

Operating Procedures: JEOL JSM- 6010PLUS/LA Analytical Scanning Electron Microscope

Purpose and Scope:

This document describes the procedures and policies for using the MSE JEOL SEM 6010. The scope of this document is to establish user procedures. Instrument maintenance and repair are outside the scope of this document.

Responsibilities:

This document is maintained by the department Lab manager. The Lab Manager is responsible for general maintenance and for arranging repair when necessary. If you feel that the instrument is in need of repair or is not operating correctly please notify the Lab Manager immediately. The Lab Manager will operate the instruments according to the procedures set down in this document and will provide instruction and training to users within the department. Users are responsible for using the instrument described according to these procedures. These procedures assume that the user has had at least one training session.

Training:

The MSE Lab Manager will do all first and last training sessions, with the last training session being the 'drivers test'. Occasionally the Lab Assistant will be on hand for the practice session(s) in between. Users are NOT to train other people. Users found training other people will have their access revoked.

If users have not set up a CORAL account they must do so before the first session. SEM training includes **3 training sessions**. The first session will be an instrument demonstration and up to three (3) students can attend at a time. MSE specimen will be used for training. Sample preparation procedures for the trainee's specimens will be discussed.

The second session will be a practice session for one student with supervision. Students can bring their own specimen at this time as long as it has been prepared properly. If additional practice sessions are needed as determined by the student or MSE Lab Manager /Lab Assistant, they will be scheduled at the end of the session.

The last session will be the Drivers test that includes a short written test. Students will demonstrate their competence in operating the instrument. Users are required to have their own samples ready by the driver's test. If students do not pass this test they will be given the opportunity to come back for another training session. The student CANNOT take the Drivers test after taking a practice session on the same day. After passing the Drivers test users will be issued a license to operate the instrument. The checklist for the driver's test is included at the end of this document.

Since SEM operation is only reinforced by repetition, it is recommended that the student has his/her own specimens to work on immediately after passing the driver's test. The MSE Lab Manager will have discretion on when is the appropriate time to start training. Students will need to be recertified by the MSE Lab Manager after 6 months or more of inactivity on this instrument.

Access and use:

The MSE JEOL SEM was purchased with Student Technology Fee's. Please be sure to read department policies regarding the use of STF purchased equipment. This funding project is focused on providing quality SEM and EDS to undergraduates who cannot gain access to the other instruments on campus. These students will always have first priority to training and access. Graduate students and other researchers will be referred to other facilities on campus.

This is a very delicate, sensitive and costly instrument and will require extra care when operating it. In light of this, after hours use will not be available at this time. The scope will be available **Monday – Friday 9am – 6pm. Users who have access to these labs for other instruments who violate the hours policy will lose access to the labs.**

Users can save their data to a lab network share computer. The SEM computer is to operate the instrument only. It is not connected to the internet and USB drives are not allowed. There is software on the computer available to save pictures and spectra and users may use that to put together their results, but further analysis must be done on users own time. This will ensure that the instrument is available to the most number of users.

Once training is complete users can sign-up for up to 2 hours per session unless other arrangements have been made. Users who try to reserve 2 hour increments multiple times a day or throughout the week will have their reservations cancelled.

USERS MUST USE GLOVES: This instrument is a high vacuum system. Finger oils, dirt, etc. can compromise the vacuum and dirty the chamber. Users **MUST** wear gloves when handling any part of the instruments, samples, or sample holders. When doing other things, like typing please remove the gloves. Gloves are provided.

Features and Specifications:

Basic specifications are:

Resolution: 4.0nm (at 20kV)
Resolution in LV Mode: 5.0nm
Magnification: 8X to 300,000X (5X possible)
Accelerating Voltage: 500V to 20kV

There is not a motorized stage but there is an internal camera for position control. EDS is available but requires 10 hours of SEM use and a separate training session. Please note that the resolutions stated may not be the same for all materials. Most materials can be imaged in the SEM except for materials that are wet or that may outgas hazardous or corrosive gasses. Whenever possible samples should have a conductive coating. This will reduce deposits on the filament and extend the life of the detectors.

