**Lab 3: Scanning Electron Microscopy (SEM)** Kelsey Lawson, 41602126

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*1) What are the 5 uses for Scanning Electron Microscopy (SEM)?*

SEM can be used to image morphology of samples, image composition as well as bonding differences using contrast and backscattered electrons, view and map the grain and crystallographic orientation, undertake micro and nano-lithography and view frozen material using cryostage. These possible techniques are also used in a variety of ways:

1) In materials science for "basic research, quality control, and failure analysis" that allows metals, alloys, ceramics, polymers, and biological materials to be examined.
2) In biological science for all sizes ranging from insects to animal tissues or even bacteria.
3) In geology soils and geological samples are analyzed based on the morphology that occurred due to weather. Also compositional details are provided.
4) In medical science, blood cells and tissue samples are compared by researchers to examine complications caused by an illness.
5) In forensic science, laboratories analyze evidence to finalize if the provided samples match the crime scene.

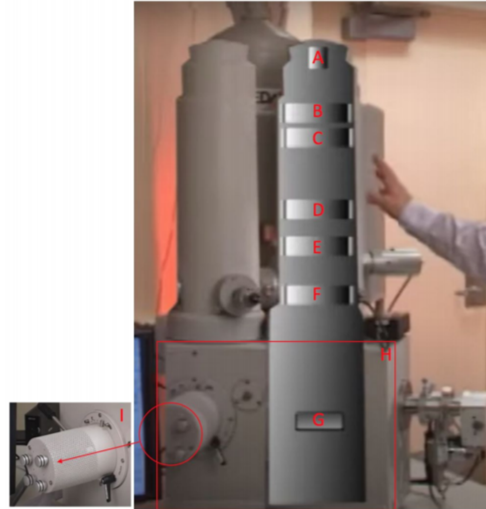
*2) What are the 3 advantages of SEM over standard light microscopy?*

3 advantages that SEM has over standard light microscopy are the resolution at high-magnification, depth of field, and microanalysis. The resolution of SEM can examine samples at a much closer distance ranging around 10nm whereas LM is only 200nm. The depth of field for SEM is 300 times that of an LM which allows an image to define the topography of a sample at higher details. Finally, SEM microanalysis includes a more detailed image that holds information regarding chemical composition, crystallographic, magnetic, and electrical characteristics whereas an LM limits the amount of information due to the prior areas.

*3) What are the 5 limitations of SEM?*

5 limitations of SEM are that it can't image wet samples, produce color images, image non-conductive samples, imaging through fluid, and have accurate height measurement. Wet samples could be damaged when placed in a vacuum when using SEM and would have to be dried prior. The images produced when using SEM are in grayscale because of the electron wavelengths being smaller than those of visible light. Non-conductive samples won't produce images because of the interaction between the negatively charged electron beam and sample hence a coating of metal or carbon to induce conductivity should be added. SEMs are not able to produce images through water or any other fluids but other special machines can. SEM measurements in the z-axis cannot be done as well as quantifying surface roughness at a small scale.

*4) a) What are the main components of an SEM as pointed out in this video, and what is the purpose of components A-E.*




The main parts of the microscope are the column, chamber, a surface that holds the microscope, and then the user console. Within the column are the following:

A: **Electron Gun** - electronic field is used to draw current off a filament to create the incident beam to impinge the sample

B-C: **Condenser Lenses** - controls the size of the beam and can determine the number of electrons in the beam that makes contact with the sample

D: **Scanning Coils** - used to "raster" the electron beam over a rectangular area on the sample surface

E: **Stigmators** - adjusts the roundness of the beam

F: **Probe Lens** - adjusts the position of the electrons onto the sample

G: **Sample** - a material of choice that is undergoing the SEM imaging

H: **Chamber** - impacts the sample where the sample absorbs the energy of the electrons and then reemits them into new signals for imaging

I: **Manual Stage Adjustments Knobs** - allow for control over stage position of The sample in the x, y, and z-axis as well as rotation

*b) Why do you think the chamber pressure must reach ~$1x10^{-5}$ mbar before you can open the column valves (also called turning the beam on)? Your answer should address the safety of the equipment.*

Allowing the chamber to reach ~$1x10^{-5}$ mbar before you can open the column valves allows uniform pressure for the sample to endure the electron beam. Without a high-vacuum environment for the beam to reach the sample, the beam itself is affected by losing its condensed state and would instead spread the electrons elsewhere instead of a specific point on the sample.

*c) Why do we need special equipment to dampen acoustic vibrations?*

Special equipment is needed to dampen acoustic vibrations in order to prevent any small vibration from showing up when viewing the sample.

*d) What happens when you put samples containing liquid in a vacuum environment such as the SEM chamber?*

The environment inside the microscope is at a high vacuum hence if any liquids are contained in or on the sample, vaporization of said liquid occurs and ruins both the sample and image.

