SEM S.O.P

Instrument:

Phenom Pro G6 Desktop SEM from Thermo Fisher Scientific

Software:

Phenom User Interface

Purpose:

To manipulate the interaction of electrons with a material for high resolution imaging.

To Begin:

- **Know the specifications of the instrument.**

Specifications	Phenom Pro G6
Source	CeB_6
Detector 1:	Backscattered Electron (BSD)
Detector 2:	Secondary Electron (SED)
Sample Stages:	High and Low Vacuum*
Sample Dimension Diameter	≤ 25 mm diameter
Sample Dimension Height	≤ 35 mm height
Resolution	≤ 6 nm SED ≤ 8 nm BSD
Light Optical Magnification	27-160x
Electron Optical Magnification	160-350,000x
Accelerating Voltage	5 kV, 10 kV and 15kV
Image Resolution	960x600, 1920x1200, 3840x2400 and 7680x4800 pixel

*Low Vacuum mode will only support BSD for charge reduction.

- **Steps to Start Up the Instrument**
  - **NOTE:** The instrument stays on, the source hibernates after 1 hour of inactivity, but users should put the source on standby once finished with imaging to prolong lifetime of the source
  - **NOTE:** Samples should be completely free of moisture or loose particles, damage to source or detectors due to improper sample use may lead to being liable for instrument repair charges.

1. The instrument should be on, if not there is a power switch in the back of instrument.
2. Log in to the computer.
   - There is no computer password.

I. Open Software

1. Phenom User Interface  should already be loaded, but if not, it is in desktop.
   - It is recommended to restart the software occasionally to ensure proper instrument communication with the software.
2. Source will be on standby; instrument will wake up in about a minute and a half.

II. Prepare Sample

- **NOTE:** Samples should be free of solvent to prevent outgassing under vacuum, which could damage the system
- **NOTE:** No loose particles should be present
  - Compressed air can be used to get rid of any loose particles, a duster can next to the system should be available.
- **NOTE:** Follow sample dimension restrictions incorrect sample loading may damage the system

1. Put on a new pair of Nitrile Gloves
   - Oils from the hands or contaminants can be introduced if not using gloves, this can damage the system.
2. Select an available sample stub compatible with sample dimensions.
3. Carbon tape is available for samples next to stubs.
   - Peel back the covered adhesive of tape and place the stub on top.
   - Then peel off the covered adhesive.
4. Place sample on top of adhesive or pipette suspension if using a volatile solvent.
   - If using a solvent, make sure solvent is fully evaporated before placing it into the stub holder.
   - Never load a sample with the stub already on the stub holder to prevent loose particles getting on the holder.
5. Use air to get rid of any loose particles.
   - The air stream should not be too close to the sample.
6. For Non-Conductive Samples Sputter Coater is required. See Sputter Coater SOP for detailed instructions.
7. Sample is now ready to load onto the stub holder.

III. Select Stub Holder

- There are two available Stub Holders that are color coded.
  - Black Stub Holder: High Vacuum
    - Standard Holder that uses full vacuum allowing for use of BSD and SED.
  - Grey Stub Holder: Low Vacuum
    - Charge Reduction Holder that uses a lower vacuum to allow free air molecules to prevent localization of electrons on a sample surface, yielding a low-resolution image and sample movement.
    - BSD only

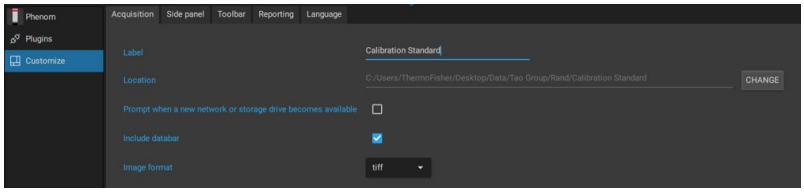
1. Select appropriate Sample Holder based on Sample.
2. Place prepared sample stub on to the Holder.
   - Ensure the stub is secured in the holder and properly mounted all the way down.
2. Adjust the height of the sample so that the tallest region of the sample is fully flushed with the rim of the holder.
   - Height adjustment ring can be rotated counterclockwise to lower the stub height.
     - Vertical marks on the ring represent 0.5 mm.

IV. Load Sample Holder Into Loading Bay

1. Open Loading Bay door by raising the tab in the front.
2. Insert the Sample Holder into the loading bay.
   - Each Stub Holder contains a sensor the instrument will read to identify the settings for the holder.
3. Close the Loading Bay door by pushing the tab down.
   - Slight force may be required.
   - A door lock sign light up on the instrument.
4. Light Optical Image will now be displayed.

