

SEM operation manual

By Jeongwoo Lee

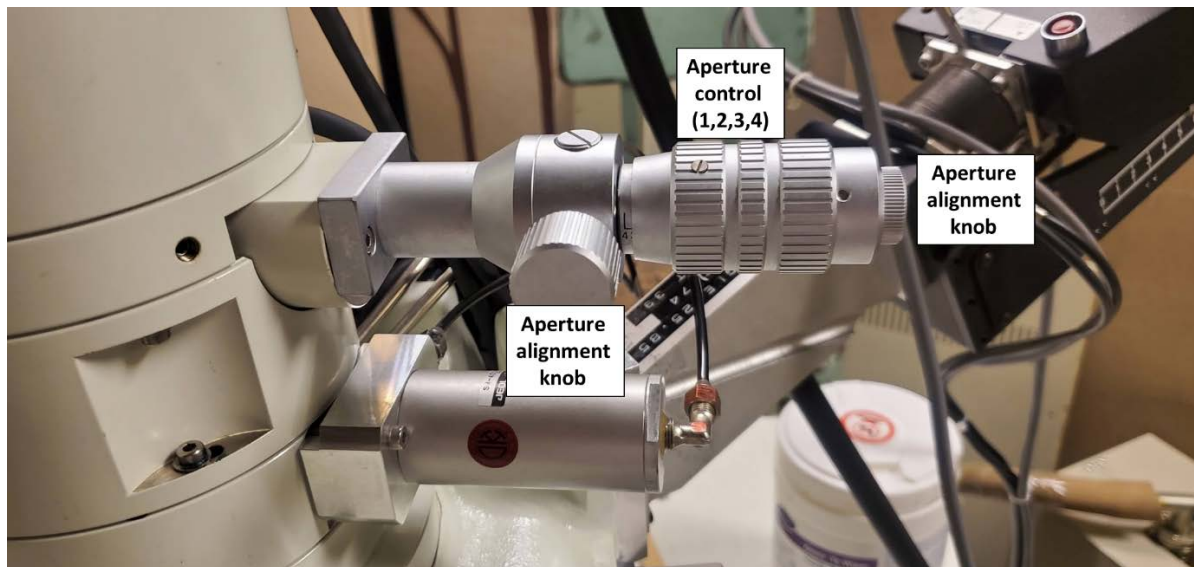
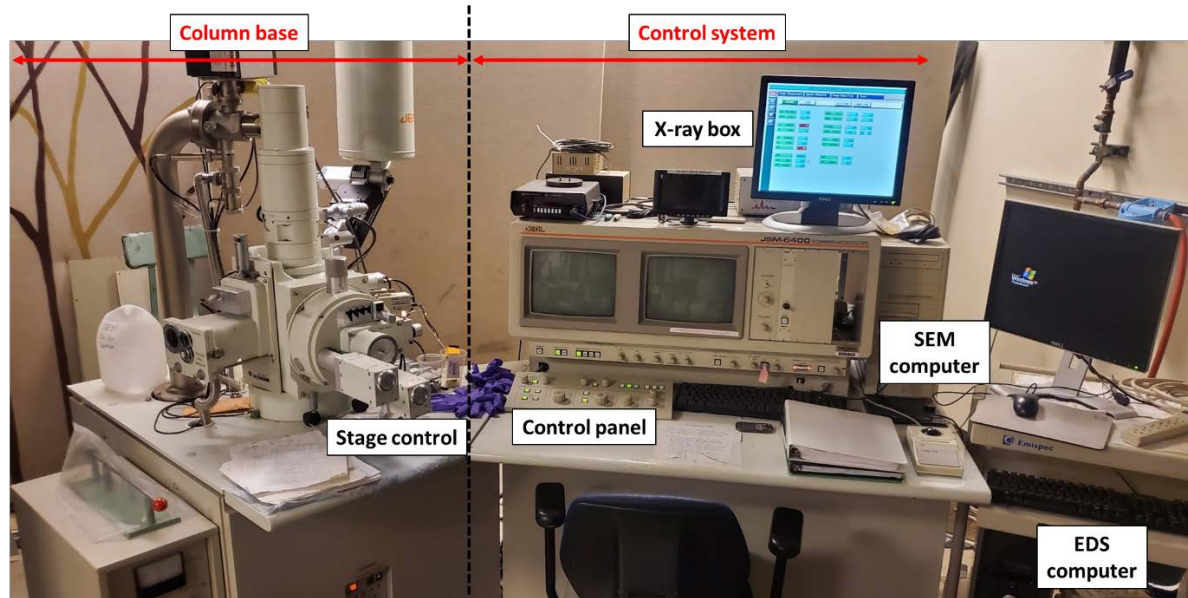
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1 Component names in this manual

Component names used in this manual are shown in Figure 1.



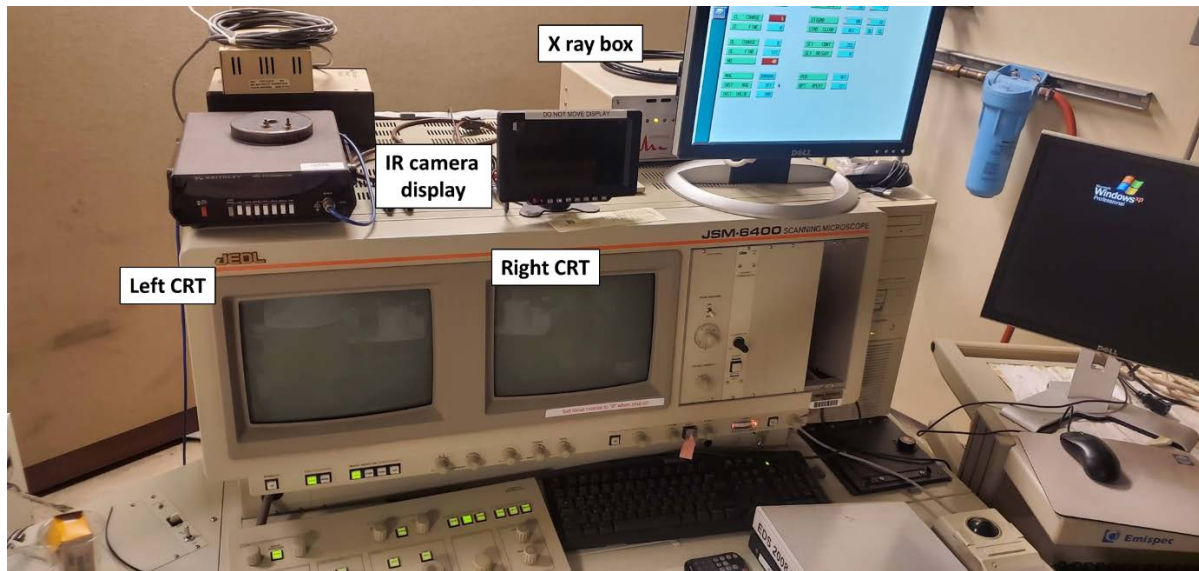


Figure 1 SEM components

2 Computer software

2.1 Vision98 software

Vision98 is installed in the SEM computer

2.1.1 Program manual

The manual is 'Vision98_UserGuide.pdf'