MSE has a number of standard sample holders available for students to use. Non-student users must supply their own sample preparation materials and holders but the holders must be of the correct type. Please see the Lab Manager for more information. Specimen preparation materials may be purchased from Electron Microscopy Sciences: <https://www.emsdiasum.com/microscopy/>.

Background

From MSE 313 Junior Lab Manual:

A scanning electron microscope (SEM) uses electrons as a probe just like an optical microscope uses light (photons) as a probe. The interaction of electrons with the object under investigation is utilized in imaging and chemical analysis and forms the subject of scanning electron microscopy. High resolution and large depth of focus coupled with X-ray chemical analysis makes it one of the most versatile instruments for microscopic examination and analysis of materials.

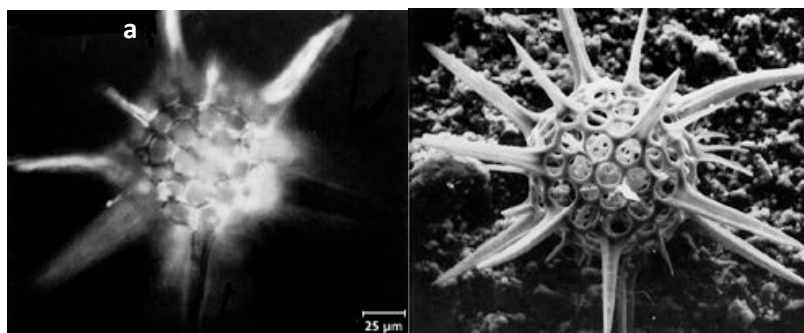
The interaction of a primary electron beam with a specimen leads to the generation of secondary electrons (SE), backscattered electrons (BSE), Auger electrons (AE), X-rays, cathode luminescence and other phenomena. The different types of electrons and energy quanta, mentioned above, arise from different depths of the material and provide microscopic information about the material.

The other feature of scanning electron microscopy is **resolving power**, which is the smallest detail that a microscope can resolve, or "see". The resolving power of electron microscopes is orders of magnitude better than that of an optical microscope because the wavelength of the probing beam is orders of magnitude smaller. The resolving power of a microscope is inversely proportional to the wave length of the radiation or particles constituting the probe. Since electrons have much lower wavelengths than light, it is possible to resolve finer details of specimens using SEM. The depth of focus of a SEM is much higher than an optical microscope. Depth of focus is defined as the ability of a microscope to bring the features of an object at different depths into focus.

Optical vs. Electron Microscopy

The attractive feature of optical microscopy is that it is so easy: samples can be analyzed in air or water, the images are in natural color with magnifications of up to one hundred to one thousand times, and modern semiconductor electronics with charge-coupled devices (CCD) allow image processing.

The optical microscope should dominate the field but it doesn't. The scanning electron microscope (SEM) is the microscope of choice because of its depth of focus and resolving capability. Examination of Fig. 1 shows a striking contrast between an A) optical and B) SEM viewgraph of a radiolarian at the same magnification.



A

B

Figure 1. (Taken from J.I. Goldstein et al., eds., Scanning Electron Microscopy and X-Ray Microanalysis, (Plenum Press, NY, 1980).)

In the optical micrograph taken at high resolution only a section of the radiolarian is in sharp focus. In the SEM image the whole specimen is in focus.

For the optical microscope, the depth of focus is the **distance** above and below the image plane over which the image appears in focus. As the magnification increases in the optical microscope the depth of focus decreases.

The three-dimensional appearance of the specimen image in (b) is a direct result of the large depth of field of the SEM. It is the large depth of field in the SEM that is the most attractive feature of the SEM. This field arises because of the method in which the data is obtained with a fine electron beam scanned over the surface and with the detected secondary electrons forming an image on the "TV"-like monitor.

