

WORK INSTRUCTION BREAKDOWN SHEET

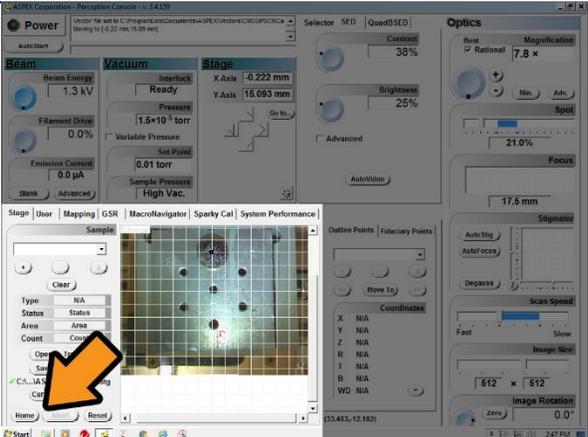
Operation: Scanning Electron Microscope (SEM) **Startup/Sample Loading/Shutdown**
Instrument: ASPEX EXplorer SEM

IMPORTANT STEPS	KEY POINTS	REASONS WHY
A logical segment of the operation when something happens to advance the work.	Anything in a step that might: 1. Make or break the job 2. Injure the worker 3. Be a Cultural Consideration 4. Make the work easier to do (i.e., “knack”, “trick”, special timing, or bit of special information).	Reasons for each key point.

VACUUM CONTINUITY POLICY FOR SEM (Read before proceeding!)

- **Sample Chamber** is under high vacuum.
- Computer and SEM are to be left **ON at all times** to insure continuity of vacuum per NanoLab policy.
- Rapid shift to ambient atmospheric pressure can cause significant damage to the SEM's components!
- Vacuum is maintained in **Sample Chamber** to minimize contamination and to decrease rate of oxidation of tungsten (W) **Filament**; thus, vacuum continuity maximizes the **Filament** lifetime.

Program Startup

	<ul style="list-style-type: none"> • Basic startup consists of computer / workstation preparation, followed by loading selected samples into the SEM. • If computer or SEM are OFF when starting session, notify NanoLab supervisor. 	<ul style="list-style-type: none"> • Computer and SEM are to be left ON at all times to insure continuity of vacuum. • Samples are to be prepared before opening the SEM vacuum chamber to minimize chamber exposure to contaminants. • Computer / programs should be started and prepared before “gloving up” to minimize transmitting contaminants from keyboard > samples > SEM.
<p>Start <i>Perception Console</i> application/GUI.</p>	<p>Double-click on <i>Perception Console</i> icon:</p> 	
<p>Connect to Aspex SEM “engine”.</p>	<p>Click on “Connect” in the Aspex SEM engine action window:</p> 	<ul style="list-style-type: none"> • This connects the computer running the <i>Perception Console</i> to the SEM itself via Local Area Network. • The <i>Perception Console</i> window title is “ASPEX Corporation – Perception Console – v. 3.4.159”
<p>When the <i>Perception Console</i> program opens, bring the <i>Stage</i> to its home position.</p>	<ul style="list-style-type: none"> • Press the Home button at the bottom left corner of the <i>Perception Console</i> under the <i>Stage</i> tab. • Doing this before opening the <i>Sample Chamber Drawer</i> helps minimize the time it is open and exposed to dust. • Note the small black dot in the gridded image on the left monitor indicating the position of the stage relative to the electron beam. 	

Sample Selection & Preparation

<p>Put on clean disposable nitrile gloves.</p>	<p>Always wear clean disposable gloves when working directly with components internal to the SEM.</p>	<p>This minimizes contamination.</p>
<ul style="list-style-type: none"> • Select samples to be examined under SEM. • Use sample stub tweezers to place sample stubs on the SEM's aluminum Sample Stub Holder. 	<p>Do not touch the sample surface to avoid contaminating or damaging the sample.</p> <p>The Sample Stub Holder is a square aluminum block with 7 alphabetized holes (A-G) kept in a clear plastic airtight box on the SEM body.</p>	<ul style="list-style-type: none"> • Use only SEM Sample Stub Tweezers to avoid dropping or damaging samples. • The tweezers are designed to engage with the concave bevel around the rim of the sample stubs. • Preparing samples before opening the SEM chamber, and keeping the Sample Stub Holder outside of the Sample Chamber Drawer helps minimize the duration the chamber is open and exposed to dust and other contaminants.
<p>Open the Sample Chamber to begin decreasing the vacuum.</p>	<ul style="list-style-type: none"> • Gently lift Sample Chamber Lever up to level indicated by label; vacuum will audibly breach with a "hiss" and the lever will halt. • DO NOT FORCE THE LEVER past this level. • Wait a few minutes while pressure equalizes. • When vacuum is equalized with ambient pressure, the handle will automatically rise to a vertical position and the Sample Chamber will lower to allow removal of the Drawer. • Avoid rapid shift to ambient atmospheric pressure as per <u>Vacuum Continuity Policy for SEM</u>. 	

