) and apply a circle of carbon tape black-side down (Ted Pella Pelco Tabs). Remove paper from the tape.

5. Firmly press your sample on the stub
6. Thoroughly dry/blow off dust from the sample stub using the compressed air gun.

General Procedure

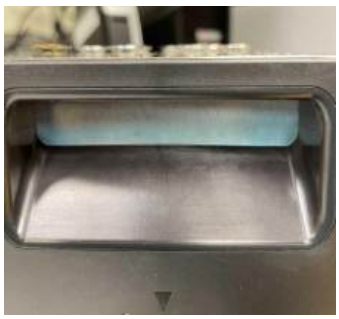
Launching Software

- Lenovo Thinkstation Computer should already be on, (if not??)
- Launch Phenom User Interface (on Desktop)
    - Windows + D to return to desktop
- Prepare your sample externally
    - See **Sample Prep**

Loading Sample (must wear gloves)

- **Make sure the stage is always on a clean surface, place it on a kimwipe before putting it on the table**
- Sample holder should be inside the SEM, click the “Unload sample” or eject button in the top left to open the SEM

- Remove the sample holder **while wearing gloves**
- Rotate knob to the right (CCW) to raise the stage to maximum height
- Insert your sample stub(s) in an empty hole using stub tweezers
- Rotate knob to the left (CW) so that the top of the “tallest” sample is flush with the rim of the sample holder. Then further lower the stage by 6 notches, which are indicated on the knob.

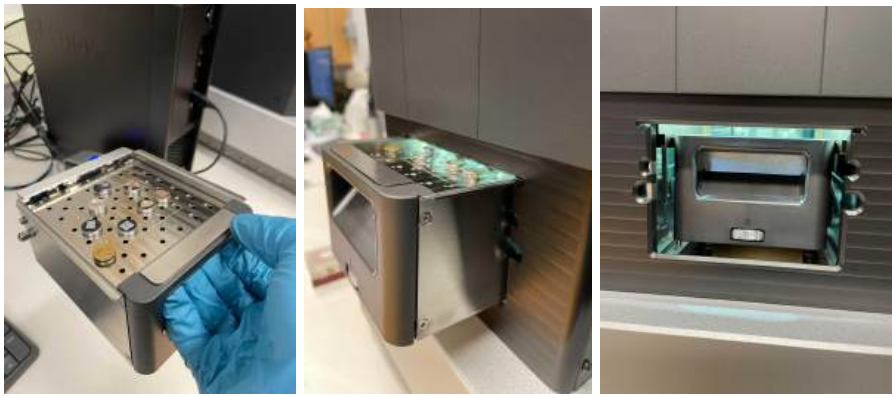


Maximum Height

Sample is flush

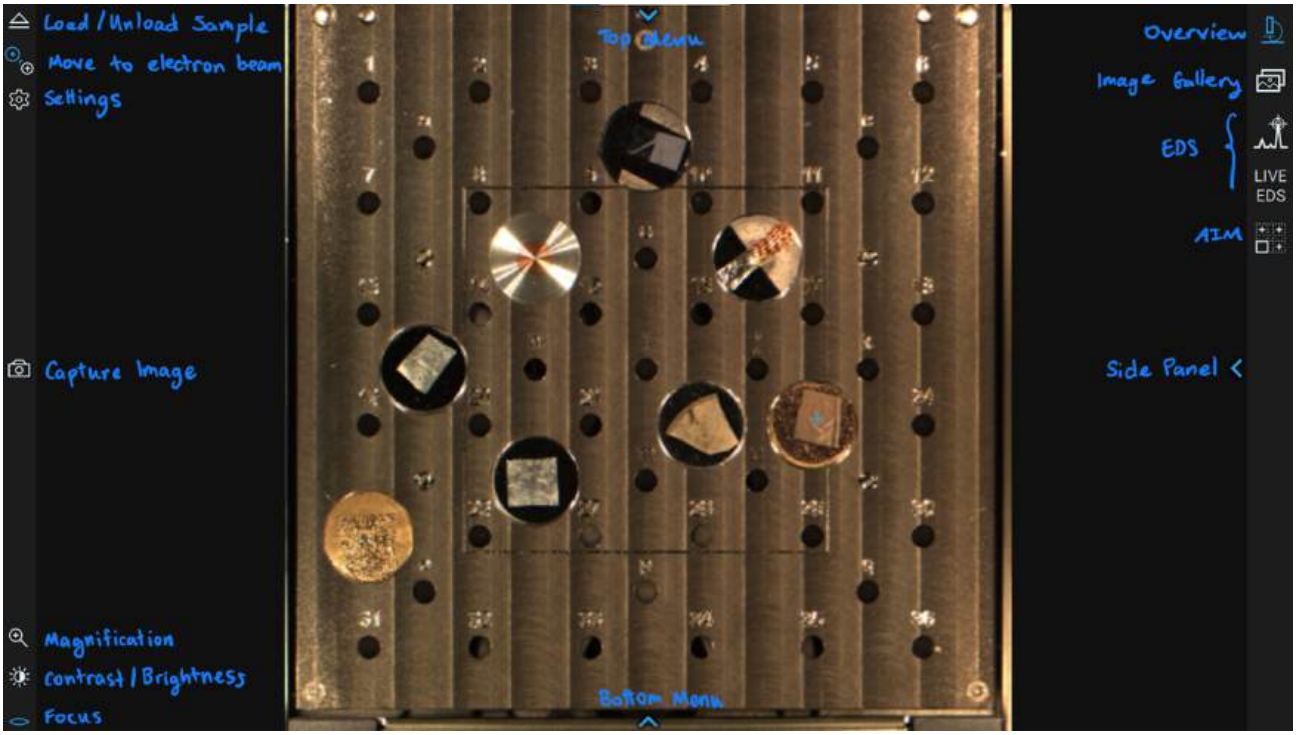
6 notches below flush

- Gently load the sample holder until it is fully inside the SEM. Note that the edge of the sample holder going into the SEM should slope slightly downwards while loading—do NOT force the holder into the SEM.



Grab sample holder and gently slide into the SEM until it stops

- Click the “Close door and move to NavCam” button (same eject as before)
- Allow the system to pump down.
- You will be directed to an optical image of the sample holder. Left-click on your sample to select the spot to focus the electron beam (e-beam) and click the “Move to SEM” button (directly below the eject button).



Imaging Toolbar (left side of screen) 

- A = auto, F = fine
- Left clicking on an icon with an “A” will perform Auto Source Tilt, Auto Stigmation, Auto Contrast/Brightness, or AutoFocus
- To adjust from Auto (A) to Manual, right click on an icon to select the preferred mode
- Click the scroll wheel or left-click the selected icon again to toggle between coarse and fine adjustments (or contrast/brightness)

- **Rotate** (coarse and fine): scroll to rotate the image (detector)
- **Source Tilt** *[optional: auto Source Tilt is good]*: either click on the grid or scroll to adjust X and Y tilt individually
- **Stigmation** *[optional: auto Stigmation is good]*: either click on the grid or scroll to adjust X and Y tilt individually
    - Magnification must be > 40kx, must center on a round feature. Optimize contrast and brightness before adjusting.
- **Magnification** (360x – 190kx): scroll to adjust (in magnification mode) or click and drag area on the image to zoom to that area (does not have to be in magnification mode)
- **Contrast/Brightness**: scroll to adjust, histogram in side panel

Contrast Icon

Brightness Icon

- **Focus**: scroll to adjust, click scroll wheel to go between coarse and fine focus. You can focus on the selected region by right-clicking the icon ("Focus in selected area").
    - Left click and drag box to move, resize region by dragging corners. Scroll to adjust focus.
    - When not in focus mode, right-click and drag left-to-right to adjust focus. (right = greater focal length)

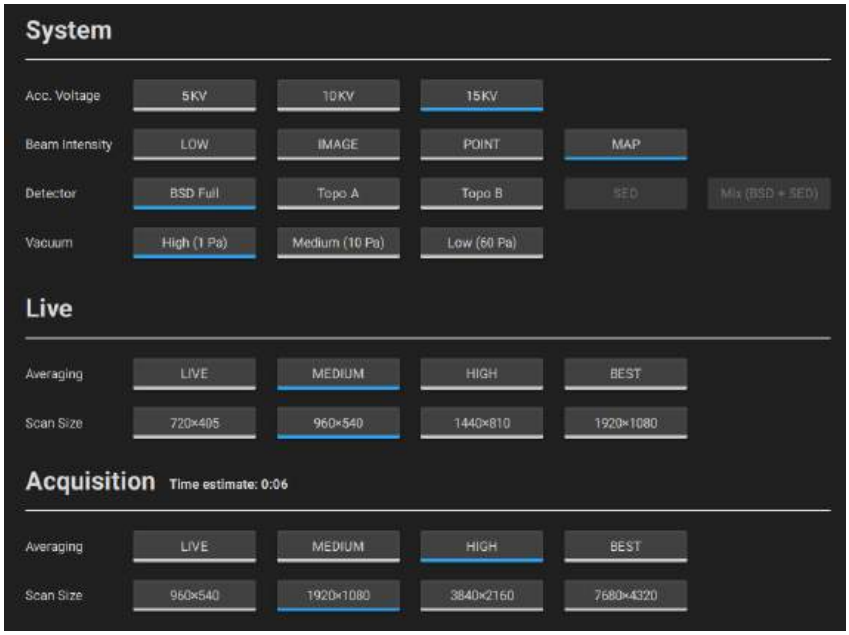
- Hovering over the camera icon will hide menus and display the image to be captured. Click to capture image.
    - You can change the name and location to which your images save in Settings Icon (top left) > Customize > Acquisition
    - The databar will automatically save with the image, but you can toggle it in settings as well.
    - Images can be exported as .tiff, .jpeg, or .png. Note that .png files will NOT save the image's metadata and therefore cannot be used in Phenom's analysis software. (We recommend using .tiff images!)
- Other
    - Gallery (right toolbar, second from top)

        - Displays image metadata for selected image, can also return to the same position where the image was taken
        - View images and the directory in which where they are saved
        - *[currently bugged, use PhenomImageViewer or another software such as ImageJ]* Click on the large image to add measurements and labels—after adding, click the hamburger menu to export the image
    - Ctrl + Click and Drag on the live image to draw a *temporary* measurement line.

Menus

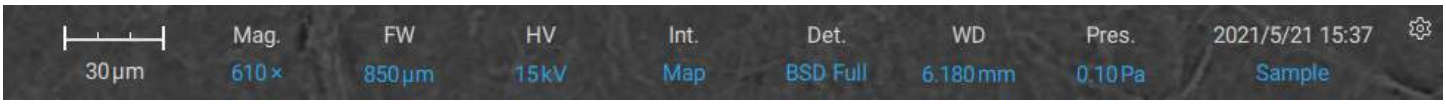
- Top Menu/System Settings: used to adjust live image and acquisition settings
    - Acquisition section displays estimated acquisition times for the selected settings
- Side Panel: used to navigate on NavCam overview, adjust imaging controls, and return to a saved position with specific settings (use "refresh" arrow).

