



Dickinson College Department of Geology

Title: **BASIC OPERATION OF THE SCANNING ELECTRON MICROSCOPE (SEM)**

Equipment: **JEOL JSM-5900 SCANNING ELECTRON MICROSCOPE**

Revision: **2.2**

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Running the SEM: General Instructions

1. Starting up the Machine (this only needs to be done if the machine has been shut down)

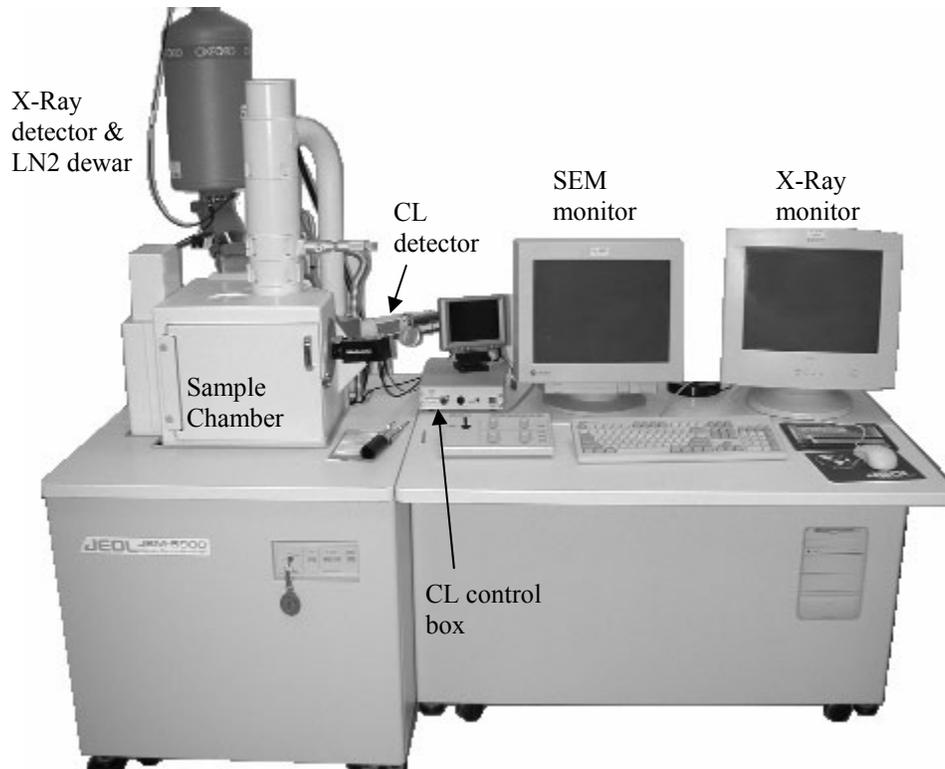
Open valve on water supply. Turn on chiller. Check to see that the water chiller is running at a temperature of 65-70 degrees. The chiller is the humming box next to the SEM--it circulates water to cool the diffusion pump.

Turn SEM key to start, and then let go. Boot up left computer. Check to see that the 'EVAC' button on the front of the SEM column table (to the left of the computers) is illuminated green and not flashing.

ALSO: If you come into the SEM lab and there is an' obnoxious alarm buzzing, it means that the liquid nitrogen that cools the X-ray detector is running low. Immediately contact Jeff Roth (x1109), or Ben Edwards (x8934).

2. Familiarize yourself with the 'One Keyboard, One Mouse' system

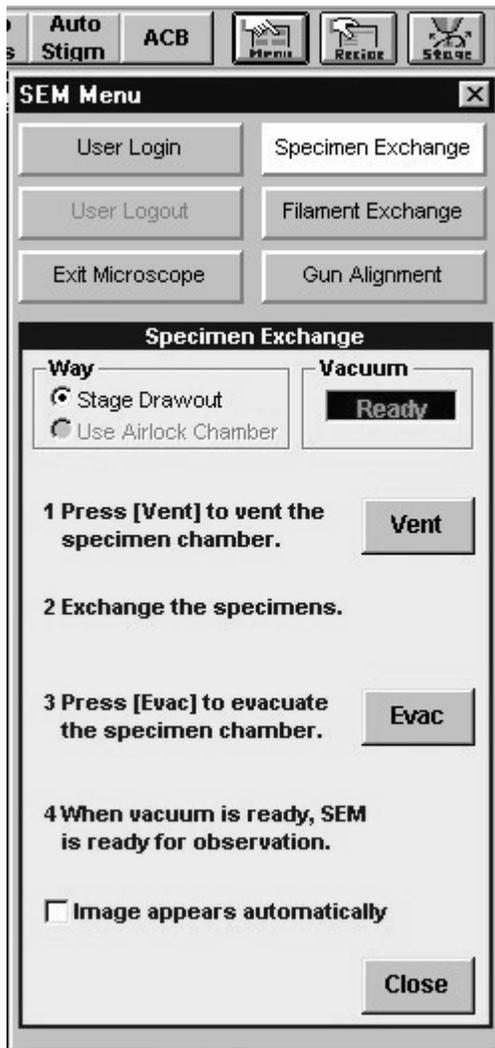
There are two computers on the SEM, but only one keyboard and mouse. The monitor for the SEM computer is on the left - this is computer #1. The monitor for the x-ray analyzer is on the right - this is computer #2. The red number on the gray box on the right-side table (below the printers) always indicates which computer the monitor and keyboard are working with. It is possible during start-up or shutdown, that the keyboard and mouse will be directed toward the wrong computer; to fix this, simply hit Ctrl-Alt-1, to switch the keyboard and mouse to computer #1 (the left computer), or Ctrl-Alt-2, to switch the keyboard and mouse to computer #2 (the right computer).