The limit on what size specimen can be resolved -- irrespective of instrument type -- is set by the wavelength. The wavelength of the visible light used in optical microscopes is between 400 and 700 nanometers (nm). The resolving powers of high-quality light microscopes are limited by the wavelength of imaging light to about 200 nanometers (0.2 microns). Scanning electron microscopy uses electrons with energies of a few thousand electron volts (eV), energies a thousand times greater than that of visible light (2 to 3 eV). The wavelength λ is given by equation 1 below.

$$\lambda = h / \text{momentum}$$

where h is Planck's constant. For 3600 electron-volt electrons, the wavelength is 0.02 nanometers. The resolving powers of ordinary electron microscopes is 1 nm (above the limit because construction details determine resolving power), a value which can be pushed to 0.1 nm.

Depth of focus and resolving power draws one to the SEM, and operating maintenance as well as vacuum requirements drives one away. Electrons are lightweight (1/1836 the mass of the proton) and are scattered or absorbed in air. The sample chamber of a SEM must be a vacuum, which limits the sample size to a few centimeters on edge. Electrons carry charge ($e = 1.6 \times 10^{-19}$ Coulomb) so samples must be covered with a conductive coating if they are non-conductive (most ceramics and polymers). Finally, the SEM is capital intensive, e.g., expensive, requires maintenance and laboratory space.

Basic Operating Procedures:

Please be sure to read this document in full before taking the Driver's test.

The SEM Chamber operates in high vacuum (10^{-4} – 100 Pa) so users should observe high vacuum protocols. Users must always wear gloves when handling anything that will be in the chamber. When users finish loading (or unloading) a specimen their gloves should be removed. Remove gloves when handling items that do not go in the chamber because oil and dirt on fingers are transferred to the doors, keyboards, vacuum control buttons, etc. and then to the surface of the gloves, rendering the gloves useless.

Specimen preparation materials like carbon tape and specimen stubs can be purchased from a number of vendors. MSE maintains these supplies for undergraduates only. All other users must supply their own. Ideally users will have their samples prepared before coming to the lab. **Users must have their own prepared sample for the driver's test.** Users are encouraged to have their own 'kits' so they know that their materials are uncontaminated. However, there are tweezers, gloves, a ruler and other necessary tools for imaging.

The SEM computer is for operating the SEM ONLY. Do not install software of any kind, or attempt to uninstall software. Do not input a usb drive or a dvd into the computer. All data is saved to a shared

folder and can be retrieved from the network share computer in the lab. There is also a log file that must be filled out.

1. Turn on the computer monitor. The JEOL software (In Touch Scope) should be open and running. A dialog indicating that the system is in Eco mode should be up. Select the cancel button.
2. Check the following:
 - a. N2 gas pressure
 - b. EVAC is on
 - c. HT (beam) is off
 - d. Take care not to fall on or bump the instrument. It is mounted on rubber feet to reduce vibrations. The turbo pump is attached just below the instrument and if it gets tilted enough it can damage the pump.
3. There is a N2 gas tank connected to the instrument. It would be prudent to check that the tank is still full. **Do not make any adjustments to the pressure.**

Sample (specimen) preparation:

- A wide range of types of specimen can be imaged in this SEM due to the low vacuum setting. However, certain types of materials must never be placed in the instrument:
 - Wet samples – either from water, or a solvent
 - Samples that outgas water vapor (porous materials) or corrosive or toxic gases.
- Specimen that are not conductive may be imaged in the low vacuum mode, however, if they can be coated they should be. Images will be better and the chamber will stay cleaner. If you need information about conductive coating, please contact Hanson Fong (hfong@uw.edu) or Tuesday Kuykendall (tuesday@uw.edu)
- Regardless of the type of material, all specimen should be kept in an evacuated desiccator or drying oven (50 C +) for 24 hours before loading in the chamber. Users must gain permission before putting anything in the desiccator or drying oven in MUE 167.
- Specimen are mounted on the surface of a stub with 2-sided carbon tape. Do not use any other kind of tape. Specimen **MUST** be mounted in the standard sample holders (figure 2). Specimen should never be mounted directly on the specimen stage.



Figure 2: Sample preparation tools

- There are a variety of mounts available. Specimen can be mounted on one stub or 4. Odd shaped specimen or specimen with a diameter greater than 10 mm (but less than 32 mm)

may be mounted on the large stub. Samples should not be over 10 mm tall. Sample surfaces should be as flat as possible.