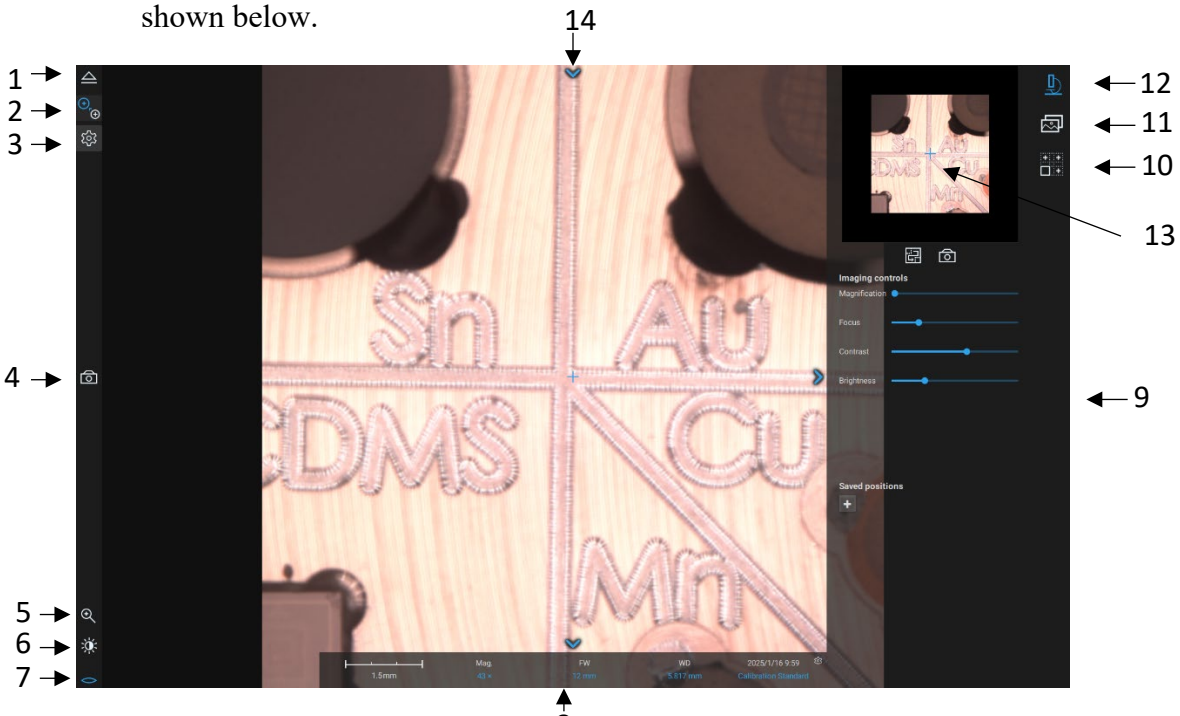
V. Setting the Save Location

1. Select the gear icon in the top left of the screen to open the settings window.
2. Under the customize tab, change the name of the Label to rename the sample for the data bar.
3. Also on the customize tab, select the change button to set the save location of all images to be taken for the session.