2.1.2 Function1: Monitor control panel status

By clicking four tabs (COLUMN, SCAN, DETECTORS, AND FRAMESTORE), current settings can be monitored. Figure 2 can be used as reference default values in case the system data was reset. Setting values in 'COLUMN' and 'SCAN' tabs will be primarily monitored.

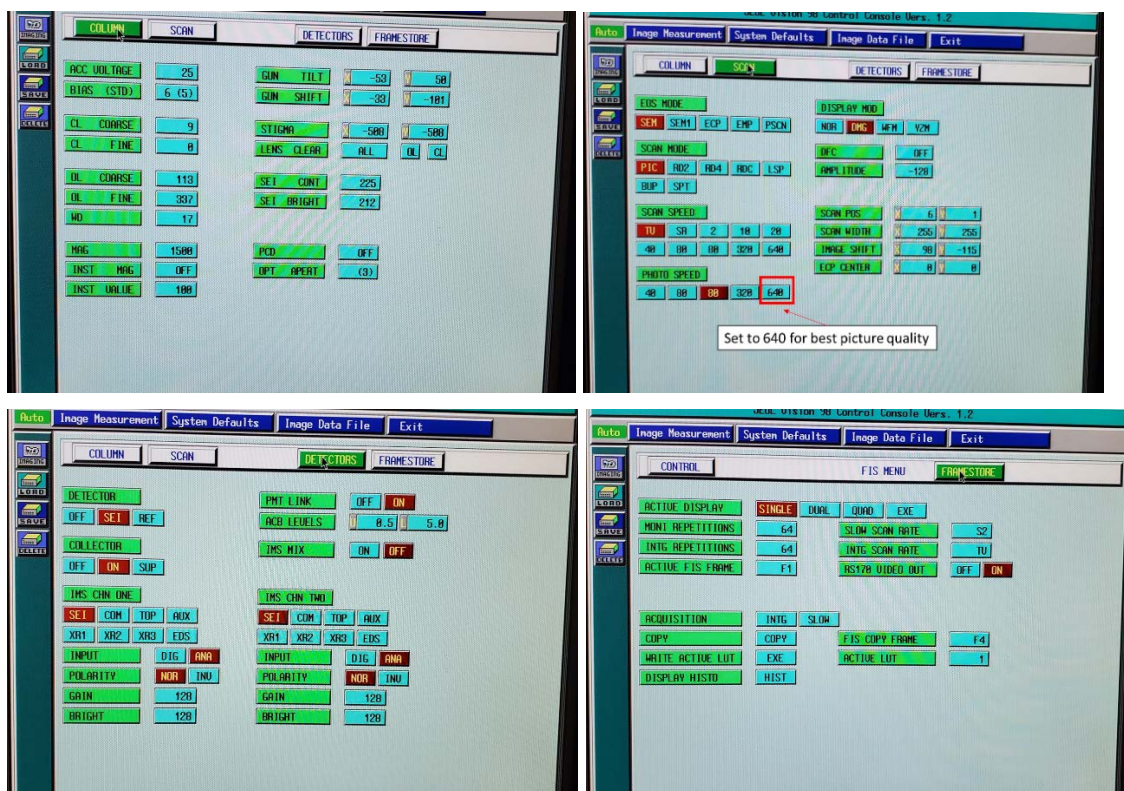


Figure 2 Vision98 monitoring status

2.1.3 Function 2: Controls sample stage motors with mouse (currently not available)

Additional work is required on checking the connection between the SEM computer and two stage motors (X and Y). If the system gets restored, please see the manual for stage motor control.

2.1.4 Function 3: Transfer image between SEM computer and control system (currently not available)

Additional work is to restore connection between the SEM computer and the control system. If the system gets restored, please see the manual for stage motor control.

2.2 EDS 2004 software

EDS 2004 is installed in the EDS computer.

2.2.1 Program manual

The manual is 'EDS2004.pdf'

2.2.2 Function 1: Retrieve image

High quality SEM images can be transferred to the EDS computer from the control system.

2.2.3 Function 2: EDS analysis

EDS analysis can be done on locations (spot, rectangular, or arbitrary) on retrieved images.

3 Operation procedure

3.1 Reserve machine

Users of SEM should reserve the machine in the Dr. Shin's calendar 24 hours in prior to use.

Failures to keeping the procedure can result in complete removal of the machine access.

3.2 Before operation checklist

- Lamps shown in Figure 3 should be checked
- Check the ion pump. Orange power indicator should be on, pressure should be in the range between $3\text{-}5 \times 10^{-5}$ Pa
- Check orange vacuum light should be on



Figure 3 checklist lamps

3.3 Sample preparation

All samples need to be cleaned with acetone first (removes contaminants), followed by methanol (removes leftover acetone). Users should wear plastic gloves to prevent contaminants on hands transferred on samples. Any types of contaminant can be vaporized under high vacuum environment that can contaminate SEM lens.

Attach sample on holder using black carbon tape. Make sure your sample is completely attached to the holder. Attach golden color copper tape to the surface of the 'metal' sample. If sample is non-metal, additional sputter coating is 'necessary'. SEM cannot visualize non-metal surface without surface sputter coating. How to setup tapes is shown in Figure 4.

Sample holders should be used to mount bakelite or samples. Use extreme caution when mounting samples on sample holders. If top surface of the sample is above the sample holder top surface, sample can crash into SEM lens, as shown in Figure 5.