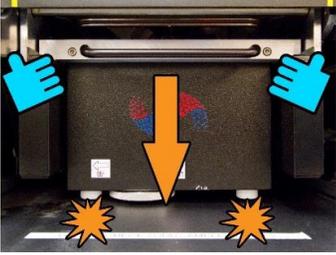
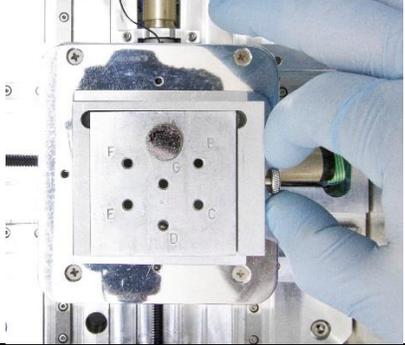
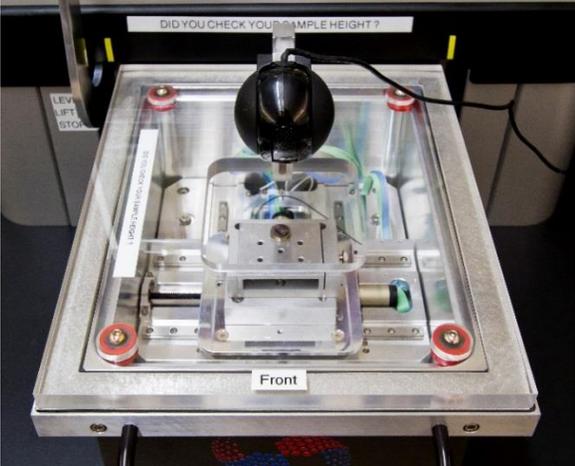
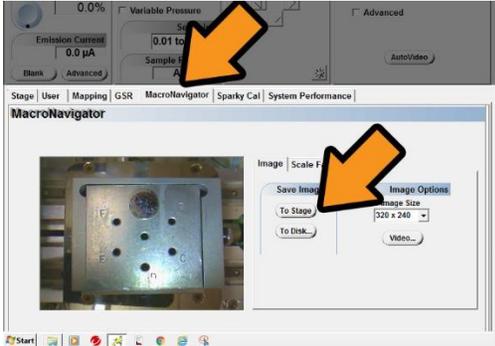
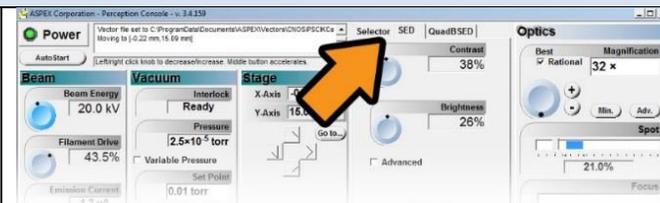
<p>Remove the Sample Chamber Drawer and lower it to the deck surface of the SEM.</p>	<ul style="list-style-type: none"> • Pull the Sample Chamber Drawer out by its handle until Operator can grasp its side rails. • Gently lift up to support the weight of the Sample Chamber Drawer by its side rails while withdrawing it from the enclosure (~11 inches total pull length). • Gently lower the Sample Chamber Drawer onto the deck of the SEM. • DO NOT strain or disconnect interior data cables between the drawer and the SEM. 	 <p>Allowing the Sample Chamber Drawer to quickly drop to the instrument deck may cause serious damage.</p>
<p>Load Sample Stub Holder into Sample Chamber Drawer.</p>	<ul style="list-style-type: none"> • Place loaded Sample Stub Holder on the “stage” in the Sample Chamber Drawer. • Secure Sample Stub Holder by tightening the Locking Screw on the right side of the Stage by turning it clockwise. • Do not overtighten Locking Screw for risk of damaging the assembly. 	
<ul style="list-style-type: none"> • Set the height of the Stage and the samples. • Insure that the top of the samples DO NOT touch the bottom of the Black Plastic Clearance Bar. 	<ul style="list-style-type: none"> • Rotate the metal Stage Height Adjustment Dial at the bottom of the Sample Chamber right (down), or left (up). • For imaging, adjust Stage height so the top of highest sample is ~3mm from the bottom of the Black Plastic Clearance Bar. 	 <ul style="list-style-type: none"> • If samples are too high in the Sample Chamber, they can collide with the SEM’s sensitive and fragile components, causing extensive damage. • The bottom of the Black Plastic Clearance Bar represents the elevation at which magnification is calibrated; ~3mm below it is the optimal working distance.

