HW/Lab 4: SEM, EDS, TEM Kelsey Lawson, 41602126

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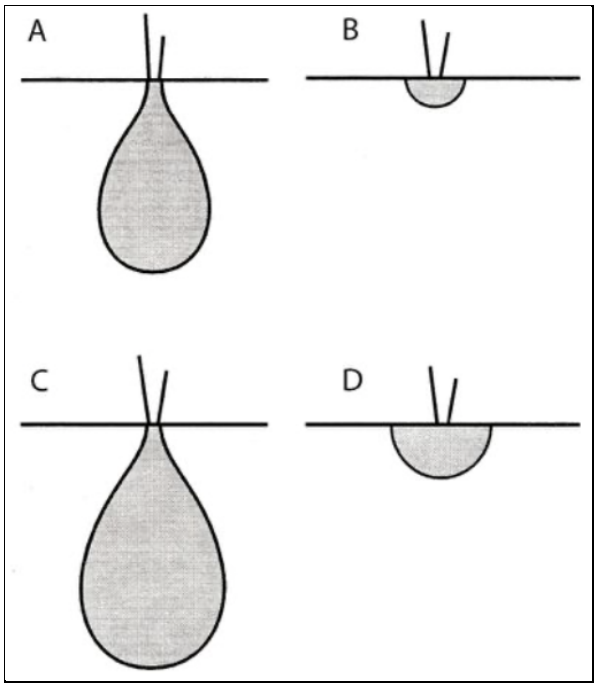
1) In SEM analysis, both the accelerating voltage (E_0) and the mean atomic number (Z) of the analyzed sample volume influence the spatial resolution of the signal emitted. Consider each of the interaction volumes drawn below (A-D), for each of these indicate which combination of mean Z and E_0 is most likely to produce the corresponding interaction volumes shown in the schematic below. Explain your results. (Class Lectures 14, 15, 16)



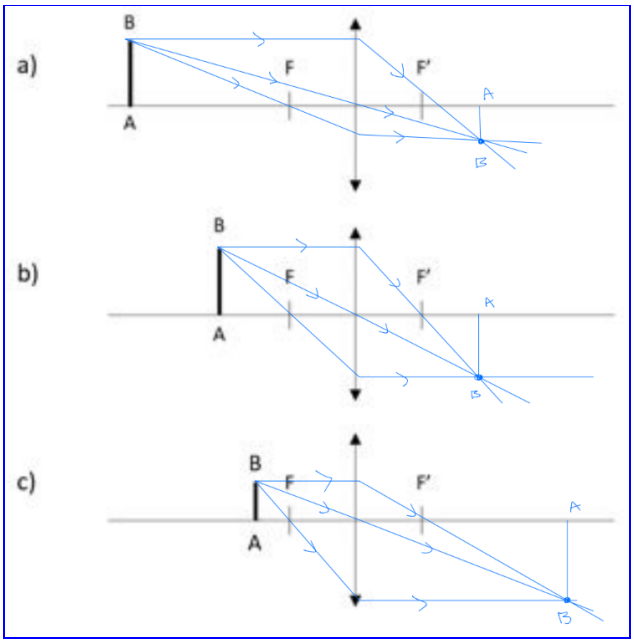
High E_0, Low E_0, High Z, Low Z

(A) Low Z & High E_0
(B) High Z & Low E_0
(C) Low Z & High E_0
(D) High Z & Low E_0

For the images shown in A & C, the reason why the mean atomic number would be low and the accelerating voltage high is due to the Kanaya-Okayama Depth Penetration Formula showing that the voltage and atomic number have an inverse relationship. When the atomic number decreases, the x-ray generation volume also decreases due to an increase in inelastic scattering with the atomic number. Further, as the accelerating voltage increases so does the amount of penetration in the solid. This can also be seen through the KO relationship where the depth penetration and voltage are linearly related, as one increases so does the other, hence the atomic number has to have an opposite relationship to keep the equation equal.