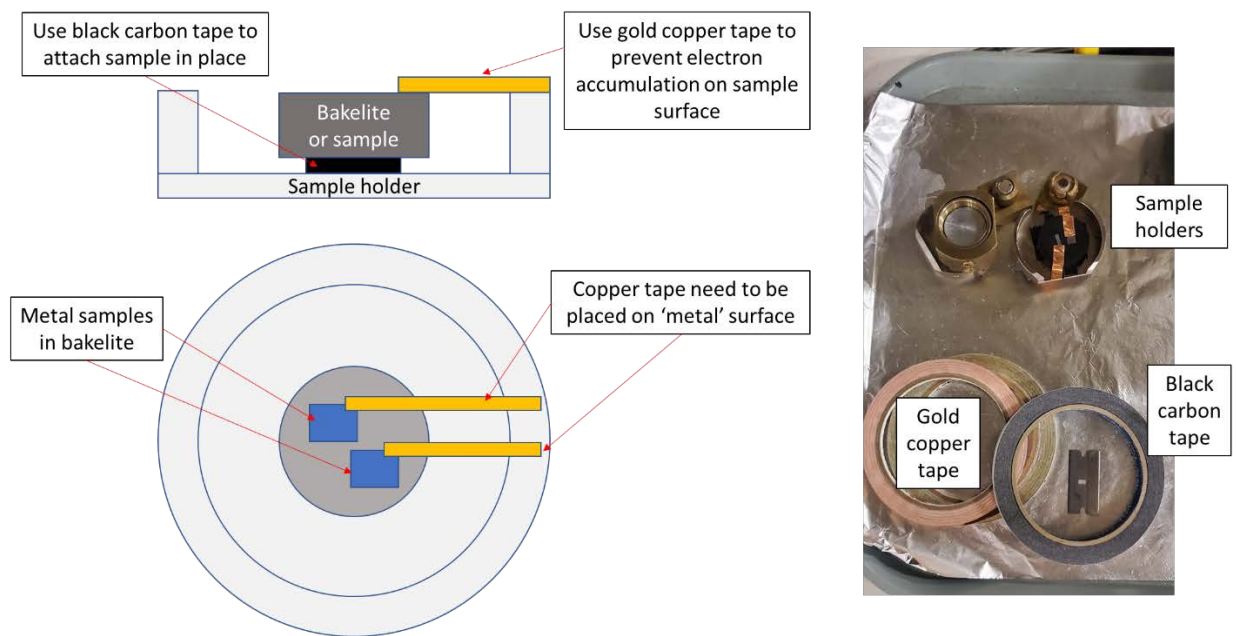


Figure 4 Using carbon and copper tapes to prepare samples on sample holder

EXTREME CAUTION!

Do not setup sample above the sample holder surface plane

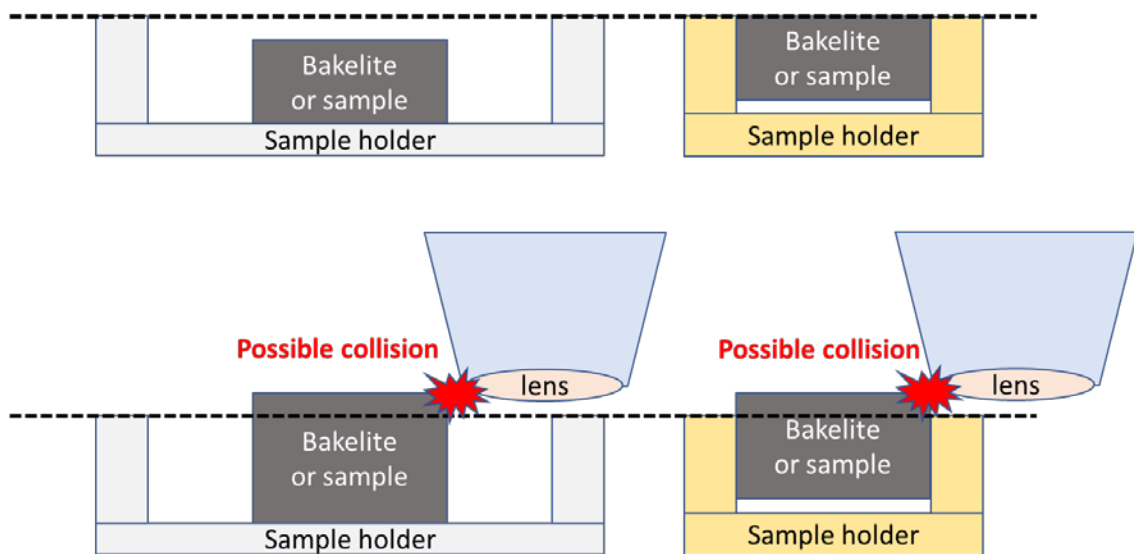


Figure 5 potential collision with SEM lens

3.4 Sample loading

- Make sure the offset knob is set to 39.
- Set stage location at home (X: 25 mm, Y: 35 mm)
- Rotation and tilt are set to '0.0'
- Connect the metal rod to hole (B) of the holder.
- Don't touch the metal rod and slowly insert into the machine.
- Make sure the disk attached to the rod is completely closing the chamber entrance.
- Push vacuum button. Wait.
- Turn on the IR camera and hit "illumination" to see as the sample goes inside the chamber before inserting.
- When the light turns off, open and lock the chamber door. Make sure top of the holder faces upward while inserting. The camera should only be turned on when detector and beam are not aligned (SEM image is highly affected when the camera is on)
- When the sample is locked on the rail, unscrew the rod from holder.
- Change working distance when camera is on. Default is 39. Make sure either top of sample holder or samples doesn't touch anything inside the chamber.
- Pull back the rod
- Close the chamber door and lock
- Hit vacuum button
- Take the rod out and close chamber entrance with aluminum disk cover.

3.5 Filament saturation

- Change mode to LSP (Vision98→tab 'SCAN'→SCAN MODE→LSP)
- Turn up brightness of monitor (known under CRT monitors).
- Locate line on the center of monitor by adjusting brightness and contrast knobs. You will see the line on the left monitor by controlling contrast and brightness, as shown in Figure 6. It is necessary to turn off room light completely.
- Set Acc voltage 20-25 kV (metal) or 10-15 kV (non-metal).
- Turn on Acc voltage
- Check if current is on. Push 'Fila' button. Check it moves from 0 to 100 (indicates filament is still usable) and turn off 'Fila'.
- Lower magnification to 10~11.