Image Capture for Macro Navigator

<ul style="list-style-type: none"> Remove Black Plastic Clearance Bar once safe sample height is verified. Place Logitech camera mount over the Sample Chamber Drawer. 	<ul style="list-style-type: none"> The Stage should already be centered in its home position from the STARTUP procedure. The red rubber bushings of camera assembly should seat in the corners of the Sample Chamber Drawer. The “front” label should align to the front of the drawer. 	
<p style="text-align: center;">Take new “To Stage...” image.</p>	<p>NOTE: The image from the previous SEM session will still be present in Stage Tab.</p> <ul style="list-style-type: none"> Select Macro Navigator tab. Click the (save image) To Stage button. The camera will then acquire an image. When the dialog box appears, enter the desired file name and click OK. Remove the Logitech Web Cam from the top of the chamber lip and place it back on the monitors’ desk surface. 	 <p>This image is used for approximate navigation around samples on Sample Stub Holder.</p>
<p>Check sample height against <i>Black Plastic Clearance Bar</i> again.</p>	<p>Repeating the step of checking sample height insures that it is not skipped, and reinforces the habit.</p>	<p>If samples are too high/close to the bar, this could cause significant and expensive damage to the electron gun.</p>
<ul style="list-style-type: none"> Remove Black Plastic Clearance Bar once safe sample height is verified. Place Sample Chamber Drawer back into SEM. Lift to engage chamber rails with internal receiver and gently slide chamber inside of SEM until it stops. 		<p>Do not slam or roughly reinsert the chamber, to avoid damaging the equipment.</p>
<p>Gently push the vacuum chamber lever all the way down.</p>	<p>After pushing down vacuum chamber lever, it should take ~3 minutes to get down to 10^{-5} Torr (indicated in the “Pressure” field of the Vacuum column of the Perception Console).</p>	<p>High vacuum is important as per SEM VACUUM CONTINUITY POLICY, to prevent atmospheric contamination of sample, and to minimize amount of air molecules which interact with electrons resulting in reduced quality of data/images!</p>
<p style="text-align: center;">Remove and dispose of gloves.</p>		<p>Minimizes transfer of contaminants between SEM, samples, and computer keyboard/mouse.</p>

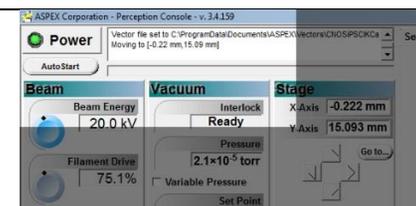
Turn on Electron Beam

- If not already selected, click the **SED** tab.
- Note that there will be no image on the right monitor when beginning an SEM session.



- The SEM has two detecting modes: **SED** and **QuadBSED**.
- SED is the default operating mode as it is easier to resolve a useful image when learning how to use the SEM.

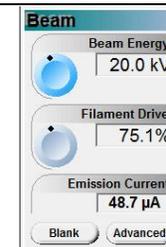
Press POWER button located in upper left corner.



This turns on the electron beam.

How do the Perception Console's controls work?

