

Typical microscopes use light to observe the details of small objects. The resolution limit of light (from diffraction effects) is typically about one half of the wavelength. Since visible light has a wavelength around 5000 \AA , the resolution limit will be around 2500 \AA for an optical microscope. To see smaller features, we need to have something with a smaller wavelength. Continuing along the electromagnetic spectrum to shorter wavelengths we reach x-rays. These would work, but they are difficult to focus down to a small spot size. An alternative, from quantum mechanics, is to use a particle with an energy which corresponds to a small wavelength. Electrons, with energies of $100 - 100,000 \text{ eV}$ have very small wavelengths and can be easily focussed using electric and magnetic fields.

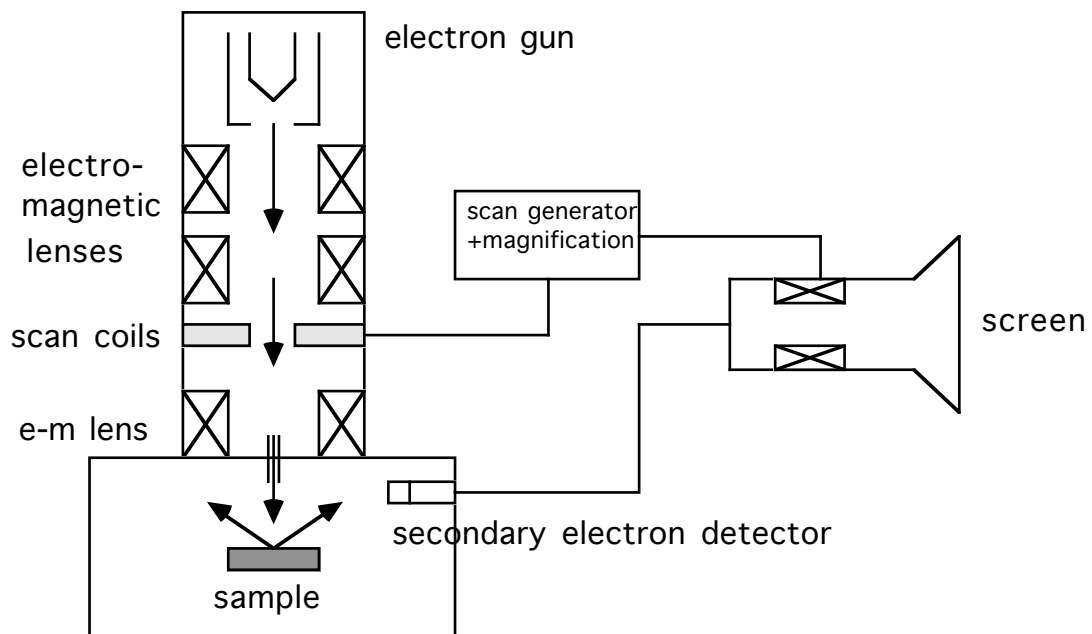


Figure 1. Schematic of typical scanning electron microscope.

A typical scanning electron microscope (SEM) is shown schematically in Figure 1. Electrons are generated from a hot filament by thermionic emission at the top of the column. The electrons are accelerated away from the filament by a high voltage (typically $20-30 \text{ keV}$) into the column. They are focussed by electric and magnetic fields down to a fine spot size at the sample. This focussed beam can be rastered (x and y directions) across the sample by deflection coils in the column. Magnification is accomplished by synchronizing the raster of the beam with the raster of a television screen connected to the detector.

When the beam strikes the sample, some electrons from the incident beam are scattered back off of the sample (backscattered electrons). Other electrons collide with atoms in the material and knock off secondary electrons which escape from the sample. The secondary electrons consist of three types, as shown in Figure 2. Type I electrons are generated directly by the incident beam. Type II electrons are secondary electrons which are generated by backscattered electrons which do not escape from the sample. Type III electrons are generated by stray electrons which strike the

chamber, sample holder, etc. and generate secondary electrons from these other materials. The resolution of the SEM is not limited so much by the incident beam, which can be very finely focussed, but by the larger area in which secondary electrons and backscattered electrons are being generated. Either secondary or backscattered electrons can be detected. The secondary electrons are relatively insensitive to the atomic number of the elements in the sample. The backscattered electrons, on the other hand, are more sensitive to composition and will show a different brightness of image depending on what element is present. Both types of electrons can be detected on the SEM, but we typically use a secondary electron detector to generate an image. Remember that the image is not produced with light. What you see is an indication of the efficiency of certain areas of the sample in producing electrons. Bright regions have lots of electrons coming out. Dim regions have fewer electrons.