- Increase the filament current. Turn 'filament' knob SLOWLY (half a peak mark every 5 sec).
Caution: Faster saturation can burn up filament.
- Height and intensity of filament current signal indicates beam current, as shown in Figure 7.
- Filament current should be carefully increased when it passes red arrow.
- Turn on knob until signal gets stabilized (1 false peak should be passed), as shown in Figure 7
- Adjust gun alignment. Control x and y one at a time. Find max signal intensity and height.
- Push "pic" to get picture. Start from low magnification

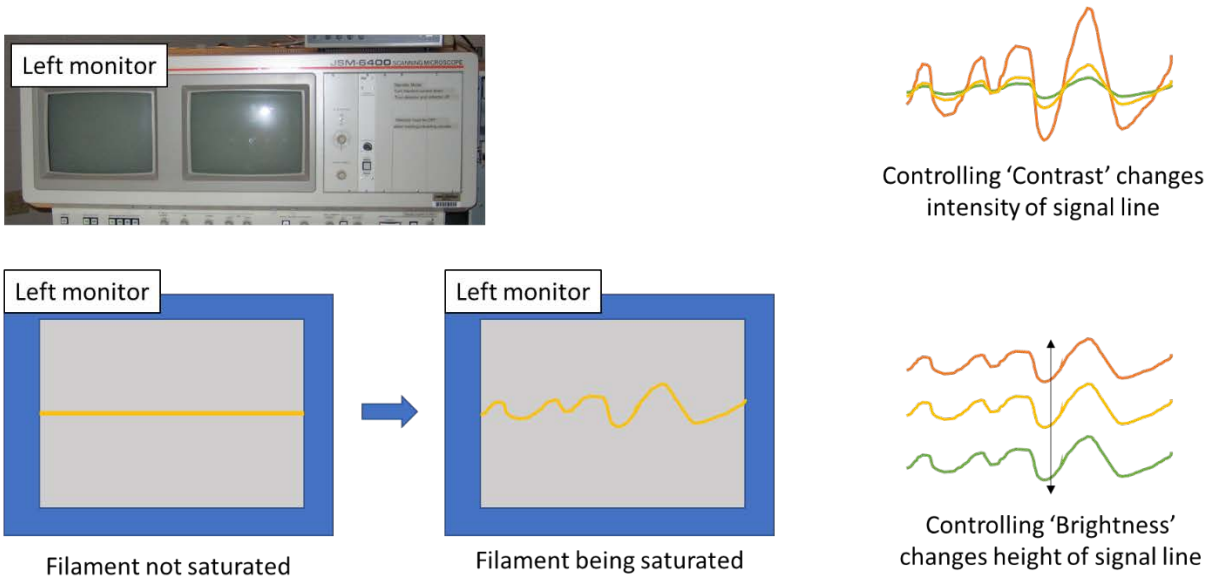


Figure 6 Monitoring filament saturation

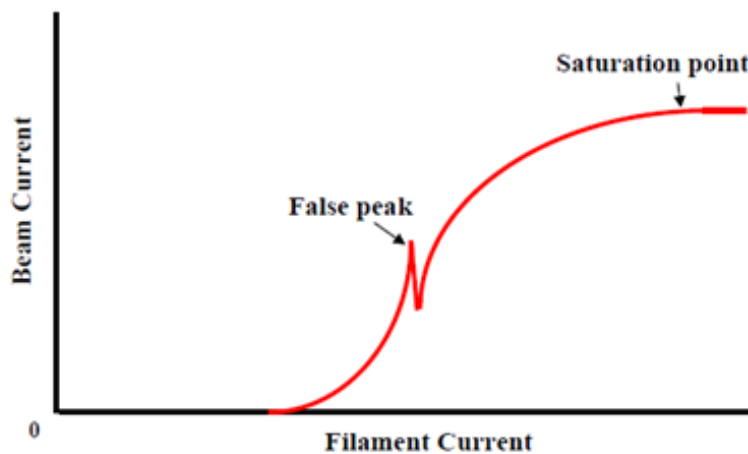


Figure 7 Filament saturation procedure


3.6 Focus and astigmatism correction

- Use 'live action' to find desirable spot to be analyzed. To quit, click right button. (Function not available now)
- Manually move stage position using X and Y knobs.
- Starting from a low magnification, focus (major/minor) on target surface spot to get the best image on CRT screen. Move to higher magnification
- Stigmatism is present when image has blurry stretch when you adjust focus knob. You should control stigmatism knobs at a point where image is between over blurry and very focused to get rid of stigmatism. Remove stigmatism one direction at a time (X and Y)


3.6.1 Higher magnification (over X10,000)

- Push 'wobbler' to start aperture centering. The screen image gets focused and de-focused continuously.
- Control x and y knob on column. The goal is to prevent the image gets deformed in one or both directions (x and y). In an ideal case, the image will be observed as blinking without deformation in specific directions.

3.7 Obtain SEM image


- SEM image can be acquired by the EDS computer, EDS2004 software.
- Set photo speed to maximum (640) in Vision98 software under 'SCAN' tab to get the best quality image
- Click the camera icon () to grab images. It is recommended to use the resolution of 1024 or higher to for publication
- Images can be saved individually (File → Save as)


3.8 EDS analysis

- Spot the position on the specimen to analyze
- After acquisition of a good image in SEM monitor, set magnification on program as same as that of SEM machine
- Click the camera icon () on EDS program

- Adjust settings of time constant and condenser lens
Preferred : 2000~4000 counts per second in EDS collected electrons,
MAKE GREEN COLOR
5~20% dead time (10% recommended)
- Low TC will reduce dead time
- Set live time to 50s (default). Increase if needed.
- When program is not responding, restart the program
- Use options to pick region to analyze
“+”: putting a beam only at a spot
“rectangular” : rectangular area of one’s choice
“freehand” : make shape of one’s own
- “table” shape icon will bring periodic table
- Select elements in the table
- Using right mouse click, hit analyze→save→export
- Select properties, quantitation tab.
- Select remove sum peaks and silicon escape peaks (Number of smooths=2)
- When making a standard file, use mouse right click, Utility→Create standard→Save→Ok. Save as “name.std”
- When making a ZAF calibration file, use mouse right click, Utility→Quantification Calibration→Browse→Select one of the standard file→Calibrate→Close. Then save as “name.fpc”
- When performing a quantitative analysis by applying a ZAF correction, use mouse right click, Utility→Quantification→Advanced→Calibration→load one of .fpc file