*e) How can we store and handle samples in a way that keeps them (i) contamination-free, (ii) condensation-free, and (iii) safe from handling-induced damage?*

Storing the samples in a desiccator box keeps the samples contaminated-free especially from any collection of water. Using vinyl gloves also prevents any possible deposit of unwanted material onto the sample. Finally blowing the samples off with a stream of nitrogen gas ensures that any possible dust on the surface is removed before imaging.

*5) According to the chart, what is the sample preparation process required for imaging non-conductive powders using conventional SEM?*

When preparing a non-conductive powder sample for conventional SEM imaging, the powder will need to be sprinkled onto conductive tape or glued to a support system. During this preparation make sure there is a conductive pathway from the particles to the stub to avoid charging during imaging. Then mount the sample in resin so the internal features can be studied. After mounting the sample polish the surface so that it's smooth. Finally, a metal coating should be applied to improve the conductivity of the sample, usually a sputter coat of gold or platinum.

*6) a) What are the two main types of aberrations in an SEM and how do they affect your probe size, and therefore your image resolution (smaller probe = higher resolution)? Assume pixel size is small enough so that it does not limit resolution.*

The two main types of aberrations in an SEM are chromatic (refers to the energy spread of electrons coming from the source) and spherical aberrations (refers to the imperfections in the lenses themselves). When chromatic aberrations have low energy there is larger energy spread with many different wavelengths of electrons passing through the lenses at the same time making it harder for the lenses to focus them down into a small probe. When spherical aberrations are at high energy the electrons are traveling at faster rates spending less time in the lenses. This means any imperfections in the lenses will be imparted less on those electrons at high energy and will be able to focus them better into a small probe.

*b) What is the sample preparation method that can be used to reduce charging artifacts when imaging non-conductive specimens?*

In order to reduce charging artifacts when imaging non-conductive samples, going to lower voltages affects the charge equilibrium on the sample, therefore, eliminating the charging artifacts to improve the imaging quality. Also, coating the specimen in a conductive material will improve the imaging and allow operation at higher voltages.

*c) What are the 3 types of electron interactions that generate the signals we commonly use in the SEM? Describe how each of these interactions occurs.*

The three types of electron interactions that generate common signals in SEM are secondary electrons, backscattered electrons, and characteristic x-rays. Secondary electrons are created when a primary electron from the beam comes down and has an inelastic interaction with the electrons of the sample. The electron that is kicked out from this interaction is defined as the secondary electron. Backscattered electrons are created when a primary electron from the beam is elastically scattered by the atoms in the sample. Finally, characteristic x-rays occur when the primary electron knocks out an inner shell electron that becomes a secondary electron. The vacancy of the inner electron makes an outer shell electron move from high to low energy and from this movement a characteristic x-ray is produced.

*d) What information does each of these 3 electron interactions provide about your sample (topography vs. composition), explain why this is the case in terms of the escape depth of the signal, and the dependence of the interaction probability on atomic number.*

Secondary electrons provide topographical information about the sample because on the electrons from the top 5 nm of the sample are able to leave the surface of the specimen and are then attracted to the detectors with a positive bias. The atomic number also affects how many secondary electrons are being produced based on the orbitals which in turn creates a brighter spot in the image with a higher Z. Backscattered electrons are deeper in the specimen and therefore provide compositional information about the sample. Similarly, the higher the atomic number the more backscattered electrons are produced. Characteristic x-rays also provide compositional information but occur at a deeper interaction volume than backscattered electrons. Because of the energy balance that takes place when an outer electron moves to the vacant inner electron spot, a photon is emitted to an equivalent energy. These characteristic x-ray amounts are unique to every element and when detected and measured, the elements in the specimen can be found.

*e) What types of detectors are used to detect each of these 3 types of signals?*

For detecting secondary electron signals, an Everhart-Thornley Detector (ETD) and Through-the-Lens Detector (TLD) can be used. For detecting backscattered electron signals a Backscattered Electron Detector (BSED) is used. Finally for characteristic x-rays, an Energy Dispersive Spectroscopy (EDS) and Wavelength Dispersive Spectroscopy (WDS) are used.

*f) Why might you want to perform a Monte-Carlo simulation to help you understand your SEM data (SE & BSE images, x-ray signal)?*

Performing a Monte-Carlo simulation helps understand what volumetric shape and energy the electrons within the sample produce from SEM. Shown in the video, the blue paths are reabsorbed electrons from the beam into the material and the red paths are elastically scattered electrons that emerge out of the specimen to be detected.