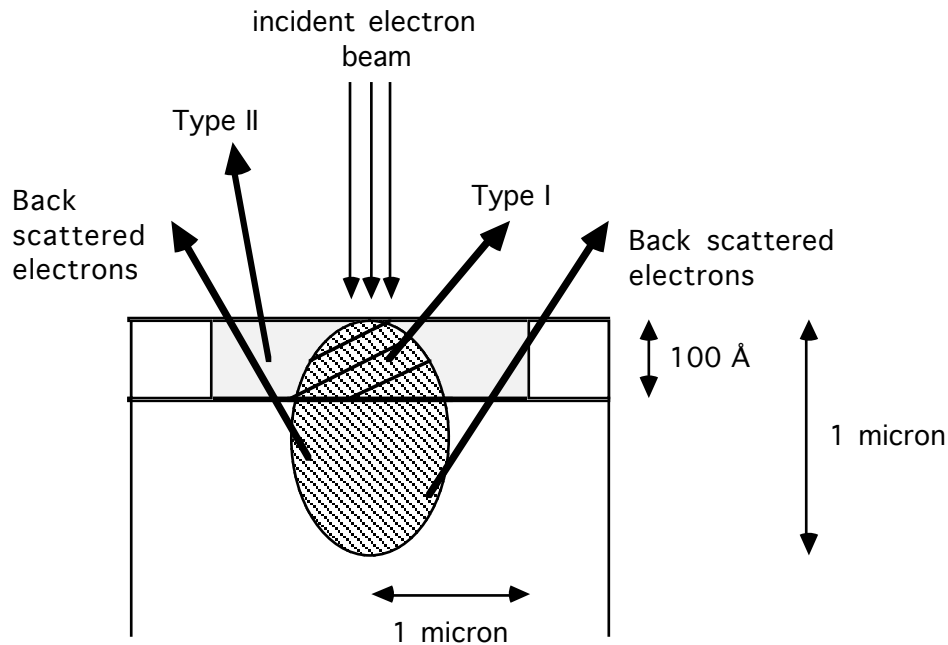


Figure 2. electrons produced in SEM

Scanning electron microscope - ISI Super III-A

Simplified instructions:

1. Check the vacuum in the system of the dial on the right side of the console. It should be all the way to the right in the green part of the scale and the green V.L. light should be on. Turn on the red OPERATION button on the right side of the console. Allow the instrument to warm up for a few minutes.
2. Switch the gauge from VAC to EMIS. Slowly turn up the EMISSION knob. Nothing will happen at first, then the filament will suddenly come up. Continue turning up the knob until the needle on the EMISSION gauge stops increasing. The filament current is now saturated. This usually occurs some place around 2:00 - 4:00 on the EMISSION knob.
3. Set the SCAN MODE to RAPID to view just a small area on the screen. Set the CONTRAST and BRIGHTNESS to about mid-scale. Set the SPOT SIZE to about the 11:00 o'clock position. Select a low MAGNIFICATION. You should now be able to see an image.

4. Adjust the COURSE FOCUS to make the image sharp and clear. If no image appears, try changing the WORKING DISTANCE setting. This acts as the coarsest level of focus. You can now go to a full screen display by changing the SCAN MODE to NORMAL.

5. You can change the MAGNIFICATION to magnify the image. You should adjust the SPOT SIZE too. When the SPOT SIZE is at MAX (fully clockwise) the electron beam spot is at the minimum diameter and the intensity is also minimized. As you adjust the SPOT SIZE you may also need to readjust the BRIGHTNESS and CONTRAST to keep them near mid-scale. As a guideline, the following SPOT SIZES work well for certain magnifications.

<u>Magnification</u>	<u>Spot Size knob position</u>
30 - 5000 X	10 o'clock position
7000 - 20,000 X	12 o'clock position
28,000 - 60,000 X	3 o'clock position

6. A micron marker can be displayed near the bottom of the screen. At low magnifications, the single bar _____ is 50 micrometers long. At higher magnifications the bar splits into two sections _____ The left section is still 50 microns long. The right section is 5 microns long. The gap between the lines is 0.5 microns long. As you increase magnification, the 50 micron section gradually goes off the screen and can not be used at higher magnifications.

7. You can use the IMAGE SHIFT controls to move around in the X and Y directions on the sample or adjust the sample position with the manipulator controls on the door of the chamber.

More complete instructions are available in the instruction manual on (or near) the machine.

SEM experiment

This is primarily an observational experiment designed to give you some familiarity with operating a scanning electron microscope.

Several samples are available. You should look at some or all of them as time permits. Observe them at various magnifications, note the sizes of features you see. Look at the differences between samples or between regions on a sample. Why do you see what you see? Why are some things brighter than others ?

You might try varying the SPOT SIZE to see what effect it has on the image. You will also need to adjust the CONTRAST and BRIGHTNESS to get a good image to observe.

Do not try changing the HIGH VOLTAGE without first turning the EMISSION all the way down. You will find that the filament may need to be realigned after changing the high voltage. Your instructor can assist you with this.

As you write up your lab report, you will need to discuss your observations. What did you see at various magnifications and on various samples. Are there any similarities or differences that you can observe ? Using the scale marker on the screen, you should be able to add some quantitative information about what you saw on each sample. This is a difficult lab to write up because the specific goal is not a well identified measurement. As you work, think about what you can measure and see with the SEM.