3.9 X-ray analysis

- Perform a quantitative analysis on a wide area of a sample using EDS analysis stated above.
- Select “work with X-ray maps”
- Go to the “Elements selection” tab and select elements of interested
- Acquire an x-ray map by clicking on the  button
- Select “work with X-ray linescans”
- Go to the “properties”

- Go to the “Elements selection” tab and select elements of interested
- Acquire an x-ray map by clicking on the  button

3.10 Sample unloading & Shut down procedure

- Turn off fila knob. Does not need to be as slow as saturation procedure.
- Turn off “ACC”
- Set brightness to 0, contrast to 255, magnification to 300kX and focus to 0.
- If you changed the position of sample by using trackball, push “drive to home” (function not available)
- Set working distance back to 39mm before unloading specimen from chamber.
- Turn on camera
- Insert metal rod into entrance of vacuum chamber and hit “vacuum” button
- When light goes out, open chamber door and insert rod into sample holder.
- Screw rod into sample holder and take holder back by pulling the rod until it reaches end
- Close chamber door and hit “vacuum” button
- Turn off camera
- Put sample holder back to its original place.

4 Background information

4.1 Tungsten filament

A voltage is applied to the tungsten filament (which acts like a cathode), causing it to heat up, as shown in Figure 8. When the filament gets hot enough, electrons are emitted thermionically. A strong electric force is present between the electrons emanating from the filament and the anode plate. This force causes the electrons to be accelerated towards the anode plate. In fact, some of the thermionic electrons are accelerated in such a manner as to stream right by the anode plate and down the column of the microscope to the sample. This electron beam is scanned back and forth over the surface of the specimen using electromagnetic lenses and the electrons that are reflected/emitted from the specimen are used to resolve an image on a cathode ray tube(CRT) or computer monitor.

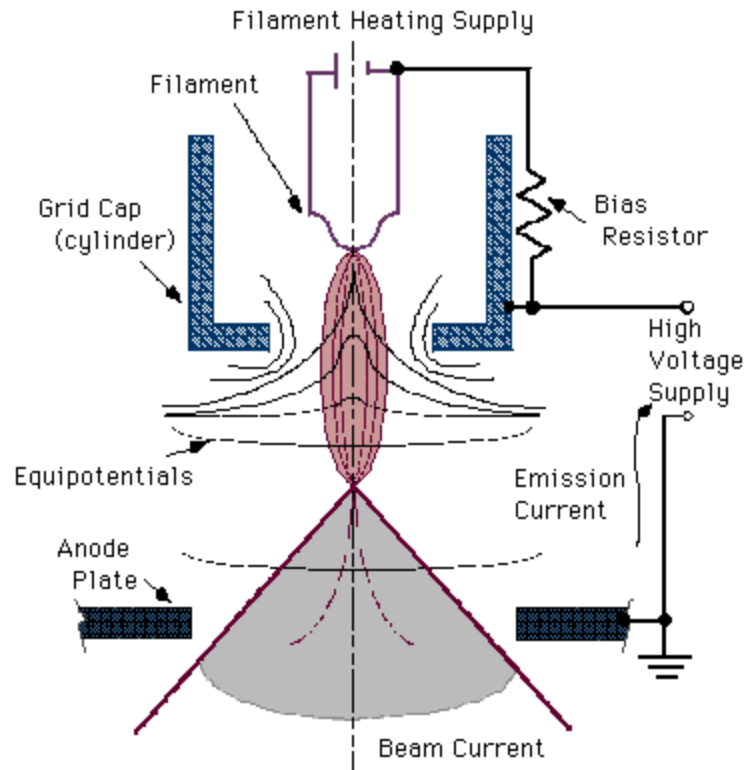


Figure 8 SEM filament basics

4.2 Basic SEM Construction

Figure 9 shows the SEM column including the gun, spray aperture, objective aperture and all electromagnetic lenses. Figure 9 shows the path of the beam, showing how the beam is de-magnified and focused as it travels down the column. The three crossovers should be easily identifiable with the forth being sample surface dependent

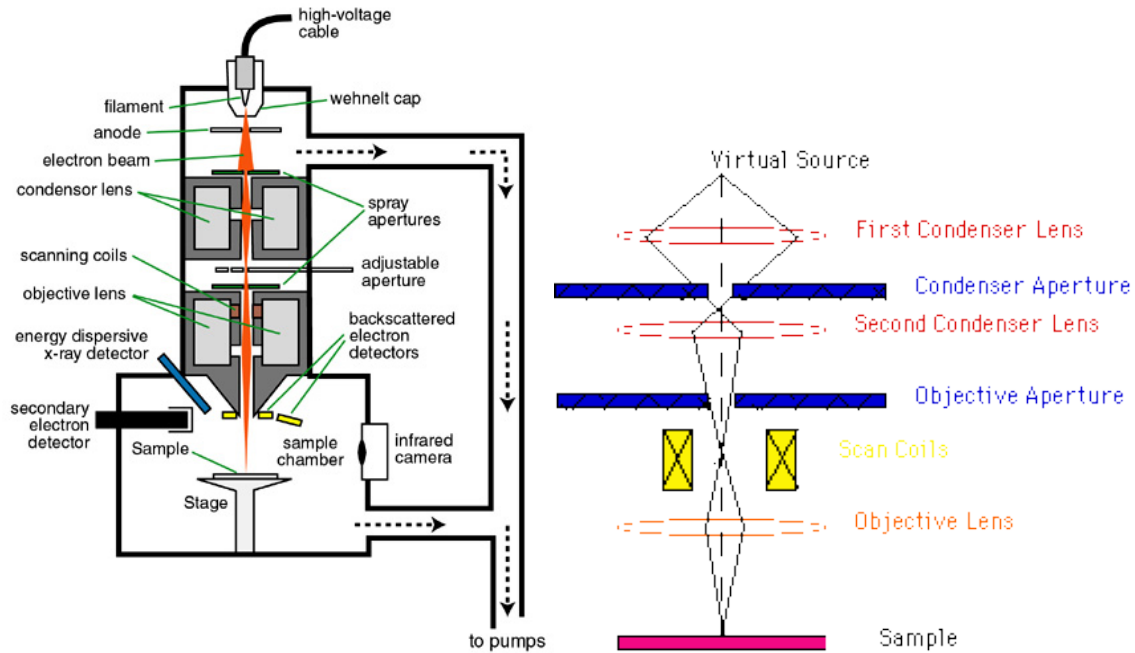
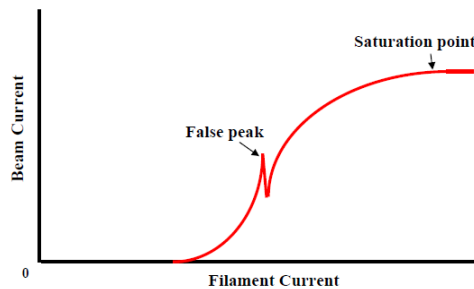