- All of the Perception Console's control "knobs" can be adjusted as follows: click and hold down the RIGHT mouse button to turn the knob clockwise (increase in value); click and hold down the LEFT mouse button to turn the knob counter-clockwise (decrease in value).
- The SPEED with which the knobs change the values can be increased by clicking and holding the **scroll wheel button** on the mouse ("middle-clicking") while turning the knobs.



If not already set, change the **Beam Energy** to 20k eV.

The maximum **Beam Energy** is 25k eV; Operator can adjust this value depending on goals of analysis.

Slowly increase the **Filament Drive** until the **Emission Current** value slows or stops increasing rapidly. NOTE: the **Emission Current** value will not display an increase until approximately 40% **Filament Drive** is achieved.

- **WITHOUT "middle clicking,"** increase the **Filament Drive** while watching the **Emission Current**.
- The **Emission Current** will typically "stall" or slow its increase after an image becomes visible on the right monitor; the **Emission Current** typically starts to plateau around 46-52 μA.
- It is not unusual to set the **Filament Drive** to a value in the 65% to 80% range.
- If **Filament Drive** value stops while holding down the right mouse button, release and restart the right mouse click.

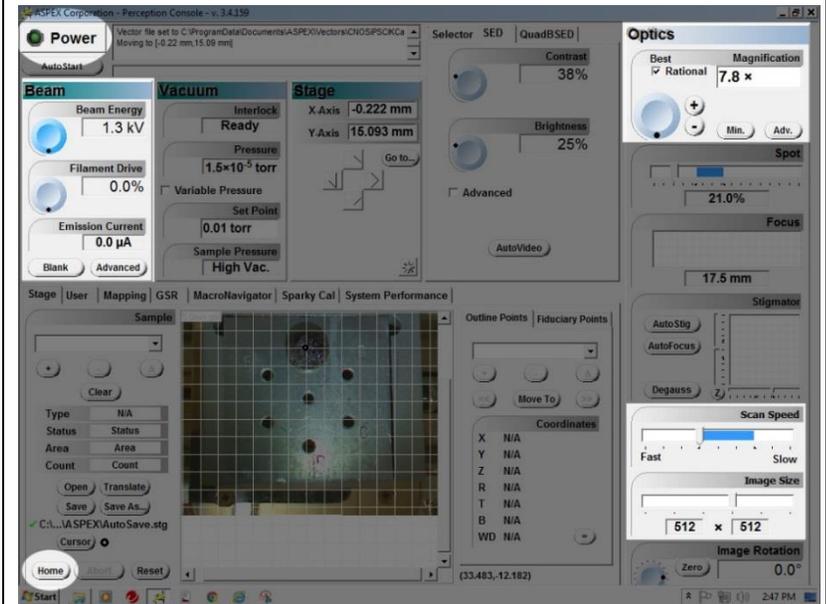
- Increasing the **Filament Drive** too quickly can overcharge the electron gun and risk damaging it or prematurely degrading its filament.
- Running the **Emission Current** too "hot" can damage the electron gun's filament and/or the specimen.
- Refer to graph/figure of "Optimal Saturation" on **Page 3-5** of Perception Suite User Documentation.
- A "bug" in the **Perception Console** program intermittently causes the **Filament Drive** value to appear to stop even though it is actually changing.

Proceed to "SEM Operations" Work Instructions (See Number: 02)

Shutdown Prep / Staging for Next Operator

Reset operating parameters to starting position.

- Increase/reset the **Scan Speed** to the “Fast” end of the blue bar.
- Decrease/reset the **Image Size** to 512x512.
- Reset the **Magnification** by clicking the “Min.” button; lower than 50x is an acceptable minimum magnification value.
- Reset the **Filament Drive** to 0%.
 - While the **Filament Drive** and **Beam Energy** should not be *increased* quickly by “middle-clicking,” it is harmless to *decrease* them quickly.
- **Power** off the **Perception Console**.
- Reset the **Stage** to “home”.