3. Loading Your Sample



Click on the JSM-5900 icon on the SEM computer desktop. This will bring you to the software interface for the SEM.



The first thing you will want to do is to load your sample. Do this by clicking on the **Stage** button  in the upper-right corner. Then select **Initial Position** at the bottom of the menu. When the sample

stops moving, select **OK**, and then click on the  icon in the upper-right corner. Click on **Specimen Exchange**, and then on **Vent** (see figure at left). This will vent the sample chamber, and allow you to open the sample chamber door.

Never reach into the sample chamber without gloves on! Use the sample exchange tool (if possible) to switch your sample with the one that is in the chamber. Put the sample you just removed into the metal desicator. If you are not familiar with the sample exchange tool or how the sample holders fit onto the stage, please be sure to ask for help. The small screw on the bottom of the sample holder should be facing you when you insert it into the sample chamber – this acts as a stop.

Once you have your sample on the stage, and before you close the sample chamber door, you should move the stage so that your sample is located directly under the pole piece, which is the place where the electron beam leaves the column. Use the joystick to move the sample stage in the X-Y direction (X is -right-left; Y is - backward and forward). Hit the 'X-Y' button (it will turn yellow) and the joy stick will move the stage in the 'X-Y' directions. You may also use the stage menu by hitting the blue **Stage** button in the upper-right corner of the screen.

The 'Z' direction is the vertical position of the stage. For a 10 mm working distance, and using the thin section holders, the 'Z' position should be about 8.0 mm. If you are using other sample holders, the appropriate 'Z' position may vary significantly. Adjust the 'Z' position of the stage using the joystick and/or the 'Stage' menu.

Be sure that the 'R' (stage rotation) and 'T' (stage tilt) positions are at approximately zero (they often are off by only a few tenths of a degree-this is ok).

Once your sample is secure on the stage and positioned approximately where you want it, close the door, latch it, and click the **Evac** button on the computer. This will begin the sample chamber evacuation process. Notice that the Evac button on the front of the SEM column table (left of the computers) is now flashing. After about

5-6 minutes it will stop flashing. At that point, the sample chamber is under vacuum, and you are ready to begin viewing your sample.

4. Getting a Secondary Electron Image

Set Your Operating Conditions (typical conditions below)

Accelerating voltage: 15 kV

Working Distance: 10 mm.

Signal: SEI (Secondary Electron Image)

Spot Size: ~30-35.

Magnification: Click on 'View' (upper left) to set the system at the lowest magnification possible at 10mm working distance (this will be 90-100X).

Consult the literature to determine the specific appropriate operating conditions that may be required for the types of samples that you are working with and the kind of imaging that you are doing.

Click on  to start bombarding your sample with electrons.

At this time, you should enter your information and the time-on into the SEM logbook.

Getting Focused: If you are lucky, an image of your sample will already have appeared on the screen. More likely, you will have to adjust the focus, contrast, and brightness to get an image. Do this by adjusting the contrast and brightness up or down until you reach the crossover between a blank dark screen and a blank bright screen. Start by clicking on 'Contrast' and dragging the mouse upward (if you are trying to brighten the screen) while you hold the mouse button. A left-click is a fine adjustment, and a right-click is a coarse adjustment. After doing this once for 'Contrast', do it again for 'Brightness'. After several coarse adjustments (right-click-and-drag) to both 'Contrast' and 'Brightness', you will find the place where the image goes from blank and dark to blank and bright. Use the fine adjustments to get a fuzzy gray image that shows some contrast (i.e., is not totally flat gray).