Figure 9 SEM components

5 Frequently asked questions

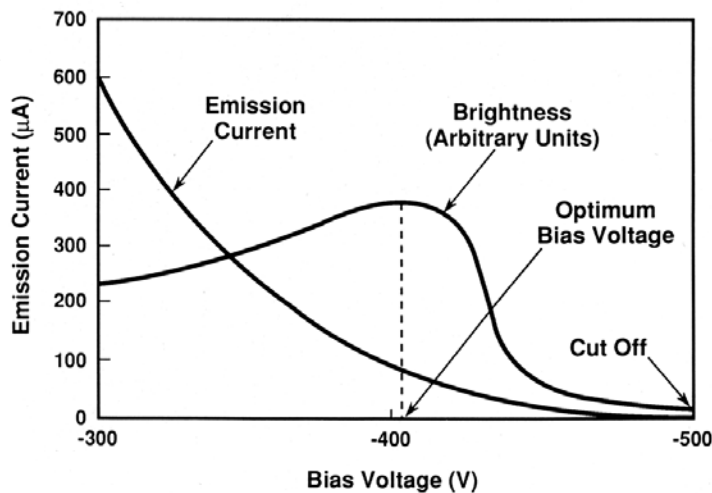
1. Why must you wear gloves when handling vacuum equipment?
 ➔ Not to contaminate the sample.
2. What conditions must be met before it is ok to press the vacuum button for the sample exchange chamber.
 ➔ No leak in sample holder metal bar
3. How fast should the filament knob be turned (approximately) and why does the filament need to be heated slowly?
 ➔ Approximately 1 min. If it is heated up faster, filament will burn out
4. Give two reasons for not under-saturating the filament.
 ➔ Resolution and contrast cannot be controlled to view the image
5. Give a reason for not over-saturating the filament.
 ➔ filament will burn out more easily

6. How does a false peak differ from gun misalignment?
 ➔ false peak is usually at about 3.4 volts caused by region of filament
7. How can you tell that the objective aperture is out of alignment?
 ➔ Image is not in same place but keep moving
8. How can you tell if the stigmation is off?
 ➔ Image stretches
9. Provide a rough sketch of the relationship between filament current and beam current.



➔ As the filament heating current is increased, so is the beam current to a point called saturation where any further increase in filament current will not increase the beam current.

10. What is a false peak? What causes it?
 ➔ This is a result of some part of the filament surface reaching emission temperature before the tip. As the filament current is increased the false peak collapses and a small, tight and more stable beam is ultimately achieved. To the user this false peak can be observed as an increase in the probe current (brightness) followed by a drop in emission and a further increase in probe current as the filament current is increased up to the saturation point.
11. How does changing the Accelerating Voltage affect your GUN BIAS? Why?
 ➔ Gun bias is to be set at proper value to get good current and also, good focusing. Too small bias can cause electrons not accelerated past anode well, result in poor focusing. On the other hand, too large bias moves the electron field moved too far away from the tip and electron emission would be lost.



12. Why is it important to fully saturate the filament?
 - ➔ The more current that is put through the filament, the greater the emission of electrons from the tip region. However, a point is reached where the emission is at maximum. This is called saturation. At saturation point, brightness is the highest.
13. What generally happens when you turn either the x or the y aperture control strongly one way and then the opposite way? (Note assumptions)
 - ➔ Assuming turning x aperture control hardly one way, image will be shown enlarged in x direction. That is, distortion in the image.
14. Ultimately what is the recommended magnification for the last stage of aperture alignment if imaging at 60,000X is desired?
 - ➔ Alignment at the magnification higher than 60,000X is required
15. What level of magnification must one reach before it is likely one will be able to observe and correct beam astigmatism?
 - ➔ at least 24,000X
16. How do you check for astigmatism and what will you see if astigmatism is present?
 - ➔ By going through focus, both over-focus and under-focus, the image will elongate in one or two perpendicular directions if astigmatism exists
17. What happens when you turn the x or the y stigmator control strongly one way and then the opposite way when in focus vs. out of focus?
 - ➔ If the image is in focus, adjusting stigmator control will ruin the quality of the image. The

image will be elongated in either x or y direction. When the image is out of the focus, turning the stigmator control strongly in one way or the other will sharpen the image at right control position, but start to enlarge as the controls are kept turning.

18. What causes astigmatism in SEM images?

→ Non circular electron beam from gun causes astigmatism

19. What happens to the image if the brightness and contrast are out of range?

→ Lines and figures in the image will be hard to distinguish

20. Compare the influence of the condenser and objective lens currents on the signal.

→ The condenser lens controls the amount of current that passes down the rest of the column.

This is accomplished by focusing the electron beam to variable degrees onto a lower aperture.

The sharper the focus, the less of the beam intercepted by the aperture and the higher the current.

The objective, or "probe-forming", lens is located at the base of electron column just above sample. The beam is again divergent after passing through the apertures below the condenser lens and must be refocused. The objective lens focuses the electron beam onto the sample and controls final size and position.

21. What is the effect of Accelerating Voltage

→ Case 1.