ISI Super III-A Scanning Electron Microscope

Supplemental Operating Instructions

Changing Samples:

When you want to change the sample:

1. Turn the EMISSION knob fully counter clockwise to turn off the current to the filament.
2. Turn off the red OPERATION button next to the EMISSION knob.
3. Press the yellow SHUT button next to the SEM column to isolate the pumps from the chamber. Wait about a minute for everything to cool down and be sealed.
4. Press the red AIR button next to the SEM column to let air into the chamber.
5. After about another minute, the air pressure in the chamber will equal the air pressure outside and you will be able to unlatch and open the door that is the front wall of the SEM chamber. (Latch is on the left as you look at it.)
6. The sample is mounted to the chamber by a set screw in the post about 1 cm directly under the sample. Turn the rotation knob on the door until you have turned the sample post to a convenient position to loosen the allen head set screw slightly. With this screw loose, the sample should lift up and can be removed.
7. Place a new sample on the post and tighten the set screw to hold it in place.
8. Close and latch the door.
9. Press the yellow SHUT button next to the SEM column to close the air valve.
10. Press the blue OPER button next to the SEM column to begin the automated pump down process.
11. Once the green VL (vacuum level) light comes on and the vacuum dial is well into the green section, you can return to the operating instructions.

ISI Super III-A Scanning Electron Microscope

Supplemental Operating Instructions

Changing filament assemblies

If the emission dial decreases to near zero and the image disappears, the filament has probably burned out. To change the filament assembly:

1. Turn the EMISSION knob fully counter clockwise to turn off the current to the filament.
2. Turn off the red OPERATION button next to the EMISSION knob.
3. Press the yellow SHUT button next to the SEM column to isolate the pumps from the chamber. Wait about a minute for everything to cool down and be sealed.
4. Press the red AIR button next to the SEM column to let air into the chamber.
5. After about another minute, the air pressure in the chamber will equal the air pressure outside and you will be able to open the top of the column assembly.
6. The filament assembly is housed inside the top of the column. The top is on a hinge which can be opened when not under vacuum. Lift the top part of the column. Notice the little bar that is also on a hinge. Allow it to fall against the filament assembly as you open the top. This makes sure that the assembly is grounded before you touch it !!
7. The filament assembly may be quite **HOT**. Allow it to cool before removing it so that you do not burn yourself. It is best to use plastic gloves or at least a clean paper towel when touching the filament assemblies. Once it is cool enough, pull the old filament assembly straight out (it is only held by pressure fittings). Set the old assembly on the SEM table near the column for cleaning and future re-use.
8. Take a new filament assembly out of the small round glass container on the SEM and push it in where the old one came out. The pins need to be lined up but there is no front and back to it - either orientation should work.
8. Close the top of the SEM column and push down lightly on the top to make sure it is sealed.
9. Press the yellow SHUT button next to the SEM column to close the air valve.
10. Press the blue OPER button next to the SEM column to begin the automated pump down process.
11. Once the green VL (vacuum level) light comes on and the vacuum dial is well into the green section, you can return to the operating instructions.

(NOTE: Once you obtain an image, it may not be of high quality. If not, it is probably because the filament is not directly centered over the column. This can be adjusted using the two large knurled knobs on the SEM column just below the hinged top part. The two knobs extending at 45 degree angles toward the front of the SEM can be used to move the filament assembly over the column. Put the SEM at low magnification and RAPID scan. Use the BRIGHTNESS and CONTRACT knobs to adjust the PHOTOMETER to around mid scale. Turn the two knobs in order to maximize the signal as measured on the PHOTOMETER. If you go off scale, you may need to use the BRIGHTNESS and CONTRAST knobs to come back on scale. When you have maximized the signal, continue operation with an improved image (I hope).

Simplified Instructions for Quantel Imaging System for SEM

The SEM has a computer-based imaging system which can allow you to capture images and store them digitally. It is old and somewhat unreliable, but when it works, it is great.

Get image on SEM

Turn on triple output DC power supply (+/- 12 V) (TP325)

Turn on black and white monitor on top of SEM electronics

Turn on large Quantel Crystal unit (behind computer on far side)
hear clicks
get image on screen
red TEXT light illuminates on the hand unit

Turn on computer
password = Tesla
cancel the Microsoft Network sign in
Start Flyvideo software

Obtain good image on monitor with Quantel Crystal unit
Set scan speed using knob on lower left of Quantel Crystal hand unit

MD1	fastest	poorest quality image
MD2		
MD3		
MD4	slowest	highest quality image

Set integration (if desired) using upper left knob on hand unit to determine number of scans to integrate over - then press INT button on hand unit
(The number of scans to integrate over will depend on how stable the image is and the magnification. At low magnification, I got good results with 32 integrations).

Apply a filter to image (if desired) by choosing a filter using the third knob on the left side then press the CRISP button on the hand unit

Freeze image using FREEZE button on Quantel Crystal hand unit

Adjust image on computer:
Click on << >> button at center to adjust brightness/contrast
Right click on image to get SAVE menu to capture image
usually want Snapshot to File”
saves files as bitmap files in the “flyvideo” folder on the C: drive

When completely done:
Click power button on lower left of computer screen to shut down flyvideo.
Turn off power to: large Quantel Crystal unit, triple output power supply,
B+W monitor, computer

See SEM instructions for SEM shut down procedure