- Can customize other sidebar features in settings. (includes contrast/brightness histogram)



Top Menu/System Settings *Side Panel*

- Bottom Menu/: Displays live image parameters and settings, including:



  - Scale bar
  - Magnification
  - Field Width (FW)
  - Accelerating Voltage
    - 5kV - most surface sensitive, most useful for thin samples
    - 10kV - best resolution
    - 15kV - highest interaction volume (more noise)
  - Beam intensity type
    - Low - best resolution at high magnification (> 20kx)
      1. useful for features < 500 nm
      2. Use with higher vacuum & higher acc. voltage
    - Image - best for 1kx to 8kx
    - Point - best for Live EDS and point EDS (smaller spot sizes)
    - Map - best resolution at very low magnification (<1kx) and EDS mapping, use with low vacuum & high working distance. <u>Can sometimes help to get a clear image if other settings aren't working</u>
  - Detectors
    - BSD Full (backscatter detector)
      1. Z-contrast, heavier atoms appear brighter
    - Topo A and Topo B
      1. Uses half of BSD to give topographical features
    - SED (secondary electron detector)

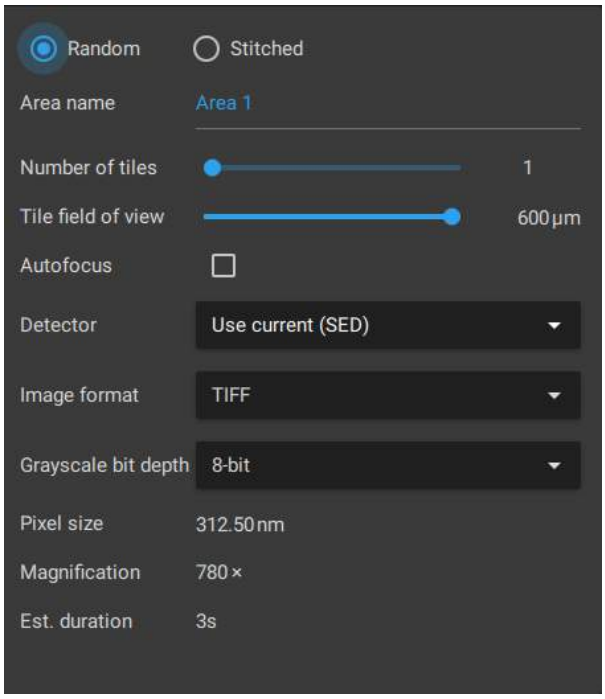
1. Requires high vacuum (1 Pa) and a conductive sample
2. Non-conductive samples should be sputter-coated with gold for SED

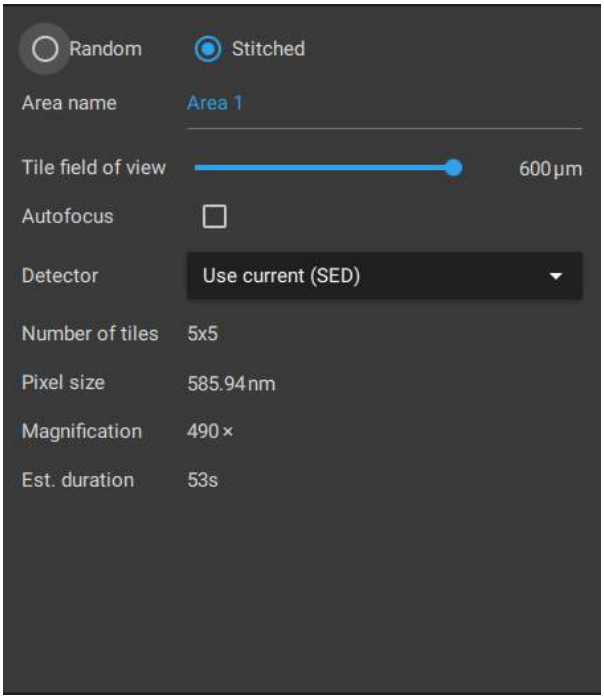
- Mixed BSD + SED
  1. Can adjust signal ratio between the detectors using slider in side panel

- Working Distance (WD)
  - Typically between 4-7 mm
- Chamber Vacuum Pressure
  - High (1 Pa) - best for conductive (including gold-coated) samples. Required to use the secondary electron detector.
  - Medium (10 Pa) - best for semiconductors
  - Low (60 Pa) - best for non-conductive samples
- Date and Time
- Label
  - Adjust by clicking the blue label name. All saved image filenames will use this label + an image number.

Automated Image Mapping (AIM)

- This feature allows you to capture a much larger image (map) of the sample with high resolution by taking multiple subphotos at higher magnifications and then stitching them together.
- Select 'Rectangle' or 'Polygon' from left menu **rectangle**
  - Double click to close your polygon
- Click and drag to highlight the area you want to capture **polygon**
- Here you can select either random or stitched





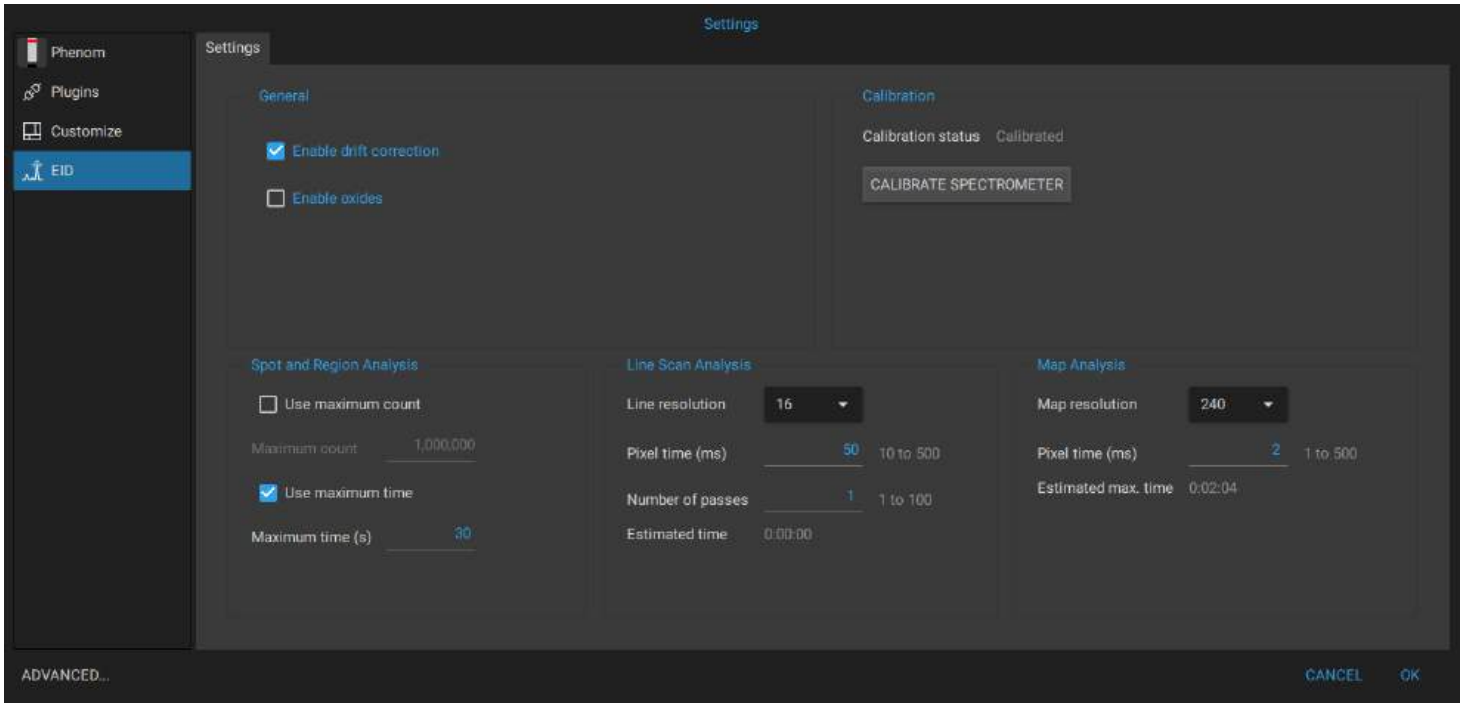
- In random, you can select
  - number of random images you want to capture (number of tiles)
  - How magnified you want images to be (tile field of view)

- Which detector you want to use
- The software gives a time estimate for scan duration (Est. duration)

- In stitched, select how magnified each tile should be (tile field of view)
  - Number of tiles shows how many images will be patched together
  - The software gives a time estimate for scan duration (Est. duration)
- Once you enter all relevant parameters, click start and enter your first name and last name as the project title.
- Don't change the project folder. You can find your folder inside: Desktop/SEM Images/Automated Image Mapping

EDS/EDX (Energy-dispersive X-ray Spectroscopy)

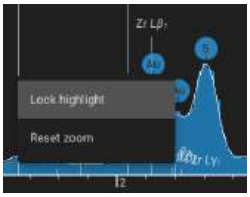
- EDS Element Identification
  - For best results, use a 15kV accelerating voltage
  - Analysis time and settings can be customized in Settings:

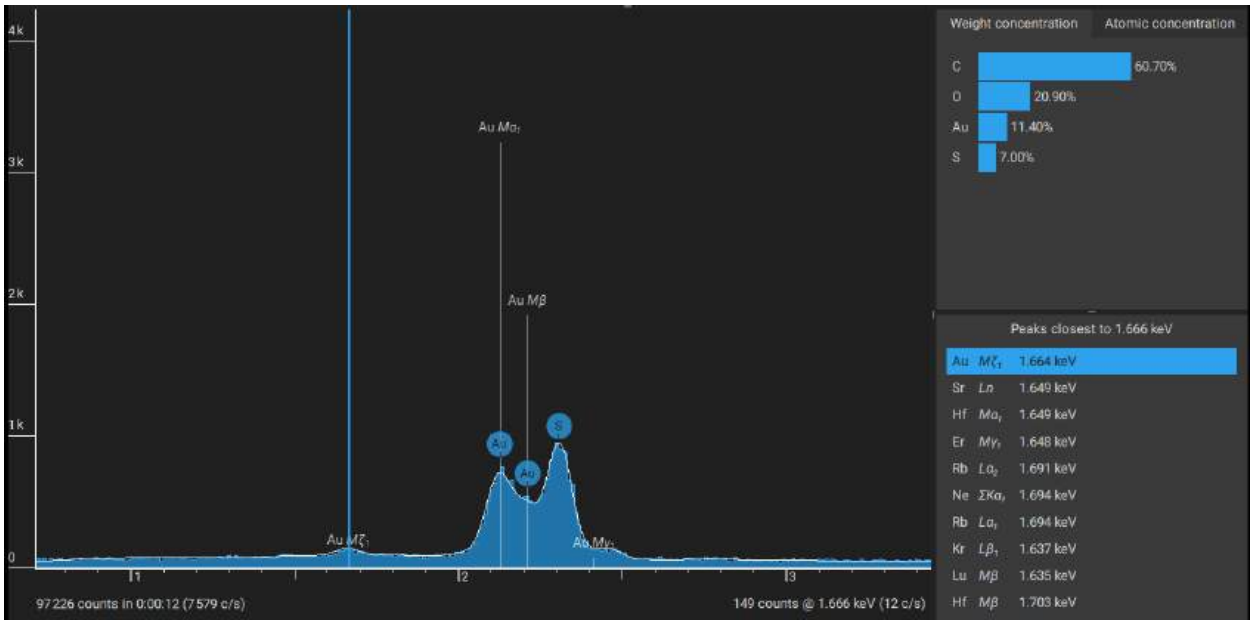


  - While the LIVE icon is present in the image, you can still adjust magnification, contrast/brightness, and focus to find a desired area.
  - On the left-side toolbar, several new icons appear for EDS:
    - Point - for best results use the "point" beam intensity; click on image after selecting the icon.
    - Region - Click and drag after selecting the icon.
    - Line - Click and drag after selecting the icon.
    - Map - "map" intensity; left-click once on the image after selecting the icon to collect EDS Map
      1. For the Map option, several elemental maps will appear next to a captured SEM image. Ctrl + Click allows you to view overlays of these maps to determine elemental distribution across the mapped region. These have