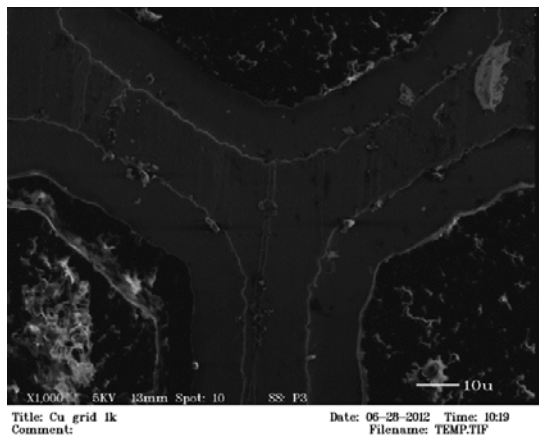
Accelerating Voltage: 5kV

Objective Aperture: #3

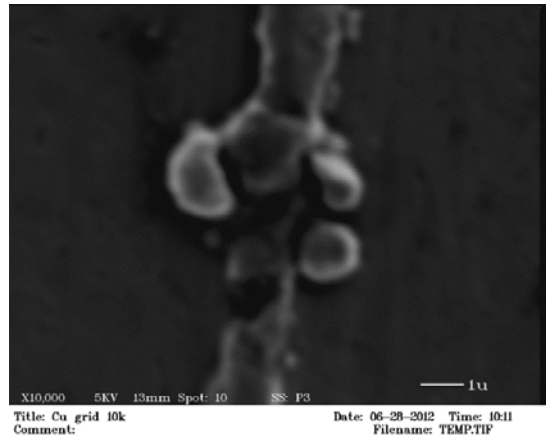
Working Distance: 15mm

Condenser Lens Number: (10)

Gun Bias: 2(2)



a) X1,000



b) X10,000

➔ Case 2.

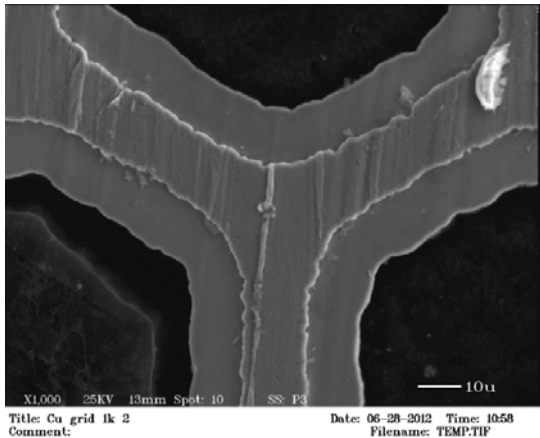
Accelerating Voltage: 25kV

Objective Aperture: #3

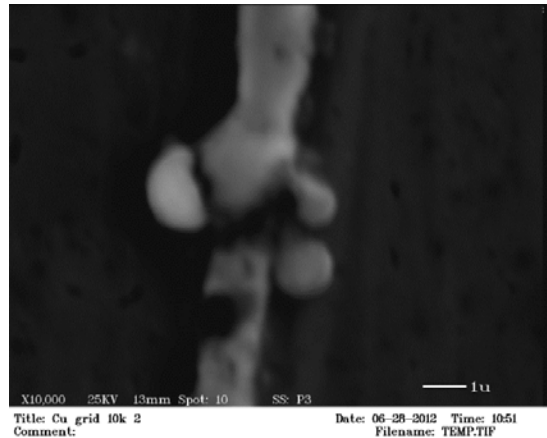
Working Distance: 15mm

Condenser Lens Number: (10)

Gun Bias: 5(5)



a) X1,000



b) X10,000

22. How does the relative sample current differ between 5 kV and 25 kV (note the gun bias). What effect should this have on the images captured?

➔ Values of relative current were not recorded. With increment of accelerating voltage, from 5 to 25kV, gun bias increased, that is, sample current must also have been increased. With high sample current, more precise shape of image can be acquired, however, more noise will be captured in the image.

23. How do the images at 5kV and 25kV compare? Describe the visible differences.

➔ At X1,000 magnification, higher voltage makes image clearer and brighter, that is, boundaries of shape are easier to be distinguished. At X10,000 magnification, voltage only does not make noticeable difference in visible images (other control factors need to be adjusted together to get better images)

24. What is the effect of Condenser Lens Current

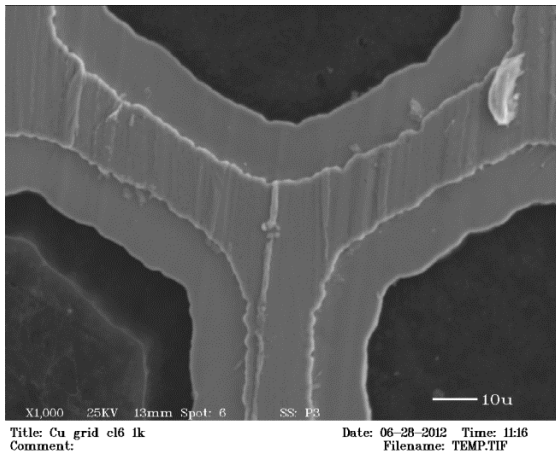
➔ Case 1

Condenser Lens Number: (6) **larger beam**

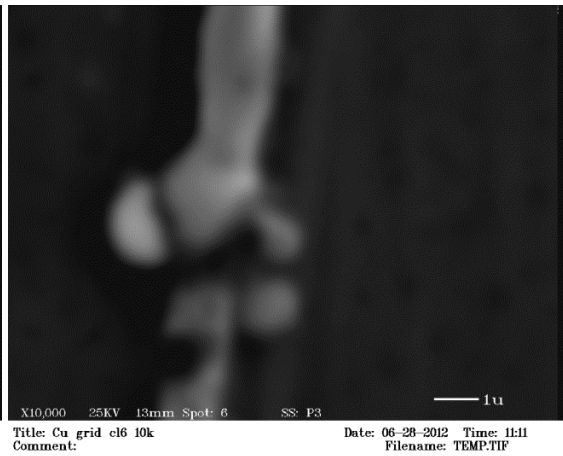
Objective Aperture: #3

Working Distance: 15mm

Accelerating Voltage: 25kV



a) X1,000



b) X10,000

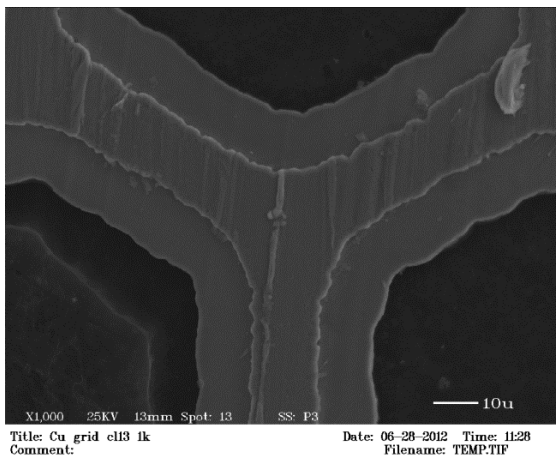
→ Case 2.