VI. Light Optical Imaging

1. Initial light optical image will appear as soon as the Loading Bay door is closed as shown below.

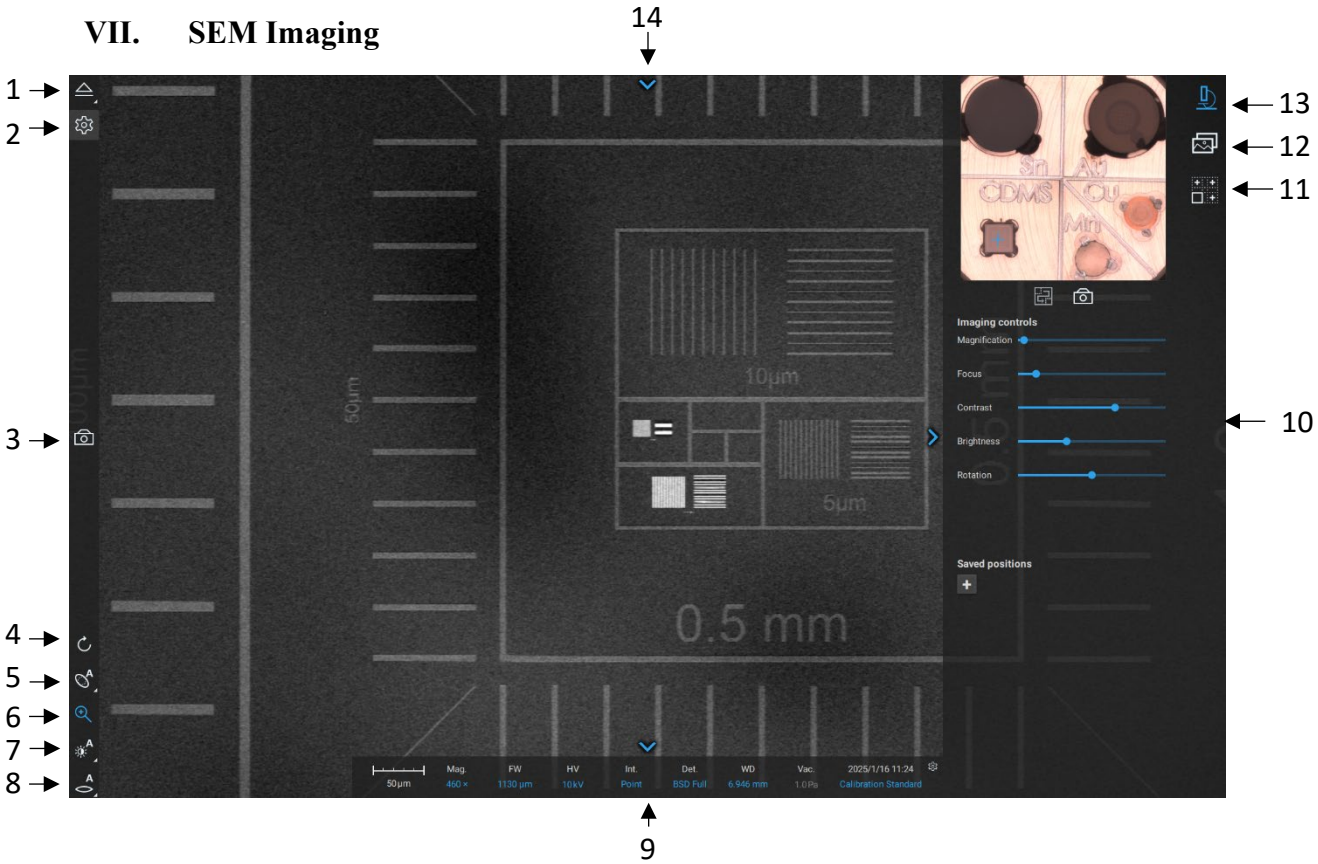


Icon Legend

1) Eject Sample	8) Live Data Bar
2) Switch to SEM Imaging	9) Right Side Pane Expansion
3) Settings	10) Map Stitch Imaging
4) Take a Picture	11) Gallery
5) Magnification	12) Live View
6) Adjust Contrast Brightness	13) Sample Overview Image
7) Focus	14) System Settings

2. Select the Focus Icon and scroll the mouse scroll wheel to bring the optical image into focus.
3. Select the Contrast/Brightness button and use the scroll wheel to adjust the image's contrast. Select the icon again to adjust the brightness.
4. Use the small image displayed in the pane on the right to select the region of interest from the sample overview image. Click the  button to regenerate the overview image if it was out of focus.
5. Vacuum down the chamber and access the SEM View using the  button.