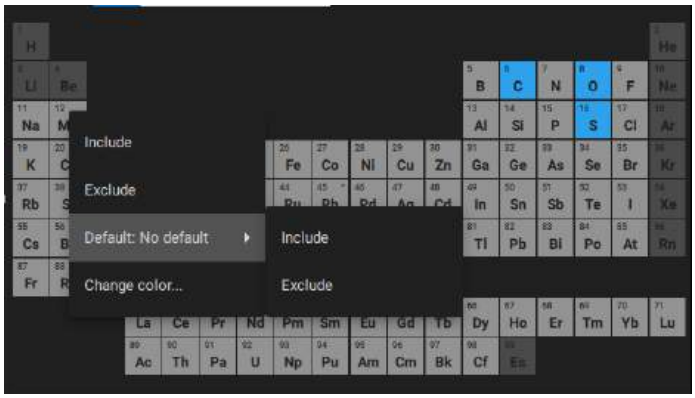
transparent backgrounds, so you can stitch them together in any photo editing software, or import them directly into a slide deck.

- After collecting a sufficient amount of your spectra, click the square stop button. Hover over the final spectra and scroll to zoom along the x-axis. Hover the cursor over peaks to see their energy and counts in the bottom right of the spectra.
- Right-clicking and selecting “Lock highlight” will show possible emission lines in a box to the right of the spectra.
  - Selecting a specific line from that box will overlay that element’s emission lines on the spectra.





- For all options, left-clicking on elements in the periodic table shows their emission lines on the spectrum. Right-clicking allows you to include or exclude specific elements from the composition calculation



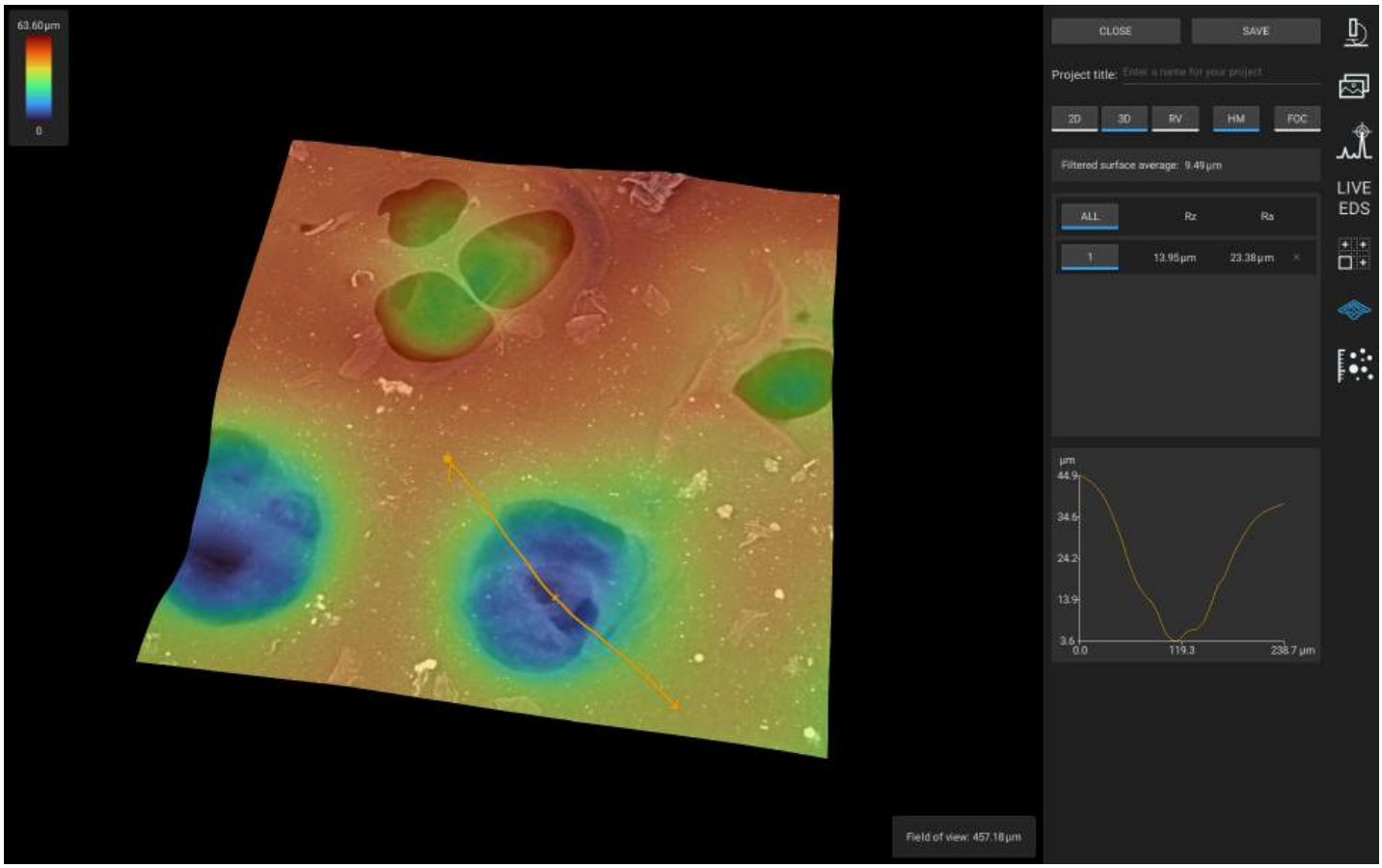
  - To save measurements, use the save button on top right and use your First Name Last Name as project title. Do not change the project folder.
  - Your folder will be saved inside: Desktop/SEM Images/Elemental Identification

- LIVE EDS
  - This is a fast method typically used to verify the composition at certain points or within certain phases seen in BSD

- Simply click on several points to perform EDS at those spots
- Click the square stop to stop the live view.
- By default, none of these measurements are saved. To save them, click “Add to project” in the top right, which will redirect you to the main EDS tab. Refer to the previous section (6a.v-viii) for elemental identification and saving.

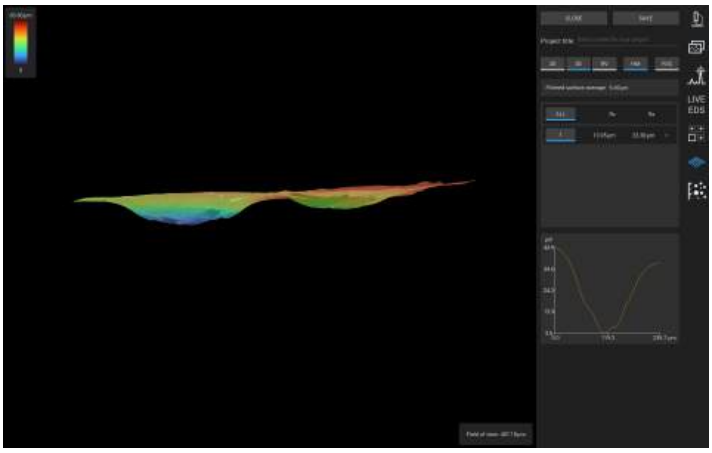
3D Roughness Reconstruction

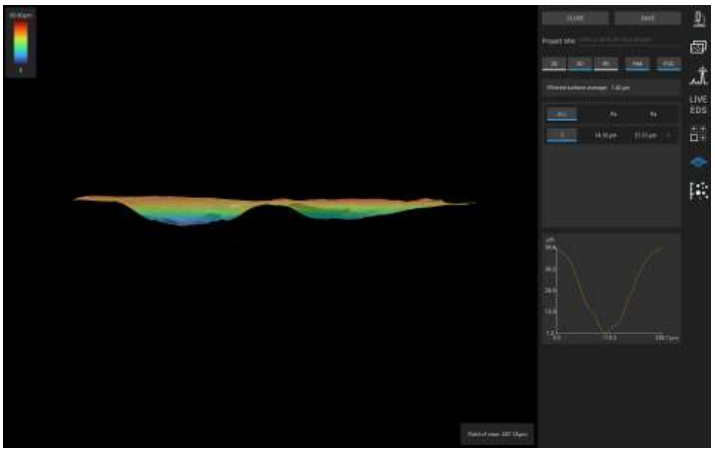
- This software takes an image from the live feed and uses a “shape-from-shading” technique to reconstruct the surface of your sample
  - Uses the shading from scans to infer the topography
- Gives best/most interesting results on more rough surfaces
- Once the sample is scanned, you can annotate and change views in which the surface is displayed

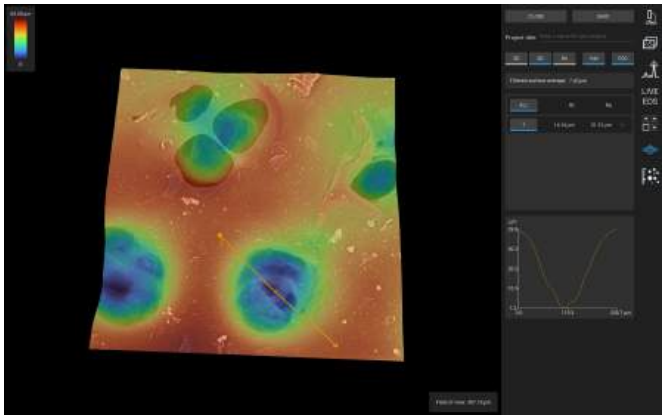


- To annotate, left click and drag to create vectors, these vectors can be moved around from the tip, mid-point, and tail
  - As seen in the example, the graph gives the height along the vector to better display topological features
  - Each vector also has roughness data, Ra (average roughness) and Rz (roughness height) along the vector
- Right clicking and dragging, while in 3D and RV modes, moves the 3D model
- Selecting the 2D view option puts the image in a top-down perspective and flattens the scan, vectors can still be made in this mode

- Selecting the 3D view option uses the preset filtered surface average determined by the computer, roughness parameters determining this can be changed in RV mode
- Selecting the RV (Roughness Profile) option allows you to adjust the cutoff wavelengths, λ_s and λ_c, at which the surface is defined
    - This allows you to manually define the waviness to define the surface rather than having the software do it for you
    - One potentially useful application of this would be setting λ_s and λ_c to be somewhat similar values to one another on the right end of the slider, this has the effect of smoothing the surface somewhat
- First Order Correction (FOC) is helpful for normalizing the surface so that some features are highlighted better
    - The effect of FOC can be seen in the below images, the left image has no FOC and is "slanted", whereas the other image has FOC and is leveled
    - The better definition of the holes can be seen in the top-down view below