- If there are 2 – 4 specimen then use the 4 stub holder. However, it is important that **all of the specimen are the same height**. If you are unsure of how to prepare and load your specimen please stop and ask.
4. Once your sample is prepared, mounted, and ready to install, select and hold in the ‘Vent’ button on the vacuum mode control until it flashes (figure 3). At this time you may ‘open’ the camera. (Please see “Instructions for using the IR-Camera” on page 11).

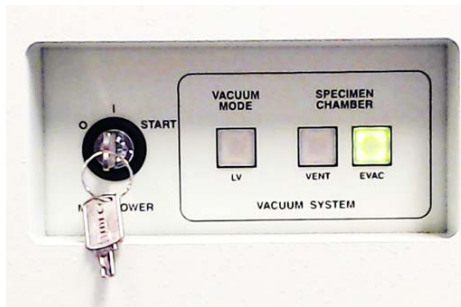


Figure 3 Front panel Vacuum Control: **NEVER TURN THE KEY**

5. About 10 seconds before the chamber is fully vented a dialog (figure 4) will pop up on the computer monitor asking for the height of the specimen above the specimen holder. Enter the height. **DO NOT** proceed if you’re not sure or have forgotten how to measure the height. **You must do this even if you are using the camera.**

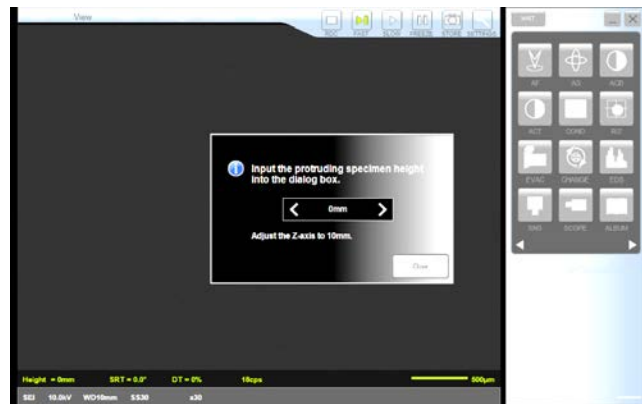


Figure 4: Dialog pop-up just before chamber is fully vented.

6. **VERY GENTLY** check that the specimen stage door is ready to open – (usually about 1 – 2 minutes after pressing the vent) It should slide forward very easily. If it doesn’t wait a few more seconds.
7. Load your mounted specimen. The holders have a dovetail slide (Figure 5) on the bottom that fits over the raised circle in the center of the stage. There is a straight edge on the stage and the

holder for orientation. Make sure you load in such a way that your hand does not go into the chamber.

- a. When changing out multiple samples please evacuate the chamber between changes. Never leave the chamber open in the room any longer than it takes to load or unload a sample.

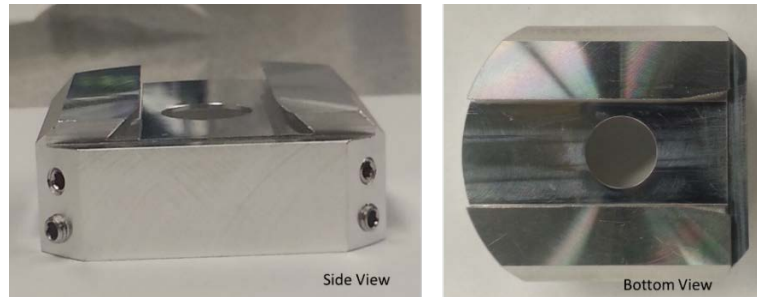


Figure 5 Side and bottom of specimen holder showing the dovetail slide.

NEVER REMOVE THESE FROM THE LAB!