2) The manner in which electromagnetic (EM) lenses focus electrons in electron optics is analogous to the way converging thin lenses focus light in light optics. Therefore, we can understand how an EM lens forms images in scanning and transmission electron microscopes with the principles of light optics.

a) *Using the properties of converging thin lenses, draw the image formed by the object A-B in the following cases a-c shown below using a ray diagram. F = front focal plane of the lens (indicated with a double-headed arrow) F' = the back focal plane of the lens.*



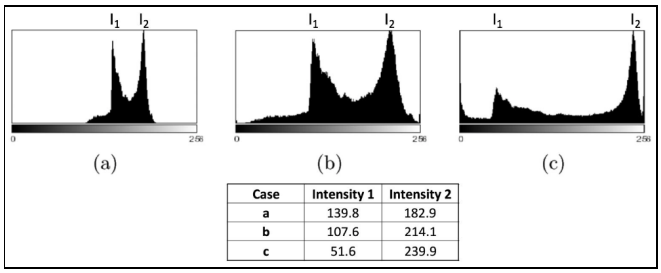
b) *Based on your ray diagrams, how does changing the object distance affect the image distance in each case?*

As the object gets closer to the front focal plane of the lens, the image distance from the back focal plane of the lens increases, or becomes larger. This means that the farther the object is placed from the lens, the smaller the image created.

c) *How is the magnification of the object affected by the relative object to image distance? Use a straightedge to draw your diagrams and refer to: Tutorial if you need guidance drawing ray diagrams. (Class Lecture 13)*

As discussed previously, decreasing the distance between the object and the front focal increases the image distance from the back focal plane. Alongside this, changing the length of AB also has a proportional relationship to the image length. Comparing 'a' and 'c' it can be seen that the length of AB in a is longer before reflecting with a smaller image whereas in 'c' the object has a shorter AB object length yet longer AB image length.

3) Each of the histograms below represents the intensity distribution of an SEM image. The intensity of each pixel in the image is measured from 0 (black pixels) to 256 (white pixels) and plotted along the x-axis. Provide a formal definition of image contrast and use it to give the contrast of each image as a fraction of the highest contrast image. (Class Lecture 16)



Case	Intensity 1	Intensity 2
a	139.8	182.9
b	107.6	214.1
c	51.6	239.9

As stated in Lecture 16, "Image contrast is a measure of relative signal intensity in an image," otherwise known as the color difference that makes an image distinguishable to identify. The relationship between contrast and image intensity can be defined by the equation below:

$$Contrast,\ C\ =\ \frac{(I1-I2)}{I1}$$

Using this equation, for each case, the contrast can be found as follows:

Case	**Contrast**
a	0.308, 0.0844
b	0.990, 0.271
c	3.649, 1

4) Give a formal definition of magnification.

a) How do you change the magnification in a scanning microscope (where you are rastering a probe across the area of interest) versus in a conventional microscope (where the area of interest is illuminated all at once like in problem 1)?

Magnification is a process to enlarge an object's appearance. For scanning microscopes, the magnification can be changed by changing the length of the scan on the specimen. Higher magnification is given to be a smaller sweep of the electron beam and vice versa for lowering magnification. For conventional, or optical, microscopes in order to change the magnification the technician has to manually adjust the lens to a higher magnification lens.

b) Why is it important to have scale bars on your images rather than just indicating the magnification of the image? (Class Lecture 17)

Adding a scale bar to an image allows for the actual object's size to be accounted for. The scale bar value is the actual length of the bar and can then show the degree of magnification the image was taken at.

5) Answer the following:

a) Describe the nomenclature used to describe the characteristic x-ray emission lines.