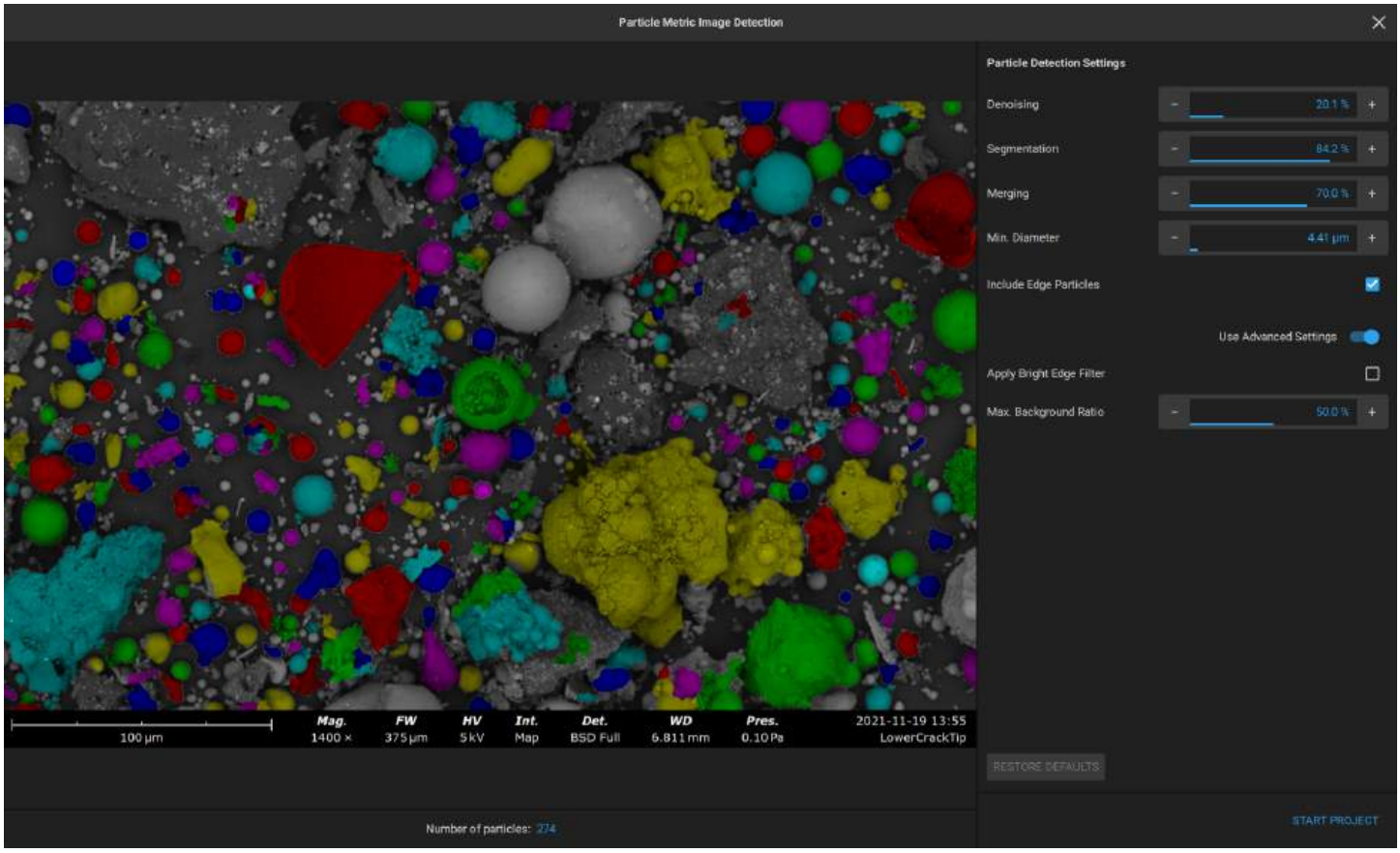




- Projects can be exported as CSV, Line Profiles, 2D/3D Images, and Word Report

ParticleMetric Software

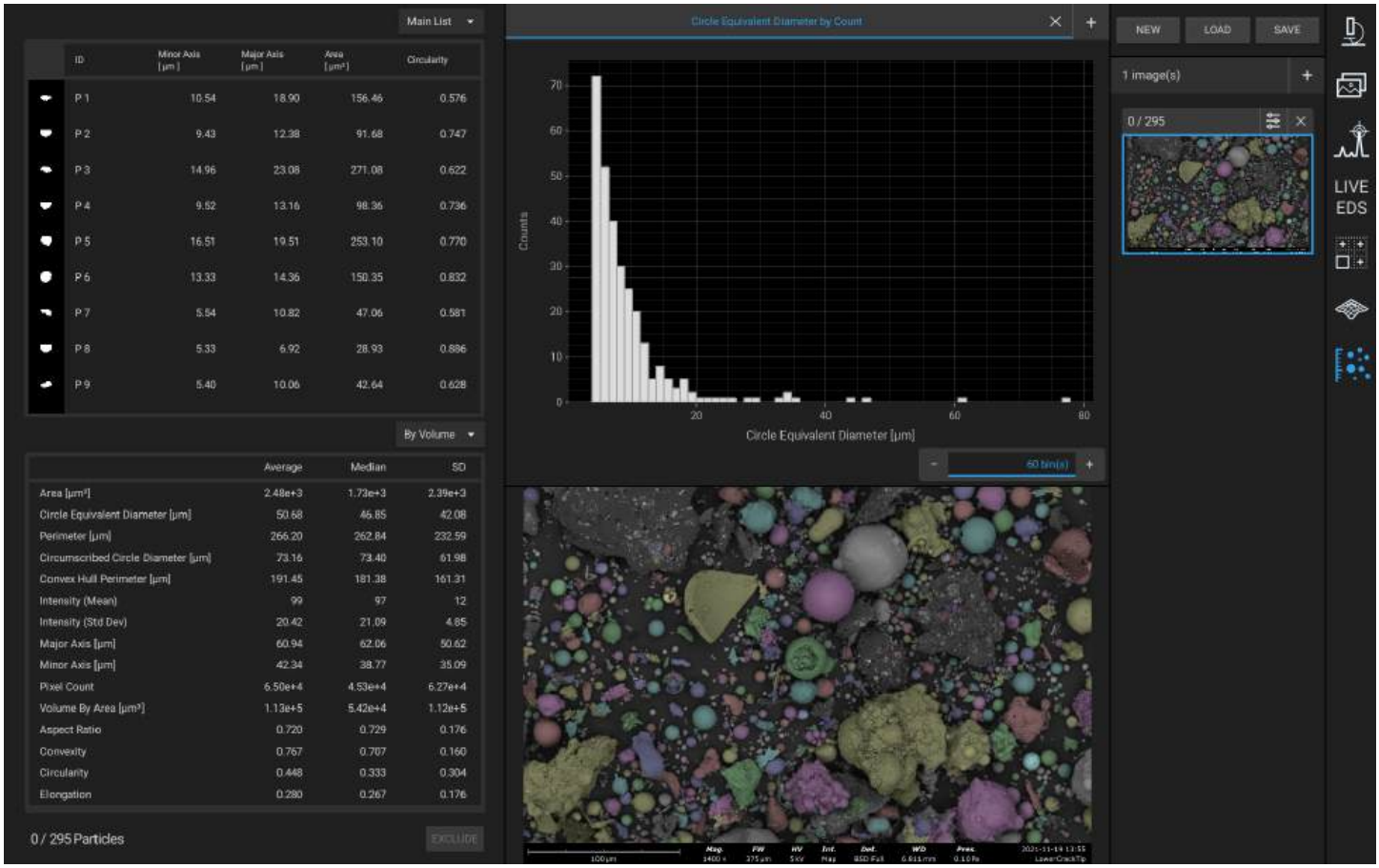
- This software is designed to detect particles in powder samples and give information about the amount of particles and other desired metadata such as circularity or major/minor axes of specific particles
    - Detectable particle sizes range from 100 nm-.1 mm
- To start using this software, click the ParticleMetric icon and you will be prompted to use the live image, load an existing image, or load a project
    - This implies this software can be used offline
    - Capabilities of the Automated Image Mapping can be paired with this software
- Loaded images are immediately taken to the Image Detection software, which comprises a complex, adjustable algorithm to detect particles in an image



- As seen in this example, not all particles observed in the image are counted, which means that some particle detection settings could be adjusted
    - **Denoising**: This setting reduces the amount of interference (non particle aspects) of the image. This setting is optimized at the default to count the most amount of particles, changing this setting makes some bigger particles defined incorrectly or merges some particles that should not be merged
    - **Segmentation**: This setting increases/decreases how segmented particles are when they are detected. Raising this setting makes larger particles more segmented, whereas lowering this setting greatly lowers the amount of particles and makes them entirely contiguous

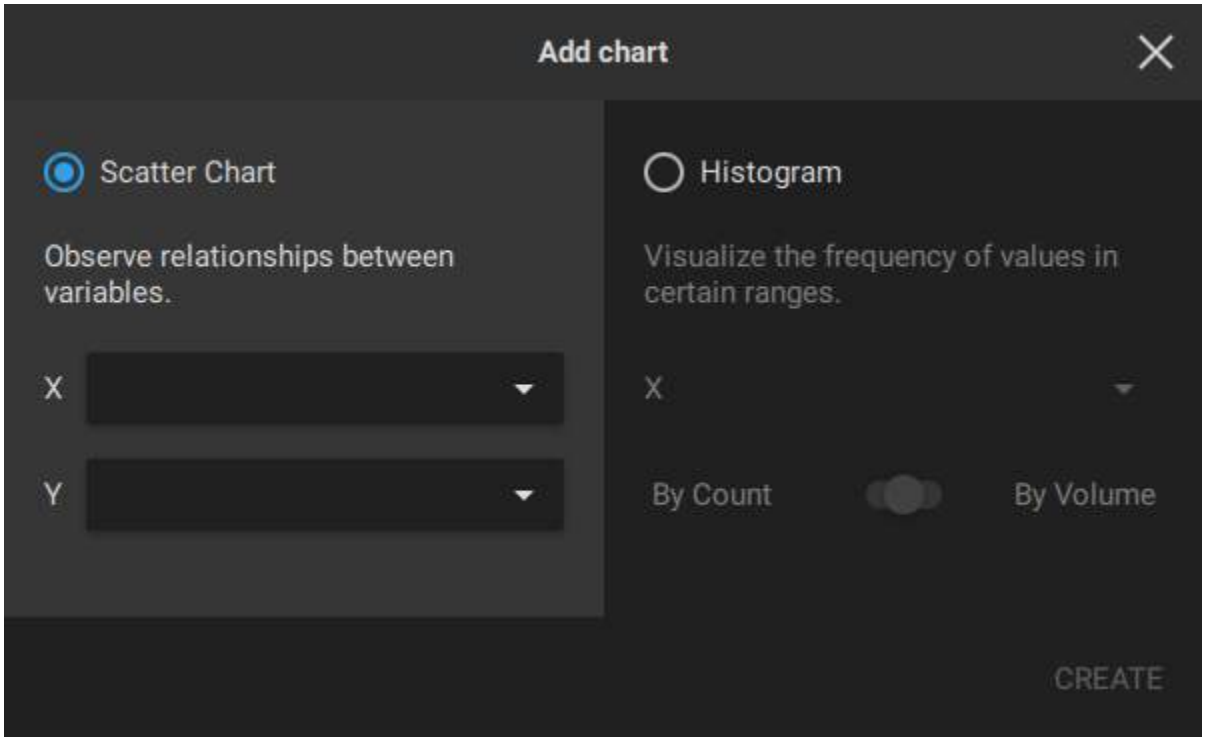
- **Merging**: This setting adjusts how contiguous detected particles are. Raising this setting makes particles more contiguous and lowering this setting makes particles more disjointed
- **Include Edge Particles**: Includes/excludes particles on the edge of the image
- **Apply Bright Edge Filter**:
- **Max. Background Ratio**: This is the amount of background in the image that is to be used in detecting particles
    - Ex. Ratio @ 100% will make the software detect the entire image, whereas lower percentages will only maximally detect that percentage of the image