Condenser Lens Number: (13) **smaller beam**

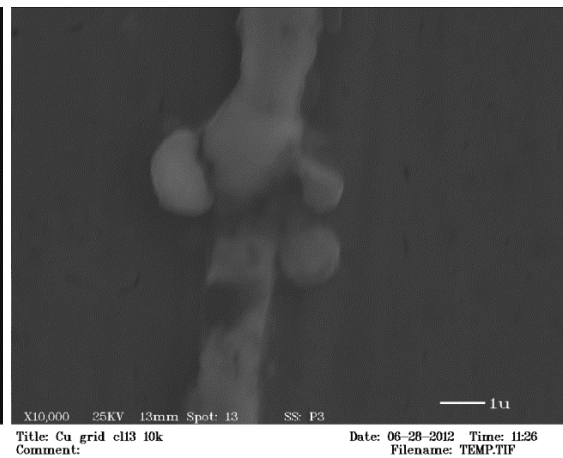
Objective Aperture: #3

Working Distance: 15mm

Accelerating Voltage: 25kV



a) X1,000



b) X10,000

25. Define resolution

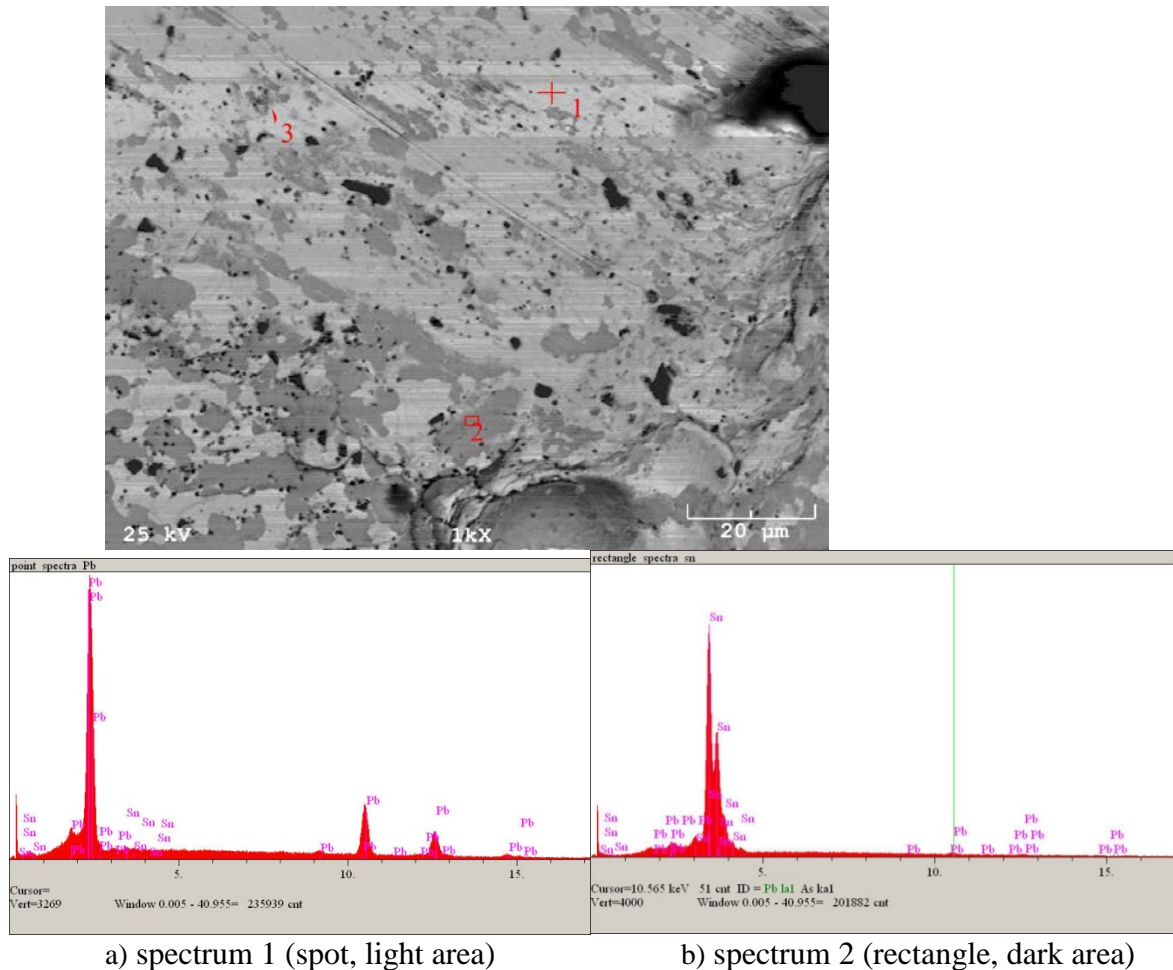
→ Resolution is the smallest length of distance between points that you can distinguish one with another. Therefore, a smaller number of resolution value of microscope means that through that microscope, points with closer distance can be resolved. The resolution of a SEM is about 10 nm.

26. How do the resolution and image quality differ between the images?

➔ As Cl number increases, beam diameter reduces. With a smaller electron beam, the numbers of electrons reflected and emitted from surface of specimen increase and more information on the surface can be acquired. Therefore, clearer image is obtained with high Cl number.

27. How consistent is the measurement of composition from similar areas of the sample?

➔ Regarding the major peak locations, spectrum 1 and spectrum 3 show similar results in analyzed composition. Minor shift of peaks might be due to difference in ways of analyzing between two methods, spot and rectangular. According to EDS spectrums, light area is consisted of Pb while black area is filled with Sn



is not flat, bumps on the surface would hinder photons moving toward the detector. In this way, number of photons detected by detector would be less than original value.

32. What does a ZAF correction do? Define Z, A, and F for this abbreviation.

➔ ZAF correction means atomic number effects(Z), absorption(A), and fluorescence(F) are considered and calculated separately from reliable models to increase accuracy.

33. Describe what you see in the Xray maps

➔ By selecting elements of one's interest, distribution and intensity of each element in selected region can be shown in the screen

34. Define another use for Xray maps (Not Phase contrast)

➔ Not only distribution of each element is acquired but weight percentage ratio is also analyzed.