8. Adjust the stage position using the XY knobs and note the specimen position and height.
9. For the MSE sample holders the top of the holder is in the same plane as the working distance of 10 mm when the Z axis is set to 10 mm. If the specimen is flush with the holder then move the Z-axis of the stage up to 10 - 11 mm. If the stub/specimen assembly is above the top of the sample holder then measure the specimen from the top of the sample holder to the highest point of the specimen. Add the measured value to 10 mm and that is the highest the Z axis should ever go. **This measurement must be very accurate and precise.** Z-axis is NOT working distance. Think of the adjustment of the z-axis as moving up and down under the detector.

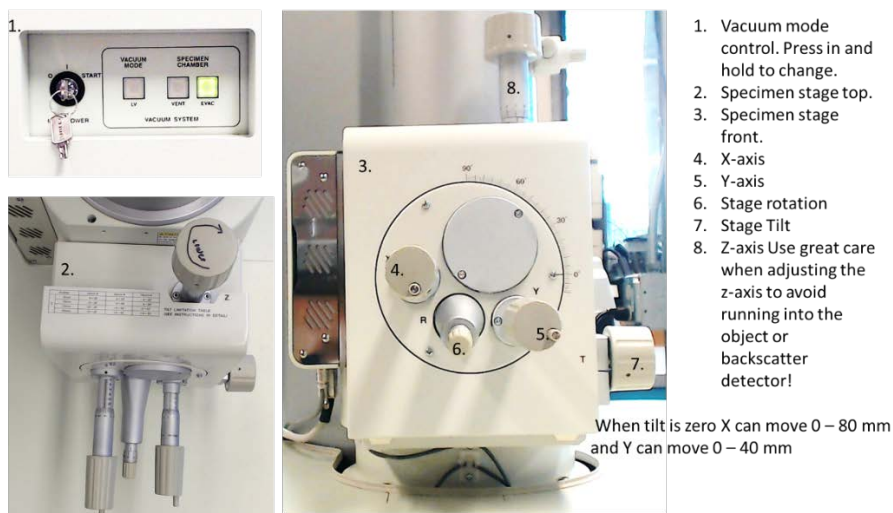


Figure 6: Manual Controls

10. When the sample is in place gently push the stage door back in – WATCH the stage as it moves back in place to see where it is with respect to the detector. If your specimen or any other part

hits the detector it will cause **catastrophic** damage! The highest point on the surface of your sample must be at least 10 mm below the detector (the 'cone').

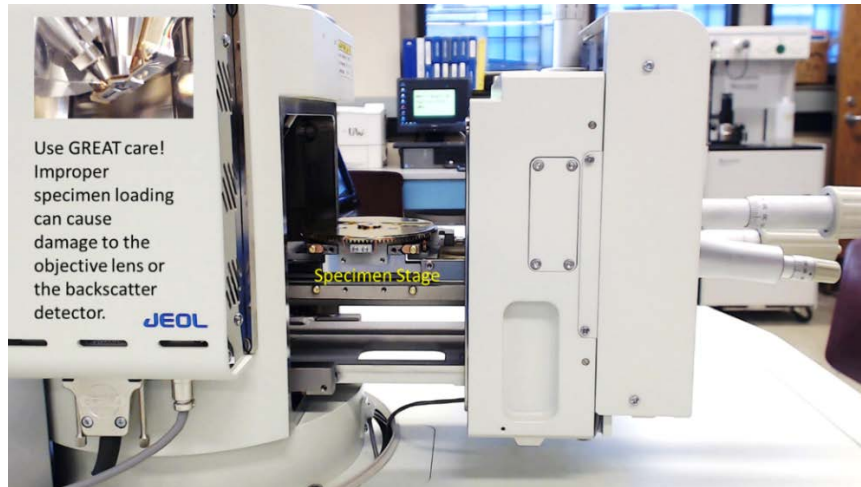


Figure 7: Open stage and Objective

11. Hold the door closed and press and hold the EVAC button on the vacuum control until it starts flashing. Continue to hold the door closed until after you hear 3 'clicks'. It will take about 1 minute to evacuate to a pressure that is safe for the beam. Be sure to wait for the green "Off" icon on the software before proceeding.
12. You may adjust the z-axis using the camera at this time keeping in mind the offset you measured.