- The Start Project button on the bottom right of the window will allow for analysis of the counted particles (setting changes can be made in this mode)



- The table in the top left of the window contains the every counted particle's characteristics
    - Individual particles can be selected by left clicking on its row in the table and can even be excluded from analysis via the exclude button near the bottom left of the actual picture
    - Observed characteristics on the table can be changed by selecting the drop-down menu when the mouse is hovered over the top of each column, the characteristics estimated by the software are in graph section of the SOP for Particle Metric
    - A table of only excluded particles can be viewed by selecting the drop-down menu above the table
- The bottom-left table has all of the information of each particle
    - When a particle is selected in the particle table, its features are displayed in this table
    - When no particles are selected, it displays statistics regarding the entire sample

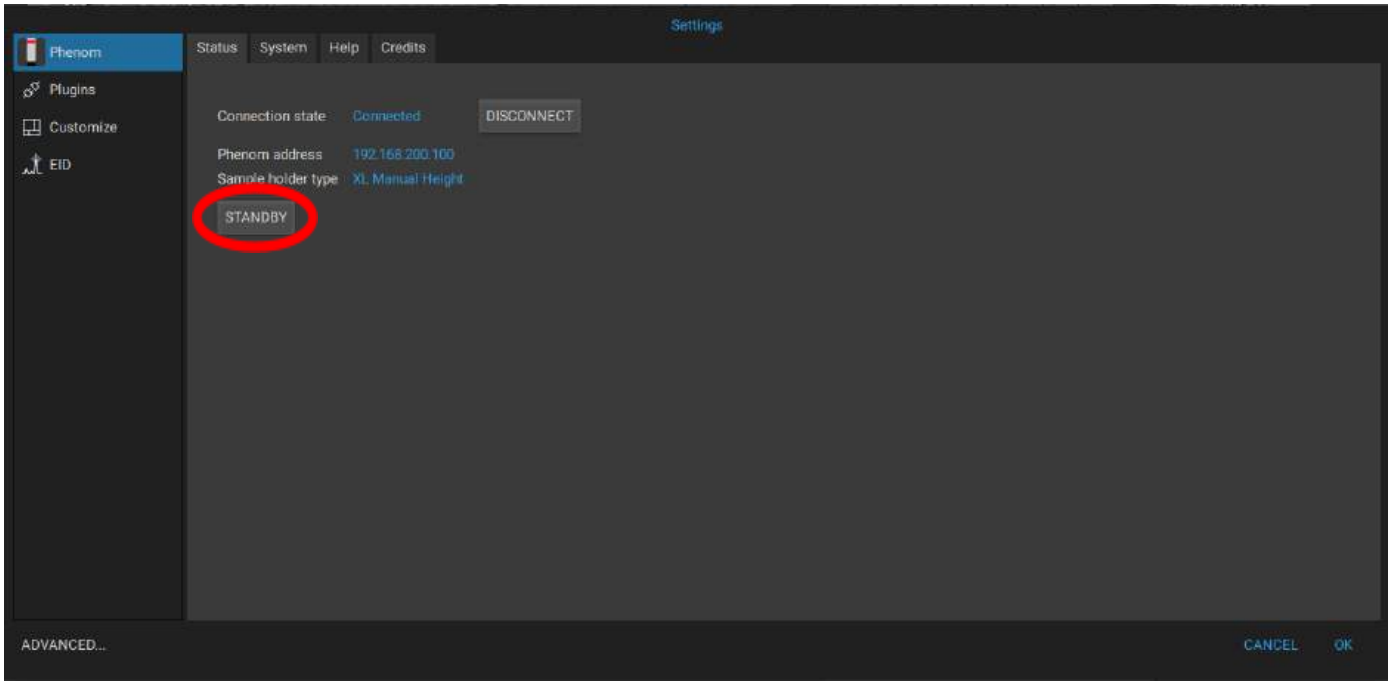
        - It is possible to change how the software sees the “entire” sample, and that is by selecting the drop-down menu above this table
            - Selecting “By Volume” gives statistics based off of the *entire* image (includes the background), selecting “By Count” only gives statistics of the detected particles
            - Ex. Having background ratio at 100% while using “By Volume” could be one approach for large particles in a sample
- The chart in the top-middle contains a chart that has variables selected by the user, to select these variables, look above the chart and there will be tabs that can contain multiple charts, to create a new one, click the “+” on the right



- The variables available for selection are: Area, Aspect Ratio, Center of Gravity X, Center of Gravity Y, Circle Equivalent Diameter, Circularity, Circumscribed Circle Diameter, Convex Hull Perimeter, Convexity, Elongation, Intensity (Mean), Intensity (Std Dev), Major Axis, Minor Axis, Perimeter, Pixel Count, and Volume by Area
    - When sections of any chart here are highlighted, the corresponding particles in the particle window will also be highlighted
    - It is possible to adjust the amount of bins in the histogram, it is below the chart
- Settings for adjusting the particle detection algorithm are in the right column of the interface where the pictures are
    - There, you can also create new projects, load old ones, save the current project, and also delete and add images
- When a project is saved, it saves the data as CSVs and even makes a WordPad document of the project

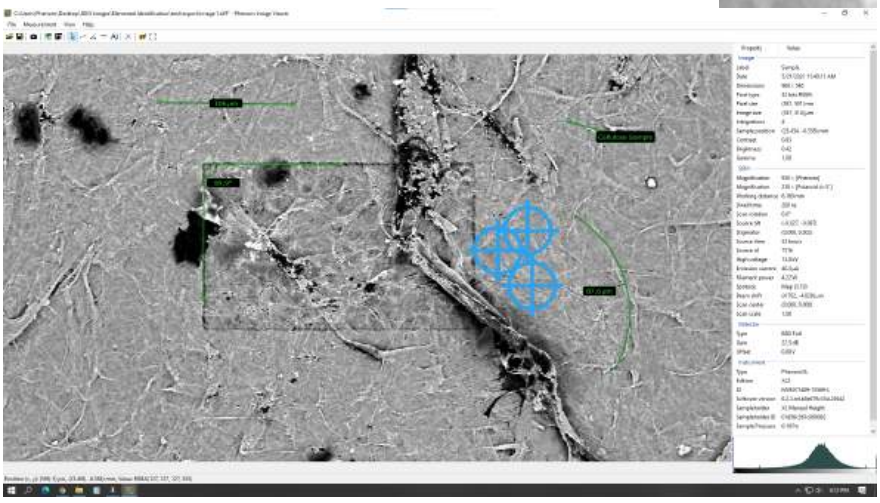
Standby

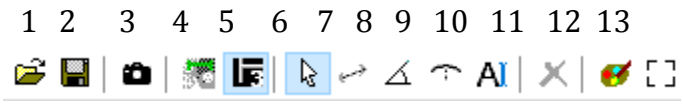
- When done with the instrument, go to Settings > Status in the top left and set the instrument to Standby
- Do not log off the computer or turn off the SEM!



SEM Image Analysis (PhenomImageViewer)

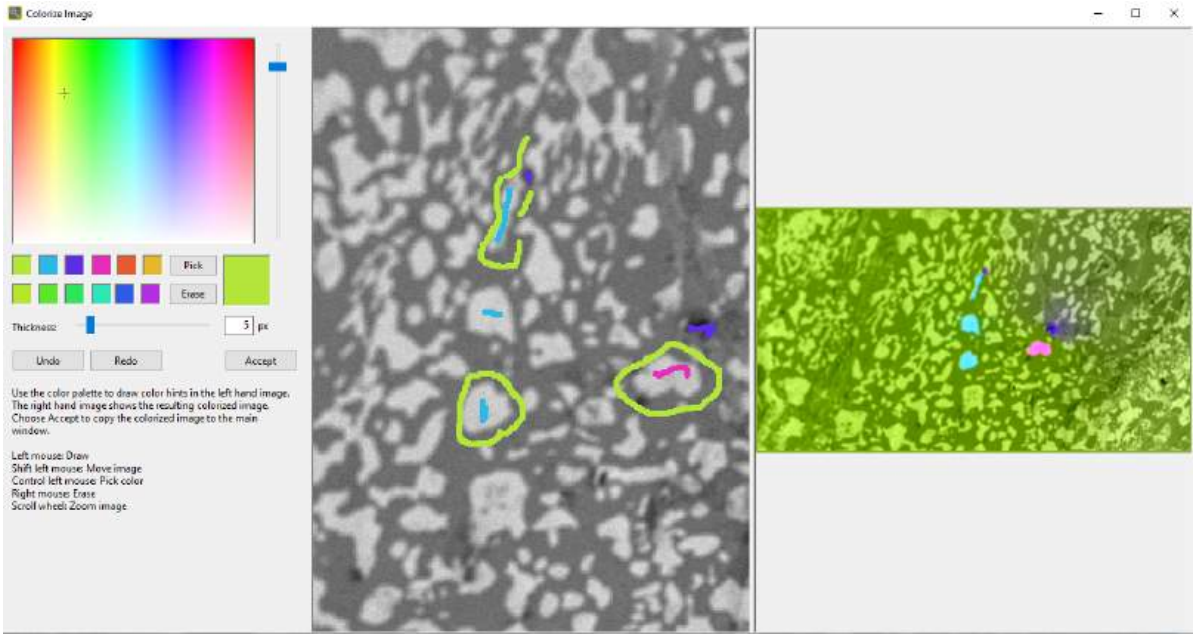
- To perform measurements and data analysis, use PhenomImageViewer.
- You may also download this software to your personal computer at this link.
- Click on PhenomImageViewer on Desktop and File>Open your image as either a .tiff or a .jpeg, both of which support SEM metadata.
- Metadata is available on the left: Ctrl+Enter to Show/Hide Metadata.
- The toolbar on the top left allows you to perform measurements.





