MAT\_SCI 360

Introduction to Electron Microscopy

Spring 2023

# Laboratory 5:

# BSE vs. SE imaging and EDS Analysis

### Introduction­­­­

 By now, you should be familiar with the wide variety of signals generated in the SEM by the interaction between the electron beam and a specimen. Up until now, you have used only one of the signals - secondary electrons. In this lab, you will learn how to use the backscattered electron (BSE) detector of the SEM to gather atomic number sensitive images and use the energy dispersive x-ray spectrometer (EDS) to analyze elemental composition.

**Learning Objectives:** By the end of this laboratory session, you should be able to:

1. Setup and take BSE images to show compositional contrast in a material.

2. Choose when to use SE versus BSE imaging and explain why you have made this choice.

3. Setup the EDS system and acquire x-ray spectra from your sample.

4. Perform a qualitative analysis of your sample.

5. Understand the effects of accelerating voltage and sample charging on the EDS spectrum.

### Part I: Imaging Modes (BSE vs. SE)

 In the previous experiments you were concerned with topographic contrast. Accordingly, we employed the Secondary Electron (SE) detector to image our samples. As you know, this detector has a small positive potential that attracts low energy electrons, thus pulling them into the detector. Secondary electrons are the result of inelastic processes, typically have a low energy (<50 eV) and are therefore attracted towards the detector. If, however, we would like to observe contrast due to compositional differences in the sample, it is useful to accumulate an image from backscattered electrons (BSE's). BSE’s arise from elastic scattering and since the backscattered electron yield is a function of atomic number, BSE's provide compositional contrast.

 For samples of a given composition, say of 2 phases, you could simply calculate the backscatter coefficient (η) of these phases so that you can identify them in the image. As given on page 77 of your text, η is dependent upon the atomic number (Z) by the following polynomial expression:

η = -0.0254 + 0.016Z - 1.86\*10-4Z2 + 8.3\*10-7Z3

For phases with more than one element, the total backscatter coefficient (ηt) is based on a rule of mixtures.:

 To obtain a BSE image you must select the BSE signal under the Image > Detector tab. Remember, BSE’s are an elastic signal, and so the BSE distribution is nearly antiparallel to the incoming electron probe. Make sure that the Chamber Scope (not just the monitor) is turned off. The BSE detector has 5 segments which can be turned on and off independently; for best compositional contrast, you will most likely want to use COMP operation mode.

If the contrast is saturated (too bright) or the signal is too low, lower or raise the preamplifier gain on the detector with the BSE Gain option.

For this section the sample is a metallurgical mount containing tantalum, copper, nickel, iron, and aluminum. Since contrast in BSE mode is sensitive to atomic number, you should be able to distinguish the different layers from the shades of grey produced in BSE signal.

**Take images containing all five layers of metal in SE and BSE modes to see the differences in image contrast. Use 10kV, 10mm WD and reasonable settings for probe current and objective aperture based on your experience from last week. Based on the images you collect in BSE mode, try to determine which metals are in the metallurgical mount.**

**To further explore the effects of accelerating voltage on the BSE image, take a second BSE image at 30 kV.**

### Part II: Qualitative EDS

 The x-rays emitted by a specimen are analyzed using either Energy Dispersive Spectroscopy (EDS) or Wavelength Dispersive Spectroscopy (WDS). When the electron beam strikes the sample, it ejects some inner shell electrons from the atoms of the sample, placing these atoms into an excited state. These atoms will relax from their excited state in several manners in order to achieve equilibrium. As a result of some of these relaxations, x-rays are emitted from the material. The resulting x-rays have wavelengths/energies related to difference in energy levels between which the relaxation occurs. Since these shell transition energies are characteristic of particular elements, the x-ray spectrum provides a "fingerprint" of the sample chemical makeup.

 The x-ray detector on the SEM is a silicon-lithium Si(Li) energy dispersive type with an ultra-thin polymer window. A window is present in front of the detector to shield it from atmospheric pressure, dust, dirt, and water vapor that rush into the microscope whenever the chamber door is opened. If any of these contaminants made their way to the detector, then they could harm the performance or even destroy the Si-Li crystal within the “eye” of the detector. Detectors have ultra-thin polymer windows that allow x-ray collection of elements as light as carbon. However, detection efficiency of light elements is still restricted to some degree.