*g) Based on what you have learned from this video, fill in the following table. The leftmost column contains 3 operational parameters you have control over when you operate the SEM. Indicate whether the relationship between the operational parameters and the highlighted elements is proportional (P) or inversely proportional (I).*

| **Operational Parameters** | **Signal Surface Sensitivity** | **Resolution** | **Signal:Noise Ratio** | **Depth of Field** |
|---|---|---|---|---|
| **Accelerating Voltage** | P | P | I | N/A |
| **Spot Size/Beam Current** | N/A | I | P | N/A |
| **Working Distance** | N/A | I | N/A | P |

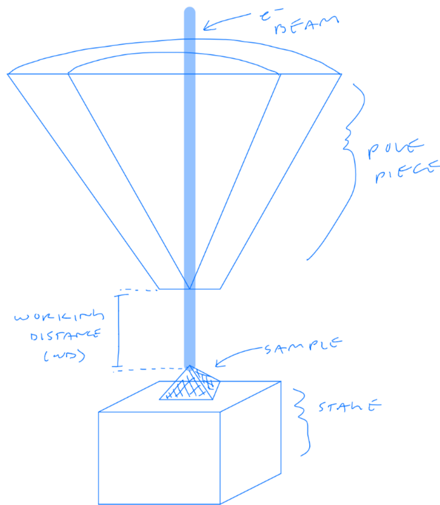
*7) Describe the steps for loading and unloading a sample into the SEM as described in the video. You should have at least 5 steps.*

The steps for a sample to be loaded and unloaded into the SEM are listed below:

1) In order to load the sample into the chamber, disengage the pump to equalize atmospheric pressure to  to open the door. There is no indication for when the chamber is ready to open so wait for a couple of minutes and try to test the door until it opens with little to no resistance.
2) Put on the vinyl gloves when you are ready to place the sample into the chamber. Make sure that you open the door of the chamber until the stop position and grab the premounted sample and place the pin into the mounting stage inside the chamber and tighten it. Measure the height of the sample to make sure that it won't hit the detector when the chamber is sliding under the column. If your sample is too tall, remount it until it fits below the detector height (the required height measurement is specific to machine type).
3) Close the chamber door while keeping one hand on the door and with the other hand re engage the pump to high vacuum level. While the pump is on and processing, center your sample on the stage and set the beam conditions for the intended imaging of the sample.

4) Make sure that the vacuum status reads 'OK' before opening the column valve. This ensures that the pressure is low enough in the chamber for the beam to be emitted into the sample.
5) Finally click on the voltage level indicated in step 3 to start to beam. This opens the column to the chamber and lets the beam hit the sample.

*8) a) Draw a diagram that includes the stage, sample, and pole piece. Indicate the working distance (WD) measurement and how the microscope measures the Z position of the sample before the "link WD to Z" process has been completed.*




When the user is initially looking at the sample the microscope does not know that the working distance measured is from the bottom of the pole piece down to the sample. Therefore, going into the navigation page on the software and changing the z-axis direction from measuring the bottom up to measuring the bottom of the pole piece down will tell the microscope where the sample's surface is.

*b) What is the purpose of the "link WD to Z" process? Describe this process in three steps.*

The purpose of the "link WD to Z" process is to tell the microscope where the sample's surface is. The process of this is as follows:

1) Click the "link WD to Z" button to sync the working distance value as the new z value indicating to the microscope that it is now measuring the z-axis from the bottom of the pole piece to the top of the sample's surface (Top Down).
2) Changing this z-axis value now that it is linked, will allow the user to zoom in on the sample and also change the working distance to the same value (the user may have to refocus).
3) If an error has occurred from a large stage movement, the microscope will indicate that the user re-link so that the focal plane value, working distance, is again linked to the z position.

*c) Why is it important to repeat the focus, link, and drive Z process multiple times?*

It is important to repeat the focus, link and drive Z process multiple times because when initially changing the movement of the stage the position isn't always accurate until the next stage positioning. Re-linking after the first time provides more accurate measurements between the working distance and the z position due to the working distance being tied with the magnification of the microscope.

*9) a) Changing the focus provides visual cues that tell us what alignments need to be done to form the smallest possible probe (and achieve the highest resolution). What are the visual cues that indicate that (i) lens alignment and (ii) astigmatism need to be corrected?*

The visual cue that indicates the lens alignment needs to be corrected is when the user changes the focus of the microscope and the image starts shifting on the screen. For correcting astigmatism, the visual cue is when the user changes the focus of the microscope and there is stretching.

*b) How do you select the correct focal plane for astigmatism correction and what is the process for correcting astigmatism?*

In order to select the correct focal plane to correct astigmatism the user will need to go halfway in between the stretching. When shifting the focus in the image, there will be a direction that the stretching occurs in. The user will have to find a midway focal point where this stretching direction can no longer be seen in both the x and y directions.

*c) After alignments have been done at low magnification, how can you perfect the alignment even further to achieve the best possible resolution?*

In order to perfect the alignment further for overall best resolution, the user should zoom in on a smaller particle shown in the image and apply the reduced window to the area. The reduced area will allow for change on the smaller area and corrections of the lens alignment and astigmatism can be done at a smaller, more precise scale.