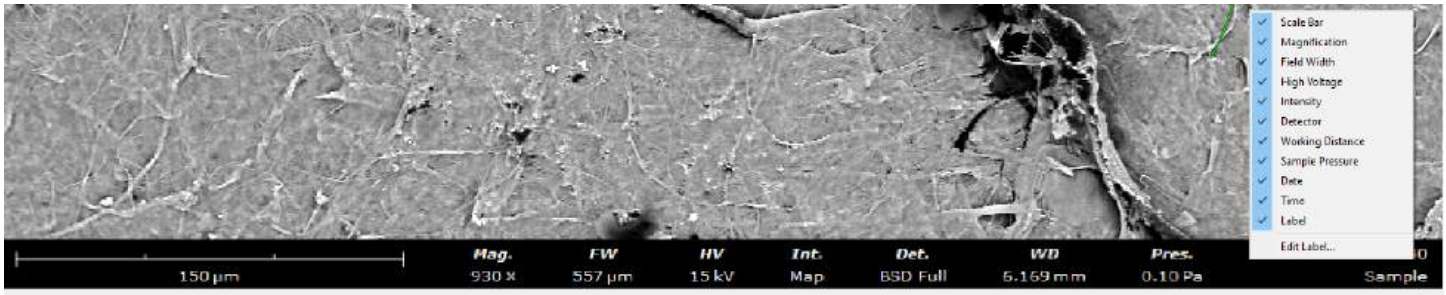
1. **Open:** Open files
2. **Save:** Save files
3. **Acquire:** This allows you to capture images within the software (not tested)

4. **Scale Bar:** Select/Deselect or Ctrl+B to Show/Hide Scale Bar
5. **Data Bar:** Select/Deselect or Ctrl+B to Show/Hide Data Bar
6. **Select:** Use this icon to move measurements
7. **Length:** Hold and drag to measure a distance
8. **Angle:** Hold and drag to measure an angle
9. **Radius:** Hold and drag to measure a radius
10. **Text Label:** Hold and drag to label a feature
11. **Delete:** Select a measurement first, then click this icon to delete it
12. **Colorize Image:**



   a. Label features you want to group with the same color on the left screen
   b. Software will automatically segment the image based on your labeling

- Right click Data Bar on the Image to Select/Deselect information to be displayed:



SEM Focusing Guide

Setting Changes

- Accelerating Voltage:
  - 5 kV: Often not enough interaction volume, useful for very thin samples where higher voltages would overpenetrate, sometimes useful for rough samples[1]
  - 10 kV: Usually the best at most magnifications
  - 15 kV: Often overkill[2], sometimes necessary at very high resolutions to resolve the image to an acceptable degree; needs a well-conducting sample
  - Additional Notes:
    - (1) See https://www.cambridge.org/core/journals/microscopy-today/article/choosing-the-right-accelerating-voltage-for-sem-an-introduction-for-beginners/C62A3149CA4AF541EB1FBCD681CBE79A : "small details of the surface could be smothered or completely wiped out by a higher accelerating voltage"
    - (2) For poorly grounded polymer samples mainly, a higher acceleration voltage can cause charge buildup that will degrade the sample in realtime; can be mitigated by lowering the voltage, adjusting vacuum pressure, or making sample conductive (sputter coating)
- Beam Intensity:
  - Low: Best at high magnification (>10000x); use with high vacuum pressure, highest voltage possible (usually, see above)
  - Image: Best at medium magnification (1000x to 10000x)
  - Point: EDS
  - Map: Best at very low magnification (<1000x); sometimes advisable to use lower vacuum pressure, but often acceptable at higher pressure if you're already at high vacuum
  - Additional Notes:
    - The choice between low and image is subjective at almost all magnifications; image is often better; try them both
    - **If nothing else is giving a clear image try using map. Once you get in focus you can either use as is or try to switch back to other beam settings.**
- Detector:
  - BSD Full:
    - Backscattering detection: default mode; heavier elements appear brighter (Z-contrast); usually provides the most useful contrast in aspects other than topography
  - SED:
    - Secondary electron detection: provides the best overall detail, including *some* topography, especially on samples with the same/similar elemental composition throughout[3]; requires high vacuum and a well-conducting sample
  - Topo A and Topo B:
    - Topography: Shine beam at an angle to provide shadowing

    - Mix (BSD + SED): Useful if you want the Z-contrast of BSD but also the benefits of SED; can be adjusted with a slider in the right side panel (the blue arrow on the right)
    - Additional Notes:
        - (3) If your sample has different physical divisions (grains, impurities, etc.) of the same element or elements of similar atomic mass, BSD may not provide acceptable contrast or detail
- Vacuum:
    - High (1 Pa): Usually the best; provided you have a well-conducting sample
    - Medium (10 Pa): Not often used; sometimes good for semiconductors or samples of intermediate conductivity
    - Low (60 Pa): Usually only used if sample is really insulating (nonconductive)
    - additional notes:
        - Bigger number (in Pa) = higher pressure (closer to atmospheric) = lower vacuum = more air left in the chamber
            - More air left in the chamber means electrons have another path out of the sample; if your sample is not conductive this may be the only way out for the electrons, which is why Low vacuum is used in this case
            - More air also means more opportunities for the electron beam to get degraded on its way to the sample and for electrons leaving the sample to get dispersed, which is why Low vacuum is only used when necessary
        - Atmospheric pressure is 101325 Pa at sea level
- Averaging (under both “Live” and “Acquisition”)
    - Live: No averaging; most noise visible, but fastest refresh rate/image acquisition
    - Medium: Averages 4 frames; usually pretty good for Live image, not bad for Acquired image
    - High: Averages 16 frames; usually the maximum necessary for both the Live and Acquired image
    - Best: Averages 32 frames; almost always overkill unless you have an extreme amount of noise
    - Additional Notes:
        - Averaging averages the pixel values from a certain amount of previous frames to reduce noise (TV static looking stuff)
        - More averaging means a cleaner image, but more lag and ghosting when adjusting a Live image, and longer acquisition time for an Acquired image
        - Averaging can very often make a terrible image look good
- Scan Size (under both “Live” and “Acquisition”)
    - The nominal resolution of the image; how many pixels wide and tall the image will be
    - Different available options between Live and Acquisition
    - In a similar fashion to averaging, higher values increase quality, but reduced refresh rate in the Live image and increased acquisition time
    - It is recommended to use 1920x1200 or 1440x900 for the Live image because of the complete lack of clarity of lower resolutions; if you need better refresh rate for focusing, try to use the “focus in selected area” feature or reduce averaging before reducing the resolution

    - For the Acquired image, the scan size depends on your needs, higher scan size means you will be able to see more details (less pixelated) when zooming in on your image file
    - Higher scan size requires longer exposure time
- Time Estimate (beside "Acquisition")
    - Approximately how long it will take to acquire the image when you click the capture button
    - Affected by changing the average and scan size parameters under the "Acquisition" section
    - ***IMPORTANT***: The longer the acquisition time, the more opportunity for the image to "shift" due to charge buildup and other factors; this will result in a blurry final image, as the acquired image will be the combination of multiple slightly moved around images, that themselves may be blurry due to exposure time
        - If your image is moving around "by itself" in the live view, you will need to take that into account
        - If you experience this shifting, reduce averaging and/or resolution to get the lowest time estimate that you can that still results in an acceptable image, this takes trial and error

Imaging Toolbar (Bottom Left)

- Unless otherwise noted, all of these settings are controlled by moving the scroll wheel once you have selected said setting from the sidebar
- Rotate:
    - Self-explanatory; rotates the image
    - Can rotate by 0.01 degrees in fine mode[1]
- Source Tilt:
    - Tilts the sample so the beam hits it uniformly
    - Usually best to use automatic source tilt: focus the image pretty well, and let the SEM do the rest
- Stigmate:
    - ***DO NOT USE***: unless you know otherwise, the stigmation has been calibrated by the technician and should not be altered
    - It is usually best to use automatic stigmation: zoom into above 40000x, focus the image best you can, and center the image on a round (or vaguely round if that's the best you have) feature, and then click the button
    - Astigmatism is not detected by only focusing the image, first you must focus the image and then put the image out of focus in both directions. If the image is "smeared" across a line when out of focus in one direction, and then "smeared" across a second perpendicular line when out of focus in the other direction, then astigmatism is present.
    - Additional Notes:
        - https://www.cambridge.org/core/journals/microscopy-today/article/correcting-astigmatism-in-sem-images/7ED43987C7916AAFBE1869522546AC84
        - Astigmatism is an issue mainly at high resolutions and causes the image to appear stretched at an angle due to the electron beam not being perfectly round
        - The value of manual stigmation is dubious

- Brightness/Contrast

    - Right clicking the button in the sidebar lets you switch between automatic and manual mode (as with every toolbar adjustment with automatic options)
        - The Automatic Contrast button normalizes the distribution of intensities of returned signals to try to return a grayscale image (This distribution is on the right Side Panel)
    - Pressing the middle mouse button[1] when in manual mode switches between brightness and contrast
    - There is no fine brightness/contrast adjustment mode
    - Additional Notes:
        - Contrast: higher contrast increases the difference between the bright and dark areas of the image; in other words, the dark parts become darker and the light parts become brighter
            - One way of thinking of it is this setting "shifts" the histogram of intensity signals detected by the electron detectors to the left (darker) or the right (brighter)
            - Up to a certain point, increasing contrast can make the image appear sharper, as it makes the edges between features more defined
            - Contrast that is too high will cause light features to all merge together, even if they have slightly different brightness, and the same is true of the dark features
            - "Maximum" theoretical contrast is an image that has only pure white and pure black portions
            - Contrast that is too low will make light and dark features indistinguishable from each other
            - "Minimum" theoretical contrast is an entirely gray image
        - Brightness: Increases/decreases the brightness of the *entire* image, regardless of whether the pixel is dark or light relative to the rest of the image
            - You will likely want to adjust brightness after adjusting contrast
- Magnification
    - Ranges from 610x to 210000x (bounds can change slightly based on other settings)
    - No fine magnification option
    - As magnification gets higher, the magnification value "accelerates"
    - Additional Notes:
        - Increasing magnification physically moves your sample closer to the detector
        - Generally, you will need to adjust focus after changing magnification, and may need to change other settings after changing it by a large amount
- Focus
    - Focus in Selected Area only has focus changes occur in that area
        - Ex. You could select Focus in Selected Area and then hit Autofocus to focus in that area
    - A good technique (sometimes) for things that are having trouble focusing is to select Focus in Selected Area and to change focus settings in that mode and then left click out of it to apply focus changes to the rest of the image

    - Sometimes just taking the stage out and then putting it back in can help with focusing sometimes
    - additional notes:
        - most important setting on the SEM (arguably); your image ***MUST*** be in focus if you want any hope of it looking good, and this becomes even more true at high magnifications
        - a simple way to think about it is that by adjusting focus, you are telling the detector how far away your sample is from it (not quite the truth but it's pretty close)
            - remember, when you zoom in, the SEM is physically moving your sample closer to the detector, so that's why you have to change focus when changing magnification
        - an out-of-focus image will appear blurry regardless of if the focus is too far or too near