Once you have that fuzzy gray image, use the 'Focus' button to sharpen the image; right-click-and-drag is a coarse adjustment, left-click and drag is a fine adjustment. **(If you are going to do X-Ray work, you won't want to touch the focus after you have set the Working Distance (WD) at 10mm. Consult the X-Ray directions to see how to do this.)** As you adjust the focus, you will also have to go back and re-adjust the contrast and brightness. Working with these three adjustments (contrast, brightness, focus), you should be able to get a good image pretty easily.

Saturating the Filament: Once you have a reasonable image, you should take a minute to saturate the tungsten filament (as in a light bulb) that produces the electrons that are bombarding your sample. The purpose of the filament saturation procedure is to ensure that current passing through the filament is not too high, a condition that will bum out filaments quickly, and not too low, a condition that will prevent you from getting good quality images.

It may help to darken the room for this procedure.

Go to the  button and select . You adjust the filament current using the first scroll bar labeled **Filament Heating**. You can make very coarse adjustments by dragging the sliding button left-and-right, but DON'T DO THIS because the rapid changes in filament current will shorten the life of your filament. You can make finer adjustments by clicking inside the scrollbar, between sliding button and the arrows, but DON'T DO THIS EITHER, again to prolong the life of your filament. To make fine adjustments, click on the 'right' and 'left' arrows themselves. The left arrow will slowly lower the filament current and the right arrow will slowly increase it.

Click the 'left' arrow several times and you will notice that your image begins to darken. Click the 'left' arrow several times more so that the image darkens significantly. Now switch to the 'right' arrow and increase the current. You will see the image get significantly brighter at first, but after several clicks on the right arrow, the increase in brightness will be much less noticeable. **After several clicks** on the right arrow with no increase in image brightness, switch back to the 'left' **arrow** and turn the filament down ONE CLICK AT A TIME until you see the image darken. At the point where the image first noticeably darkens, the filament is properly saturated.

Adjusting the Filament Tilts: After changing filaments, and only occasion otherwise, it may be necessary to adjust the physical tilt of the filament in the gun assembly. The tilts are adjusted in the 'X and 'Y' directions using the scroll bars immediately beneath the Filament Heating scroll bar under the Gun Alignment menu (see 'Saturating the Filament' above). To adjust the tilts, click on the sliding button and shift it slowly left or right, and observe the brightness of your image. The idea is to adjust the 'X' and 'Y' tilts to maximize the image brightness (i.e., maximize the signal).

Note About SEM Filaments: The filaments purchased directly from JEOL are prealigned in the factory, and require very little in the way of tilt adjustment or manual centering. Third-party or 'retipped' filaments (reconditioned) are less expensive but will be much less well behaved. Purchasing these less expensive filaments will greatly complicate the filament change process to the point where the savings is not likely to be worth the headache and likely decay in the performance of the instrument due to -poorly aligned filaments (i.e., stick with the JEOL filaments!).

Changing Magnification: Depending on your sample, you may also have to adjust the magnification to get the image that you want. An easy place to adjust the magnification is by using the knob set that sits between the computer monitor and the keyboard (notice that many of the adjustments can be made either through the computer or by using the knobs).

For most work on geological thin sections, you will want to be at the lowest magnification possible, which will be around 90-1 00X. If you click on 'View' in the upper left of the computer screen, you will toggle between the lowest magnification possible at the operating conditions that you have set, and the last higher magnification position that you used.

Adjust the magnification to where you want it, and continue adjusting the focus, contrast and brightness until you get the image that you want.

Final Focus: Your final focus adjustments should be made at a magnification that is significantly higher than the magnification at which you will be working. So for thin section work at about 100X you should make your

final focus at a magnification of 2,000-4,000X. If you get a reasonably sharp image at this magnification, the image will look perfect at 100X, which is where you will actually be working.

Stigmation: While you are still at high magnification, and once you have made your final focus adjustments, you should adjust the 'Stigma-X' and 'Stigma-Y' to try to improve the focus slightly. Moving between fine focus and Stigma adjustments at high magnification may provide small, but sometimes significant improvements to the quality of your image.

Can't Get a Sharp Image? Check the following in order, -"

- Check the filament saturation (see 'Saturating the Filament' above)
- Go through the 'Final Focus' and 'Stigmation' procedures (see above)
- Align the final aperture (see separate instructions under 'General Maintenance')
- Repeat the 'Final Focus' and 'Stigmation' procedures (see above)

If you still can't get a good image, and if the SEM has been used fairly heavy for several months, it may be time to switch apertures. This procedure is described under 'Alignment of Final Aperture' in the 'General Maintenance of the SEM' (separate document).

If all else fails, call the JEOL service guys in Landover, MD (570-490-2777).