**Part IIa: EDS identification/Point and ID**

Begin by starting the EDS software, known as Aztec on the computer directly above the SEM Control Screen. The system will ask you to if you wish to create a new project and select a folder to place the project in. Press the “browse” button and select D:\data\MSE360\_2023… as your project folder. The system gives the project a default name of Project 1 which you can change to reflect your lab session and number. Once the EDS software is running, you will notice that it is designed like a flowchart with buttons moving from the left to right at the top of the screen:

Ensure that the first box on the left has EDS SEM selected, and the second box shows “Point and ID” selected by drop down button.

An important concern you must account for in EDS analysis is dead time, which can be monitored on the Aztec control screen in the “rate meter” window. Dead time is controlled by adjusting the process time, or pulse processor speed in the detector system. A fast process time (1) will produce a very low dead time, whereas a slow process time (6) will increase the relative dead time. When collecting at a faster process speed, the X-ray peaks tend to broaden, and overall resolution decreases. Slower process times produce narrower, higher resolution peaks, but more counts are thrown out and collection times are longer. As a rule of thumb for general Qualitative analysis, a dead time between 30-50% is a good target. Dead time can be adjusted in the *acquire* spectra tab, clicking *settings*, and selecting a faster or slower *process time*.

To investigate Qualitative EDS analysis, we will look at the same sample as Part I. To begin, go to the image tab and look at the *settings* button in the top center of the control screen. Select an appropriate pixel resolution and be aware that you can adjust the scan speed with the *dwell time* drop down control. You can next push the green *start* button on the main screen to take the photo. After the photo is complete, move on to the next tab: *Acquire Spectra.*

The main toolbar in the Acquire Spectra tab is on the left-hand side of the photo taken in the previous frame. As with the previous menu, there is a settings tab in the upper center screen that you can select to adjust energy range, number of channels, process time, acquisition mode and time. Typically, select 20kV, 2048 channels (~1eV/Channel), live time, and a set number of seconds (e.g. 30s). At this point you can take a spectrum of the full field by simply pressing the *Start* button near the top center of the screen. You can also use the toolbar illustrated to select points, rectangles, spherical areas, or irregularly shaped areas. Select the rectangle tool, and have the system collect a 30s spectrum from each of the five metals you identified with the BSE detector in part 1. Note how the deadtime and counts change in the ratemeter window when the system moves from metal to metal.

After you have collected your spectra, move on to the *Confirm Elements* tab of the Aztec Program. In this window, you can select each of your spectra from the data tree on the right-hand side of the screen. The quickest way to start with identification is to use the Auto ID tool at the bottom of the screen to get preliminary identification on your peaks. You can also use the mouse to double-click on a peak and see suggested matches for it on the right-hand side of the window. You can select elements individually using the periodic table to see all of the relevant energy lines and a representation of their expected positions and heights. The Settings button in this screen gives you the option of showing modeled peak shapes and a theoretical spectrum if desired. Select each of your spectra collected from the five metals and confirm the elements present. You should have identified layers containing Ta, Cu, Ni, Fe, and Al.

The Final window in the Point and ID screen is to *Calculate Composition* based on the spectrum you have collected. You can select either *Weight* or *Atomic* percent as options for output.

In this window there are several available templates on the left-hand side of the screen. The uppermost of these is the summary table. With this view, it will show you the elements identified and confirmed for the spectrum selected in the data table on the right-hand side of the screen. In the settings window, you have the option of selecting specific elements from the periodic table as a fixed list or using the current active spectrum for the composition. You can also put in a de-convolution element if you have coated a sample with a conductive material that is not part of your composition. This will calculate the composition with the element present, and then subtract it from the values in the composition output.

For each of your spectra, you can generate a report using the *Report Re*sults button on the upper right-hand side of the screen. You can also right click on the spectrum window and export the spectrum data in raw EMSA format. The standard report output for the Point and ID settings is an excel report called Image-Spectrum-Quant, which will create a single page excel file with these three items on the page. After you have generated your first report, you can select the *Append* option from the reports drop-down menu, and a new page will be added to the existing excel file. Create a report for your Spectrum 1 and append it so it includes each of the 6 spectra collected so far. Also export the raw data from each of your 5 layers in EMSA format.