Sample Preparation

- 90° and 45° stubs
    - When working with the 45° stub, it can be useful to lower the sample below the six notches you are supposed to lower it when it is leveled with the stage
        - This can give the SEM more working distance, which can allow it to focus on some features better
    - Focusing on features with 90° stubs can be difficult, so if you are preparing a sample and intend to use the 90° stub, try to make it closer to the edge of your sample so that focusing is easier
- Metals
    - Generally the least intensive preparation for samples since they are inherently conductive
    - Necessary to cut metal sample into a small enough shape for the SEM
    - Helpful to polish the metal in a polishing lab to get more defined grains/phases
    - Despite the fact that metals are conductive, the sample needs a place to ground when the electron beam impinges on it, so the electrons need a path out of the sample. In some cases this may mean using some copper tape to connect the sample to the conductive part of a stub
- Powders/Particles
    - Powder samples, when prepared incorrectly, can damage the SEM by "kicking up" particles from the samples that are loosely attached
        - Loose particles can attach themselves to electron/X-Ray detectors as well as apertures within the SEM
    - To minimize damage, place a conservative amount of powder on a stub with carbon tape on it, then ***use an air gun*** to blow off any loose particles
        - To ensure *no* damage, look away from concentrated areas of powder to prevent any movement
        - Lowering acquisition time can also possibly provide better images with regard to movement
    - Colloidal graphite (in the flammables cabinet) can also be used to ensure powders don't get kicked up. Start by attaching tape to an SEM stub, then brushing a minute amount of

colloidal graphite onto the tape. Then sprinkle the powder onto the colloidal graphite and firmly press onto the stub to ensure it is stuck. After this, use the air gun to remove residual powder

- The sputter coating process can help prepare powder samples by removing any loose particles in the vacuum
- When using the SEM for powders, make sure the **vacuum is set to low**
- Carbon tape can interfere with EDS, so excluding C from readings is helpful

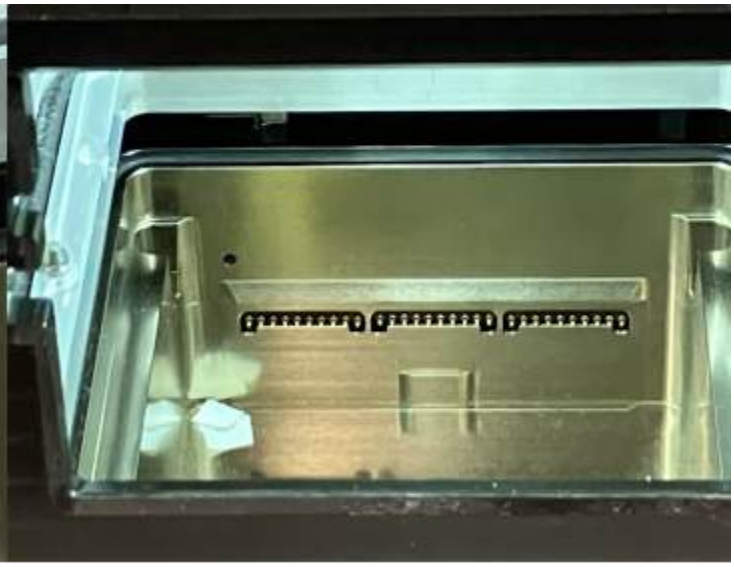
Colloidal Graphite

SEM Maintenance Guide (only for TOs)
(update this as you gain more info about maintenance)

Cleaning the Vacuum Seal:
Any contaminants from around the stage can be transferred to the rubber vacuum seal within the SEM chamber. This will appear as dust around the black ring atop the stage's compartment. After dousing a kimwipe with alcohol and wearing gloves, one can wipe the vacuum seal clean of dust to ensure there is a proper seal during usage.

Cleaning the Chamber:
There should not be any contaminants inside the chamber where the stage goes. Large contaminants (like pieces of carbon tape) should be removed with gloves. Contaminants can also get on the sealing of the chamber. Do not try to clean the chamber with a solvent.

Dust contamination on rubber seal



Paper contamination in stage chamber

Crating the SEM (to Send it for Repairs):

Every 2 years or so the SEM is serviced by a technician who comes in and runs diagnostics on the machine. In the event the machine is not easily reparable, it will have to be sent back to the Nanoscience shop where it will be fixed in a controlled environment. Prepping and crating the SEM is comprised of the following steps (the technician will be there to help you):

0) **Note:** You should schedule the SEM as 'Down' on SUMS 14 days prior to its servicing.
1) Booting down & disconnecting the machine
2) Boxing any separated parts (stage, loose screws/objects, cables, white box, vacuum pump, etc.)
3) Crating the SEM machine
4) Shipment preparation

Booting Down & Disconnecting the Machine:

The technician should be able to complete this step themselves. They likely will start by booting the machine down, which will take some time.

Boxing Separated Parts:

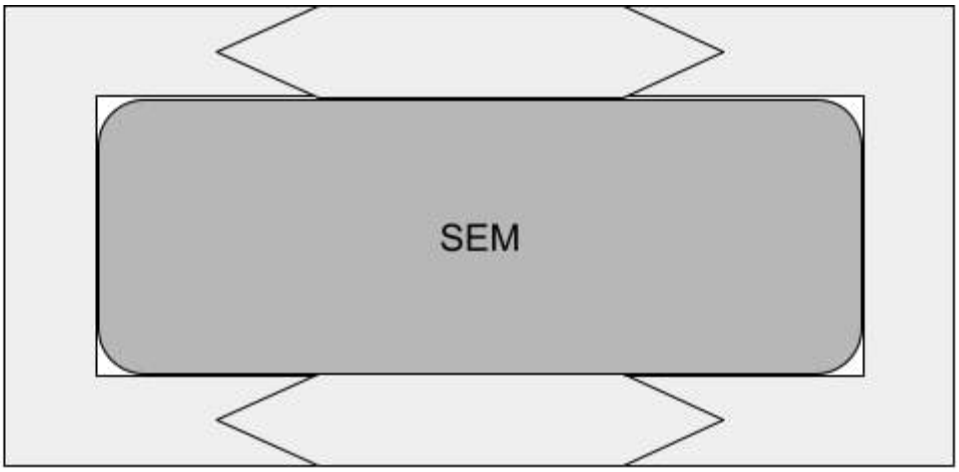
It is the responsibility of MILL staffers to hold onto any parts that may come off of the SEM during its use. These should be properly stored in a clean container where they can be kept until the next scheduled servicing. The technician will likely disconnect the SEM's cables in the back, the vacuum pump, and power source. There is a white box that comes with the SEM and contains important parts. It is usually kept on top of the shelf above the TGA. Staffers should provide cardboard boxes (and packaging stuffing) to store all of these items.

Crating the SEM machine:

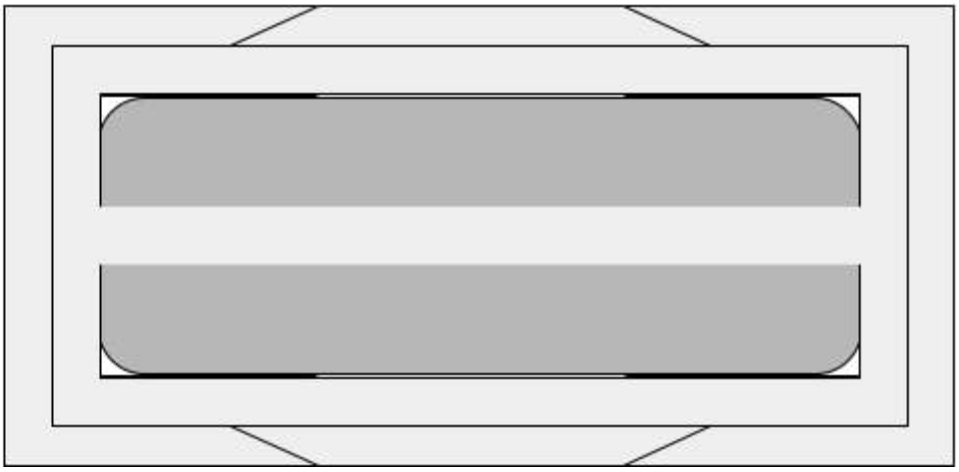
The SEM comes with a shipping crate that looks like the following:

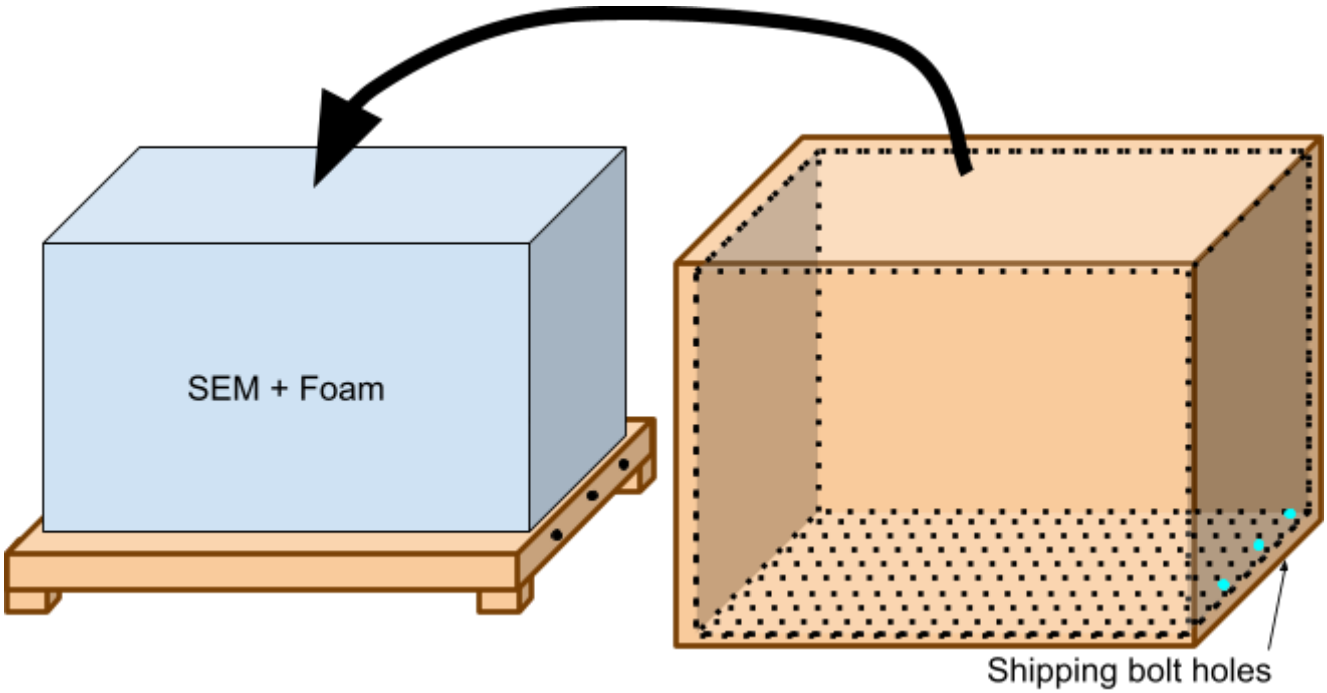
Inside the shipping crate should be the foam which the SEM comes with. Do not throw any of these out since it can take ~2000 dollars and more of your time to replace them. The actual crate comes in 2 parts (a top and a bottom), while there are (I think) 5 pieces of foam. **CAREFULLY** place the SEM into the crate, cradled by the foam as shown in the diagram below:

Without top



With top





On the bottom of the short sides of the shipping crate are places for shipping bolts (see image below).