5. Getting A Backscattered Electron Image

So far the images you have created are 'secondary electron' images. These are the images that show 3-dimensional details in high resolution. If you are interested in the shapes and 3-dimensional aspects of your sample, you will probably always use the secondary electron detector, which is the **SEI** selection under the Signal operating condition. However if you are interested in the composition of your samples, and if you plan to do X-ray work, you will probably want to learn to use 'backscattered electron' images.

Once you have a secondary electron image on the screen, you can switch over to the backscatter detector by selecting BEIW under the  menu (make sure that the infrared camera is turned OFF, or you will not get an image). When you do this, you will see a small box in the bottom-right that is labeled BEIW with the selections 'Compo', 'Topo', and 'Shadow', with scroll bars for contrast and brightness at the bottom. Select 'Compo' and adjust the brightness and contrast until you get an image that shows contrasting shades of gray. To get a good backscatter image that brings out relatively subtle compositional contrasts, you need a strong signal--a Spotsize of about 60 seems to work pretty well (the spot size will probably already be at 60 from the last user).

The bright areas in a backscatter image are made of relatively high atomic mass elements, and the dark areas are made of relatively low atomic mass elements. In rocks, this translates into the bright areas that are Fe-rich (olivine, pyroxene) and dark areas that are Fe-poor, and Si-rich (feldspars and quartz).

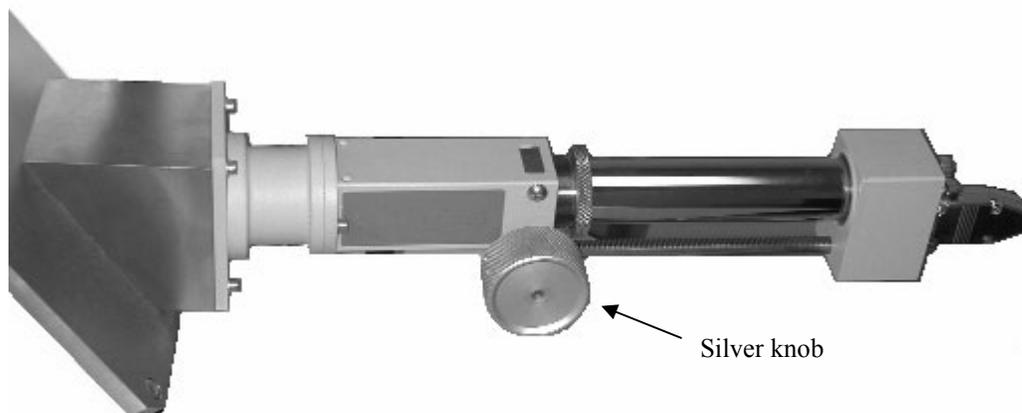
REMEMBER that you won't see different shades of gray if your sample is compositionally uniform.

REMEMBER ALSO that you won't see anything if the infrared 'ChamberView' camera is on while you are trying to get a backscatter image (the signal from the camera interferes with the backscatter detector).

If you switch down to the 'Shadow' setting under 'BEIW' you will notice that the image takes on a 3-dimensional look. This feature, which seems to work well at the factory setting of 10 (check the number next to the word 'Shadow' under the 'BEIW' menu), provides an excellent sense of both compositional and topographic variation across a sample.

6. Getting a Cathodoluminescence Image

In order to obtain a cathodoluminescence (CL) image, the CL detector must be moved into place. Great care must be taken here not to crash the CL detector into the stage. Therefore it is recommended that the chamber be vented, and the sample holder moved down, so that you can look into the chamber while moving in the CL detector (this can also be done by using the infrared camera, but the oblique perspective makes it difficult). The CL detector is moved into place by turning the large silver knob on the side of the arm labeled Centaurus.



Once the detector is in place, bring the stage up fairly close to the detector, being careful not to hit it. If you vented the chamber, close the door, and reevacuate the chamber. Using secondary electron imaging (SEI), you can fine-tune the orientation of the CL detector – the electron beam to shoots through a hole in the detector arm, so that you can get an image. You will have to adjust the focus to get an image as you have changed the z-position of the sample holder considerably.

Once you have a focused secondary image, turn on the CL detector by pressing the 'Power' button on the CL control box (labeled "Centaurus") – it will light up green. Now change your signal to **BEIC**. This will bring up the image from the CL detector. You will most likely have to adjust the brightness and contrast on the CL control box to get a good image.

When you are finished with the CL detector, change the signal back to secondary (SEI), and turn off the power on the CL control box. Extract the CL detector arm by turning the large silver knob.

7. Finishing Up

When you are done with your sample, you may remove your sample or switch samples, by selecting **Specimen Exchange** under the Menu button, or you can shut down the microscope by selecting 'Exit Microscope' under the 'Menu' button. This will turn off the high-tension to the filament, and close the software.