Software:

1. The JEOL software should always be on and open. There are three main sections (Figure 8). Navigate between the three sections using the left/right arrow keys in the center.

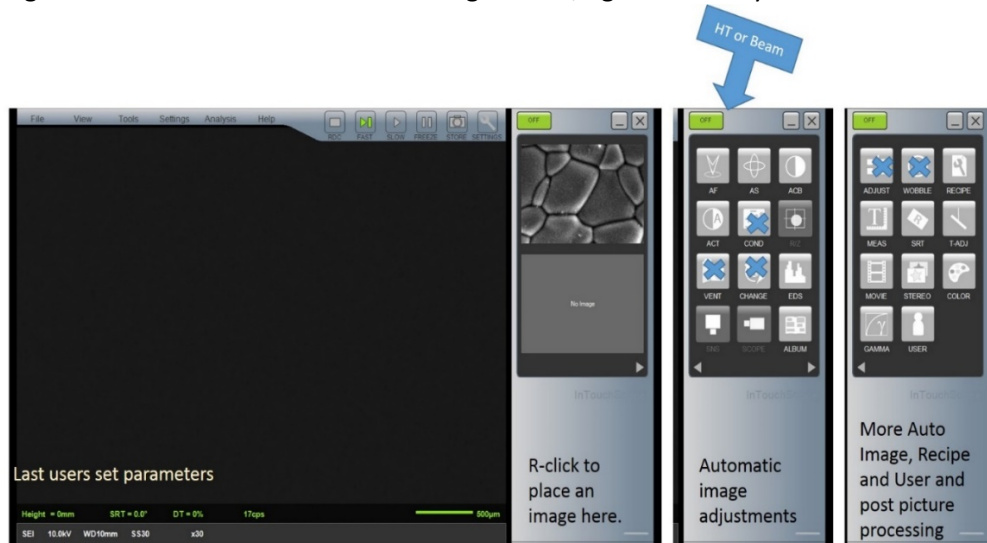


Figure 8: Software user interface

2. Users may adjust the parameters by selecting them from the bottom of the screen (white text).

3. When the chamber has evacuated to a safe pressure the on/off/wait button (upper RH side) will be green and off. **Wait an additional 5 minutes** then press the button to turn on the beam. This is also a good time to open the camera (see camera operating procedures). Note the increase in the count rate. This should not exceed 150 counts!
4. If there is no image, press the ACB (Auto contrast and brightness). An image should appear. If the magnification is at 30X you will see the round outline of the objective. Increase the magnification to 50X.
5. You may make a small adjustment in the Z-axis by manually raising the stage. Do this with great care using the camera. Never adjust the Z-axis 'blind'! Do not exceed the 10 mm plus offset established earlier. Once you are satisfied that the Z axis is in the correct position increase the magnification. Do not adjust the Z axis again. Use the camera to determine the distance of the top of your sample from the detector. When you have focused at 100X minimize the camera and verify that the counts have gone back down.
6. It is usually preferable to get a focused image at a higher magnification then lower the magnification for imaging. A step – by – step guide is available.
7. There are a number of automatic features in the software that are not used since we don't have a motorized stage. Some of the operations selections on the main page are not used because they cause unexpected behavior – using them runs the risk of having the beam turn on or the chamber venting when the user is finished. (see figure 8, crossed out buttons).
8. The SEM comes equipped with a traditional operation keyboard (figure 9):