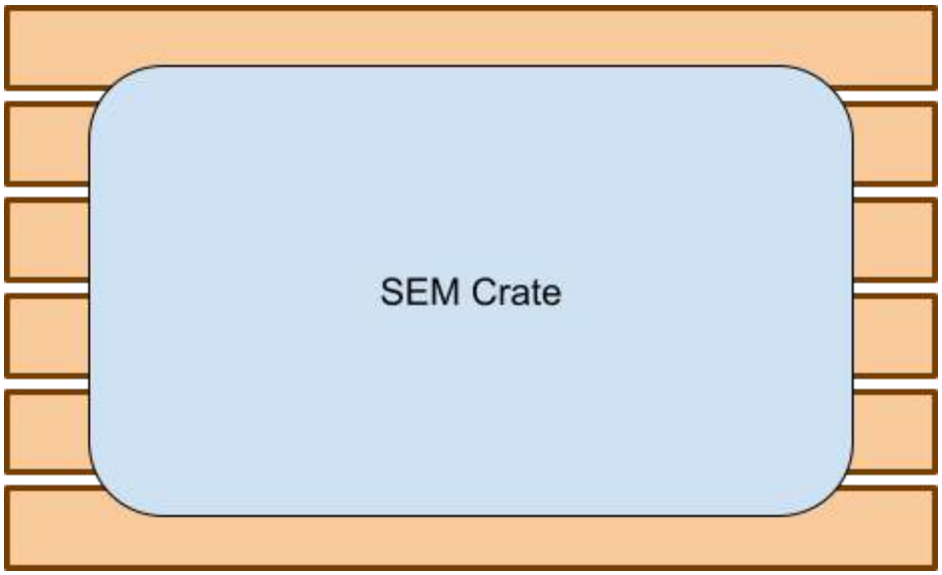
After crating the SEM screw in these bolts. The other components (power supply, vacuum pump, etc.) should be neatly stored and secured in some cardboard boxes.

Shipment Preparation:

To prepare the SEM crate & part boxes for shipping, you will need the following items:

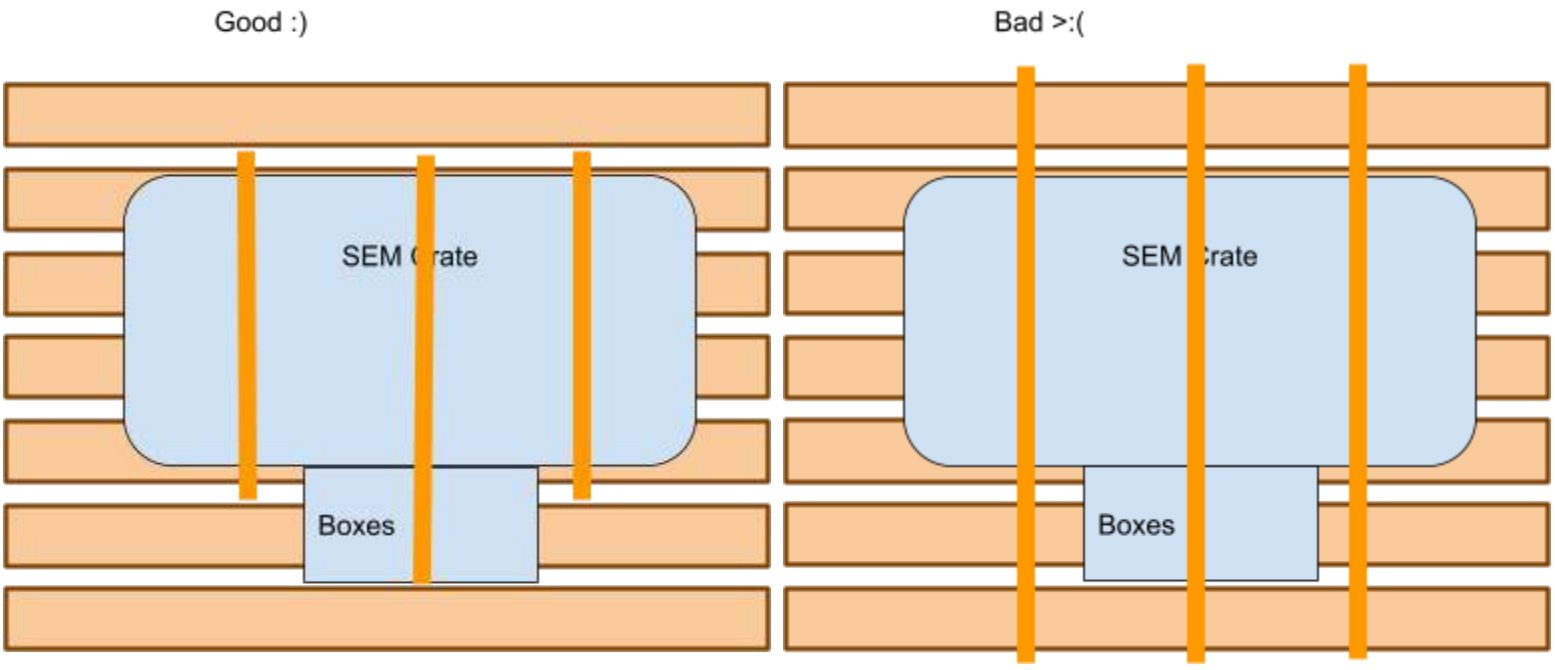
1) At least 5 shipping straps
2) A ~41”x40” pallet
3) Duct tape
4) Plastic wrap
5) Pallet jack (check basement)

Start by carefully placing the SEM crate onto the center of the pallet (make sure the pallet is in the hallway and can easily be transported out the building with a pallet jack). Make sure the long side of the SEM is in the same direction as the boards to ensure maximum stability:



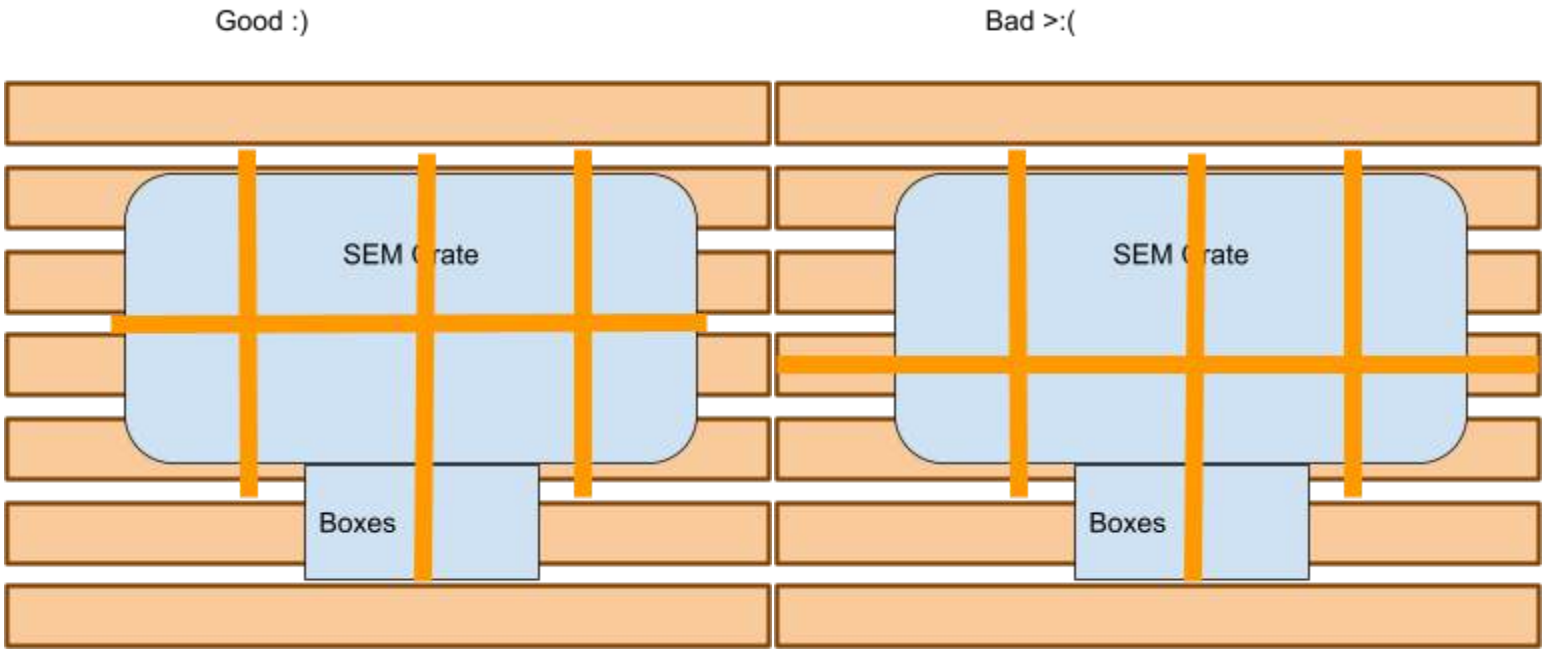
Place the cardboard boxes wherever is convenient and has the most space. Ensure they are close up against the SEM crate. Use duct tape to make sure they stay closed and/or stick them to each other.

Wrap at least 3 straps around the SEM and pallet widthwise. Include the cardboard boxes in at least one of these straps, and have just the SEM attached with at least 2. Make sure the straps are both the tightest they can be and are around the smallest width of the pallet as possible (see below):

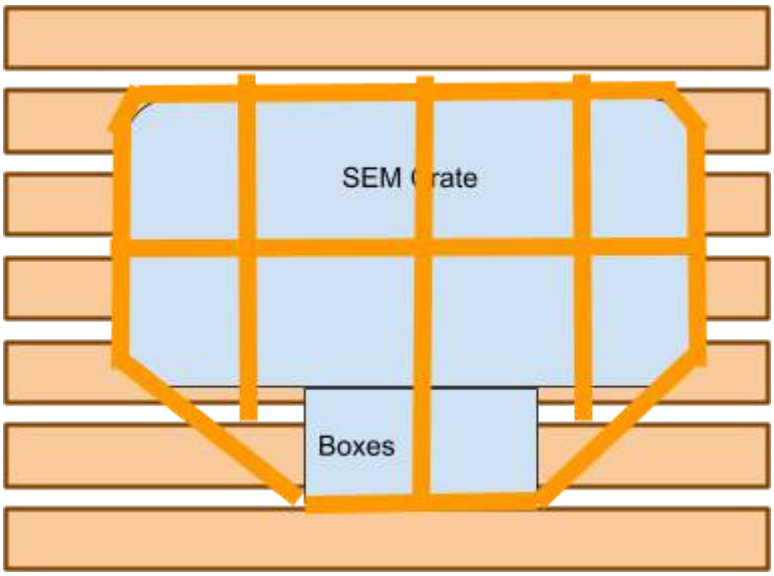


The straps should go under the first layer of the pallet, but not the second (to prevent friction as the pallet is moved from breaking the straps)

At least 1 strap should be used to go around the SEM lengthwise. Again, ensure the straps are both the tightest they can be, they are around the smallest length of pallet, and they only go through the first layer of boards (see below):



Wrap at least 1 strap around the circumference of the SEM and boxes (see below):



The strap should be at the middle height of the boxes.

Use plastic wrap to go around the SEM crate and boxes. Start from the bottom, then wrap around them up to the top. Then go from the top back to the bottom. Ensure a tight fit.

Finally, put the pallet jack into the pallet and move this setup to your desired location.

When finished, the result should look something like this:


Mighty


FEDEX FREIGHT
Pick up
KEEP UPRIGHT
UP
TIP-N-TELL
thermoscientific


Open Here
Open Here
Open Here