**Part IIb: Qualitative EDS**

For this portion of the lab, find an area where you are focused on three different metals in your field of view as in the image below (either horizontal or vertical, but alignment should be parallel to one major axis to simplify the experiment):

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Target each metal represents approximately 1/3 of the field of view, but we will be able perform measurements on the photo to get a better feel for the area represented. I have measured the length of the box and units above, and I can see it is approximately 82mm across, with the first section measuring 29mm (35%), the second 29mm (35%), and the third 24mm (30%). The EDS system has a tool shaped like the letter “A” in the imaging window that will allow you to select a caliper and quickly take measurements of your relative sizes, or you can do this with a ruler after the lab. In the *Scan Image* tab of the Aztec program collect an image of the three metals.

Once you have taken your photo, proceed to the *Acquire* tab and check the settings to ensure that your deadtime is reasonable (30-50%). Set the acquisition to collect a 60s spectrum and then collect a spectrum of the full field by pressing the *start* button. Now collect a rectangular area spectrum from each of the three layers for 60s. Proceed through the Confirm Elements and Calculate Composition Windows as with Part IIa and generate an Image-Spectrum-Quant report for the spectra (you can append to existing report). Also export your full field and individual layer spectra in EMSA format.

**Part IIc: EDS Mapping**

For this section, return to a low magnification condition where you can see all 5 layers of metal in the sample. From the second tab in the button bar, select the drop down menu and select *Map.* The mapping menu is slightly different from the *Point and ID* menu as seen below:


As with the previous parts, each of the tabs in the menu bar has individual settings for selecting imaging conditions and then map conditions.

Acquire a photo in the Scan Image Tab (all controls are the same as with *Point and ID*), and proceed to the Acquire Map tab. In this section, select a relatively low resolution (512) image quality, and keep the process time at a condition that gives you approximately 30-50% dead time. Select a fixed duration acquisition time, and have the system collect 10 frames for your map. For the energy range and number of channels, select 20kV and 2048 (~1eV/Channel). Hit the *Start* button, and the system will begin collecting data from your full field of view. Note that there is a toolbar on the left-hand side of the screen that does allow users to collect maps from a selected area within the field of view as well (at right). Proceed to the Construct Maps button. In this final screen, you should be able to select our five elements of interest (Ta, Cu, Ni, Fe, and Al). Using the Report button, select a report format such as Electron Image and Maps or Layered Image and Maps and either save as a new report or amend to the existing Point and ID report.

**Qualitative EDS - Discussion**

In Part IIb, you collected a 60s full field spectrum of three layers as well as individual layer spectra. We aligned our image in part IIb in a manner that we can calculate the relative areas of each layer. Compare this with the quant numbers generated by the EDS system in your full field EDS output. You may notice that the quant numbers are different from the area fractions of each element. Using the raw data collected from individual layers and the full field map, compare the ratios of the dominant peak energies between the single layer maps and the full field. Does the peak ratio give you a better calculation for the relative amount of a given metal?

Looking at the maps just collected, you should notice that the Tantalum and Copper maps look a bit unusual, with overlapping distributions for both elements. Since we have already completed Point and ID on these layers, there shouldn’t be any confusion that we are looking at two metals. The reason for this overlap, is a peak overlap between Tantalum (Lα 8.14 KeV) and Copper (Kα 8.04 KeV). Because EDS mapping uses a range to assign elements to a location, the relative proximity of these energies is creating an overlapping distribution even though we are sure that these are two distinctive layers. How can we use the system to improve the mapping results for Ta vs. Cu?

**Tangibles:**

The laboratory report is due *one week* after your laboratory session and should be submitted electronically. Each group should hand in one laboratory report for all three members. Please follow the instructions at the end of this laboratory report. In summary, for laboratory report you should be prepared to hand in the following:

1. Micrographs and explanations of the micrographs comparing the SE and BSE images from the two samples. Explain, in detail, the differences between the SE and BSE images and the differences in BSE images with different accelerating voltages.

2. An EDS spectrum from each phase in the Laminated Metal sample with correct identification of all peaks on the spectrum as described in the qualitative EDS section. Discuss any unusual peaks identified in the spectra.

3. Comparison of the area fraction, peak ratios, and automated quant on the region of 3 metals identified in part II.

4. EDS maps showing system generated maps and maps created to correct for peak overlaps.