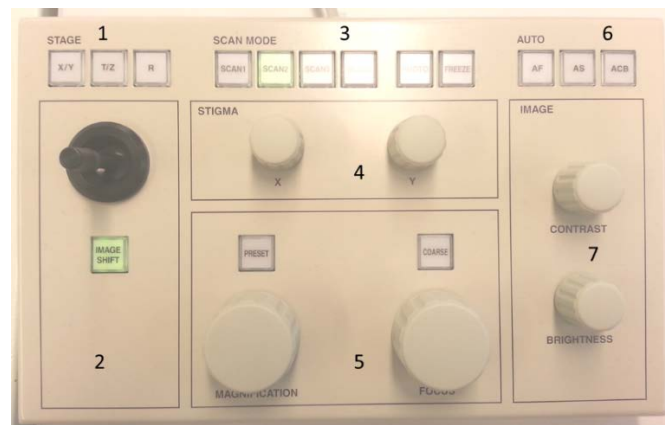


Figure 9: Operations Keyboard

9. At very high magnifications its usually more advantageous to use the manual knobs for Magnification and focus (5), stigmatism (4), and contrast and brightness (7).
10. In addition to the auto-processing in the software, there is a AF, AS, and ACB (6) on the keyboard as well. Scan speed can be selected here as well (3). Scan 1 and 2 are fast scans and resolution isn't very good but it's easier to see any adjustments or movements.

11. Astigmatism will also need to be adjusted. Users will have an opportunity to practice this. The only way to learn how to obtain an ideal image is to practice.
12. Scan 3 is much slower but the image is better. Scan 4 and Photo both acquire the image to be saved. This freezes the image.
13. Select scan 2 or 3 to unfreeze the image.

Getting the best image and obtaining valid spectroscopy data takes practice and is often material/sample dependent.

Shut down procedures:

1. Turn off the beam.
2. Press and hold the vent button until it flashes. When the height offset dialog pops up close it (unless you are installing a new sample then follow start-up).
3. If you are finished, make sure to close the height input dialog. If it is left on it can cause the software to freeze.
4. Open the chamber door.
5. Lower the stage
6. Turn the stage so that your hand enters from the side.
7. Remove the sample holder. (with gloved hand) and carefully remove your sample. NEVER leave a sample in the chamber.
8. Close the chamber door and press the EVAC button until it flashes.
9. Please leave the area clean for the next user.

NOTES:

- Never leave the chamber vented
- Minimize the amount of time the chamber door is open.

Abbreviations

SEI - Secondary electron image

BSE – Backscattered Electrons

kV – Energy of incident beam

WD – working distance

SS – Spot Size

For an in-depth treatment of SEM and the theories of operation please read:

Goldstein, Joseph et.al; Scanning Electron Microscopy and X-Ray Microanalysis, 3rd edition, Springer Science, 2003 ISBN: 978-0-306-47292-3

Instructions for using the IR-Camera

- Load your sample as instructed.
- The camera window is minimized on the task bar. When the camera is minimized the light source is off. When the camera is called up the light source comes back on. The control box for the camera is sitting behind the beam tower. There are a series of lights on the front that indicate when the camera is on and at what intensity.
- The camera can be on with the beam on or off but bear in mind that the light source on the camera is increasing the intensity counts on the detector.
- Keep the intensity of the camera low enough that the cps (counts per second) do not exceed 100 cps (status line in green on the bottom of the screen).
- Keep the spot size below 30 when the camera is on.
- NEVER have the camera on when in EDS mode. This will damage the detector.
- When adjusting the Z-axis never go past your measured offset, but also carefully observe the location of the top-most position of your sample.
- For using the camera with the tilt function please see Tuesday or Hanson.

Certification Checklist for STF-JEOL 6010 SEM

Date: _____

User Name: _____

Email: _____

Supervisor: _____

Department: _____

Tested by: _____

Test Specimen: _____ **NOTE: User must have their own sample for certification.**

<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User filled out the user log on the work share computer
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	Policies are read and understood
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	Sample is properly prepared - it is not wet, corrosive or a hazardous material.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	Start-up procedures are followed.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	Sample specimen is properly mounted.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	Sample specimen height measured and noted.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	Specimen height offset has been entered in height record in computer
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User can correctly load specimen onto stage.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User understands the relationship between working distance and specimen height offset.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User turns Z axis up or down correctly.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User is able to correct stigmatism.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User is able to record an image at 20000X using spheres sample within 30 minutes of turning on beam.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User follows shut down procedures
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	Sample specimen/holder is properly extracted from chamber.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	Chamber contamination is minimized throughout user interaction.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	SEM work area is cleaned up
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User knows how to save and retrieve images and data.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User completed the written portion of the Drivers test.
<input type="checkbox"/>	Yes	<input type="checkbox"/>	No	User has signed contract