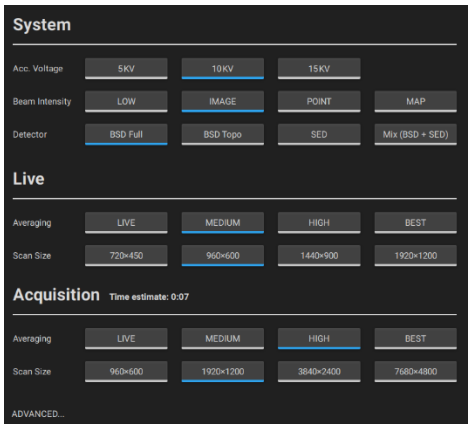
VII. SEM Imaging



Icon Legend

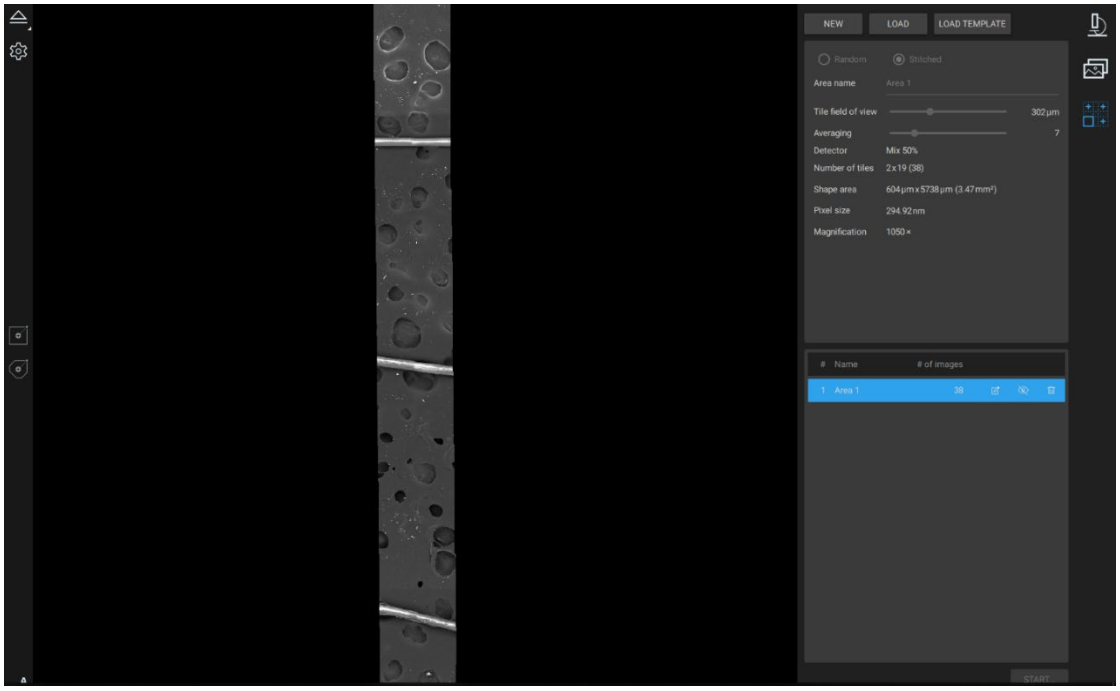
1) Eject	**8) Adjust Focus**
2) Settings	**9) Live Data Bar**
3) Capture Image	**10) Right Side Pane Expansion**
4) Optical Image Rotation	**11) Map Stitch Imaging**
5) Adjust Stigmation (Do not use)	**12) Gallery**
6) Magnification	**13) Live View**
7) Adjust Brightness Contrast	**14) System Settings**

1. Select the System Settings Pane on the top of the screen to change the settings of the acquisition. Here one can swap detectors, adjust voltage and beam intensity, and modify the live and acquisition image averaging/scan size.
   - **If using the gray holder (low vacuum imaging), scans are BSD only.**



2. Select the rotate icon to rotate the image using the scroll wheel
3. Stigmation is not needed for magnifications below 40,000x. **Only use stigmation if trained by facility staff.**
4. Select the magnification icon to go to greater magnifications using the scroll wheel.
5. The Auto Contrast Brightness icon can be selected to try and automatically optimize contrast and brightness settings for the image. If manual control is preferred, right click this icon to access manual control.
6. Select the Adjust focus icon to autofocus on the image. Manual control can be accessed by right clicking this icon.
7. Once the feature of interest is in view and in focus, select the camera icon on the left of the screen to capture an image.
8. To save points of interest across the sample, click the icon located on the right pane to save that position and quickly come back to it later.
9. To stitch together images of the sample, go to the mapping tab via the icon.

   Draw a shape around the area of interest using and then specify the settings for the stitched map. Hit start to begin the mapping scan and choose the save location.

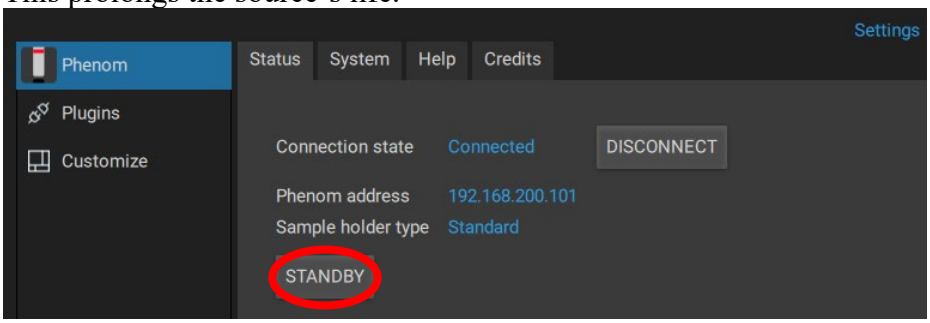


Example of stitched image mapping

10. To look through all images taken during this and previous sessions, select the gallery icon  .

VIII. End of Session

1. Exit SEM Mode and unload the sample using the  icon.
   - Right clicking this icon will give the option to return to the navcam instead of unloading the sample
2. Once the sample chamber is unlocked, carefully raise the chamber door and remove the sample holder. Return all stubs and holders to their homes, and discard any waste generated.
3. Close the sample chamber carefully, then select the settings icon.
4. Under the Phenom tab, select "Standby"
   a. This prolongs the source's life.



5. Collect all images from the desktop via flash drive.