In order for characteristic x-ray emissions to occur, the incoming stimulation kicks an electron of the inner shell out which then causes an outer electron to fill in the vacancy and in doing do emits energy in the form of a characteristic x-ray. In regards to what outer electron is filling in what shell the following nomenclature applies:

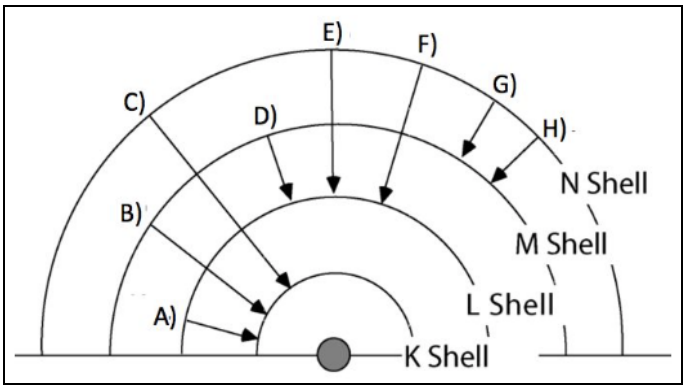
$K_{\propto}$ = L → K shell

K_{β} = → M to K shell

$L_{\propto}$ = M → L shell

$M_{\propto}$ = N → M shell

b) Label the principle x-ray emission lines shown in the figure below. (Class Lecture 19)



A: $K_{\propto}$ E: L_{β} (Outer Shell)

B: K_{β} F: L_{γ} (Inner Shell)

C: K_{γ} G: $M_{\propto}$ (Outer Shell)

D: $L_{\propto}$ H: M_{β} (Inner Shell)

6) Answer the following:

a) *Define the ionization overvoltage.*

Ionization overvoltage is the minimum amount of energy required to remove a valence electron within an atom.

b) *What is the ideal ionization overvoltage to achieve the highest x-ray emission possible and why?*

The ideal ionization overvoltage is as follows: $U = E/E_C > 1$

Where E is the electron beam energy, or the incident beam, and E_C is the critical excitation energy of the shell - the element under analysis in the sample that has an x-ray emission line of some energy. The ratio given above states that the incident beam has to have enough energy to excite the sample-specific electrons to then give rise to the x-ray emission line.

c) *What is the ideal accelerating voltage to use in the SEM if you want to perform energy dispersive x-ray spectroscopy (EDXS) on a*

i) *Silicon sample*

K_{∞} = 1.739; Assuming this is the critical characteristic x-ray emission needed, the accelerating voltage would have to be a minimum of 2 kV in order to excite the electrons.

ii) *Palladium sample*

L_{∞} = 2.838; Assuming this is the critical characteristic x-ray emission needed, the accelerating voltage would have to be a minimum of 3 kV in order to excite the electrons.

(Use this chart to help you. Recall the accelerating voltage range available in an SEM is typically 1-30 kV.)

d) *In addition to considering the critical ionization energy of the electron shell of the element of interest, what other factors should you consider when choosing the accelerating voltage in SEM for EDXS analysis? (Class Lecture 18)*

Although the accelerating voltage needs to have enough energy to excite the electrons, keep in mind that not all the energy being penetrated into the sample is going to be directed in the same way as some of that energy can be absorbed directly into the sample, backscattering can occur or that if x-rays do occur they can be absorbed and create florescence.

7) Describe the three methods of quantitative EDXS analysis. What are the benefits and drawbacks of each of these methods? (Class Lecture 21)

The first method is "semi-qualitative analysis" that compares the data collected from remote standards however, it is difficult to avoid errors from the differences of the microscope, configuration, and samples. Next, "fully standardized quantitative analysis" takes the spectra from the samples and standards using the same instrument for collection and can, therefore, provided higher accuracy in the analysis due to uniform parameters. Lastly is modeling or simulating the spectrum using first principal standards but the amount of instrument and sample parameters needs to be precisely known.