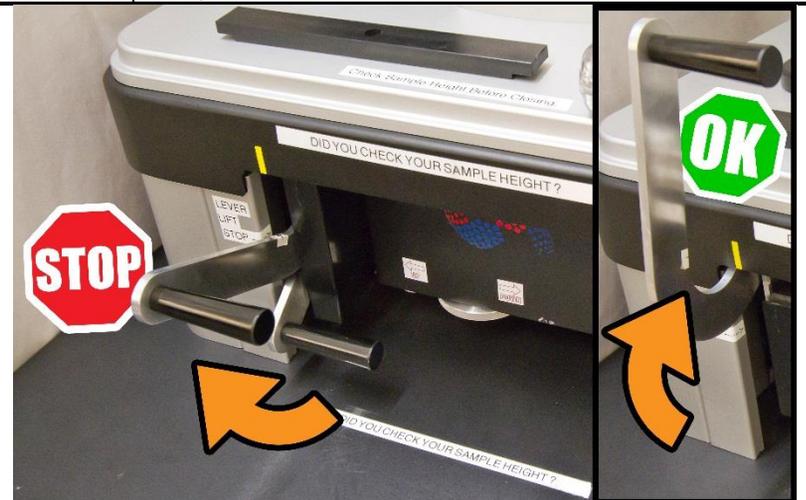
Put on clean disposable nitrile gloves.

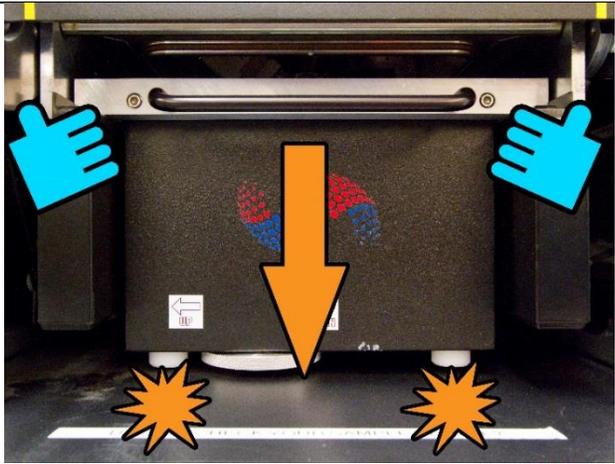
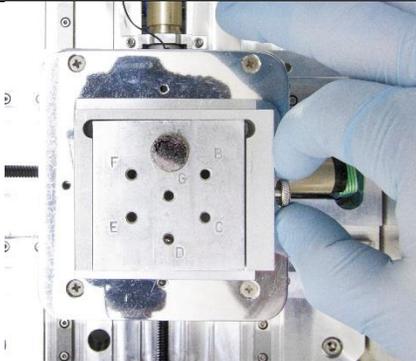
Always wear clean disposable gloves when working directly with components internal to the SEM.

Minimizes contamination of **Sample Chamber** and samples.

Open the **Sample Chamber** to begin decreasing the vacuum.

- Gently lift **Sample Chamber Lever** up to level indicated by label; vacuum will audibly breach with a “hiss” and the lever will halt.
- **DO NOT FORCE THE LEVER** past this level.
- Wait a few minutes while pressure equalizes.
- When vacuum is equalized with ambient pressure, the handle will automatically rise to a vertical position and the **Sample Chamber** will lower to allow removal of the **Drawer**.
- Avoid rapid shift to ambient atmospheric pressure as per Vacuum Continuity Policy for SEM.



<p>Partially withdraw the Sample Chamber Drawer from the vacuum chamber.</p>	<p>Gently pull the Sample Chamber Drawer by its handle <i>just far enough</i> so that Operator can easily access the sample stage Locking Screw.</p>	
<p>Remove Sample Stub Holder and reestablish vacuum quickly, but carefully.</p>	<ul style="list-style-type: none"> • Loosen the Locking Screw and remove Sample Stub Holder from SEM. • Gently push the Sample Chamber Drawer back into the vacuum chamber until it stops. • Lower the lever to reestablish vacuum in the Sample Chamber. 	 <p>Closing the Sample Chamber Drawer ASAP minimizes the chance for dust and contaminants to enter the SEM.</p>
<p>Transfer samples from the Sample Stub Holder to a temporary transfer stage.</p>	<ul style="list-style-type: none"> • Use only the designated tweezers to transfer the Sample Stubs from the Sample Stub Holder. • Replace Sample Stub Holder into clear airtight box. 	
<ul style="list-style-type: none"> • Gloves may now be removed. Remember to finish transferring your files and to take any personal USB drives with you. • Close the Perception Console program, but leave the computer on. 		