8) In quantitative EDXS analysis, the intensities of the peaks are not accurate measures of weight fractions (or atomic fractions) of the elements analyzed. We have discussed the need for corrections to obtain more quantitative assessments of the chemistry of a sample being analyzed in class.

a) *Explain what the designators Z, A, and F refer to, and for each give a brief explanation of the physical principles behind each contribution to the quantitative corrections.*

Z refers to the difference in mean atomic number that includes correcting for backscattered components and a stopping power component. A refers to the differences in the absorption of x-rays that correct absorbed or escaped x-rays in the sample. F refers to the differences in the production of secondary x-rays known as fluorescence caused by reabsorbed x-rays.

b) *Why should your sample be flat, homogenous, and void-free to enable an accurate comparison with standard samples? If these requirements are not met in each case (e.g. the sample is rough, inhomogenous, and is porous) describe whether you expect an over or underestimate of each of the Z, A, or F corrections. (Class Lecture 21)*

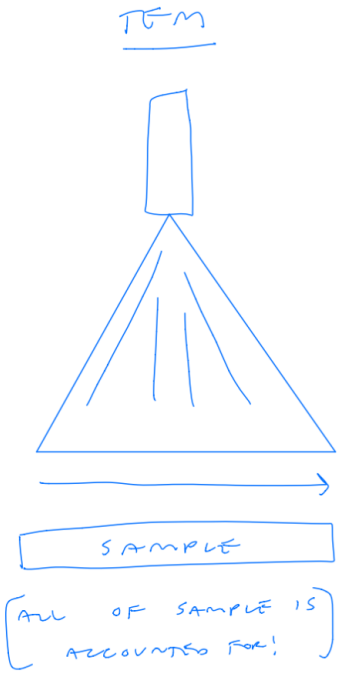
For accurate comparisons of the unknown sample to the standard sample, the unknown ideally should be flat, homogenous, and void-free. If the sample is not flat and has voids, it can affect the reabsorption of x-rays skewing the accuracy of the interaction volume. For nonhomogenous specimens, the depth profile is skewed to detect the same answers but at different locations which will result in incorrect analysis. When the sample contains all of these issues, the Z correction should be undercorrected, A should be overcorrected and F should be overcorrected.

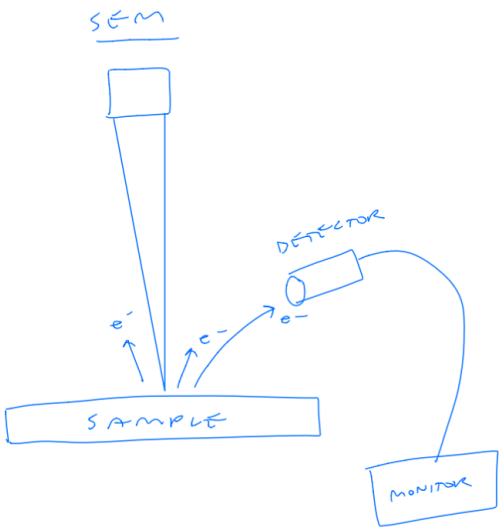
9) List at least three ways that Focused Ion Beam (FIB) capabilities are put to use in modern approaches to microscopy and microanalysis. Briefly describe each process/procedure/approach and the unique attributes of each. (Class Lecture 22)

FIB can induce deposition for materials that result in chemical reactions that can enhance or degrade milling. FIB can also be used to mechanically test nanomachined pillars by using micromachine micron-sized posts. The pillars are then put under compressional loads that result in shear deformation. Finally, FIB can help prep samples for transmission electron microscopy, TEM by removing site-specific thin samples for analysis.

10) Which electron microscopy technique is capable of higher resolution, scanning (SEM) or (TEM) transmission electron microscopy? Give your answer in terms of the expected interaction volume in each case, and draw a schematic to illustrate your point.

TEM should have higher resolution due to the electrons they are gathering data from. Transmitted electrons pass through the sample whereas scanning electrons refer to the reflected electrons off the sample then creating an image. Due to the reflection requirement of SEM, the reliability that all the electrons in the beam are being analyzed without any missing is very low. TEM can be analogous to visible light microscopy but with better spatial resolution.
(Image of comparison below)


TEM
SAMPLE
ALL OF SAMPLE IS ACCOUNTED FOR!


SEM
DETECTOR
e⁻
e⁻
e⁻
SAMPLE
MONITOR