

INTRODUCTION TO THE SEM/EDS OR “EVERY COMPOSITION TELLS A STORY”

John T. Cheney and Peter D. Crowley

Department of Geology
Amherst College
Amherst, MA 01002
jtcheney@amherst.edu

Purpose

This lab is designed as an introduction to the investigation of minerals with the Scanning Electron Microscope (SEM) and the X-ray analyzer (Energy Dispersive Spectrometer - EDS). We will explore the relationships among the optical image, the backscattered electron image, and the chemical composition of minerals in thin-sections that we have studied optically. Specifically we will examine the interaction of a mineral with a high energy (20 kv) electron beam focused to a diameter of a few microns. Three different detectors of the SEM/EDS will be used to study minerals in thin sections. *Additional concepts that may be introduced in this lab include: formula calculations, solid solution, exsolution, and chemical zoning.*

A. Optical Review of Assigned Sample

Be careful not to touch the polished surface of the thin sections used in this lab as they have been coated with carbon in order to enhance the flow of electrons from the sample when it is in the SEM.

1. Identify the minerals and describe their shape, size, and habits. Note distinctive features within individual mineral grains such as systematic variations in color, pleochroism, extinction, or refractive index (relief).
2. Use the video camera-computer-petrographic microscope to capture and then print an image of an interesting or representative area of the slide. The image should be at a magnification consistent with the grain size. For example, a low power (10X) field of view for a medium grained rock is a good starting point. Try 3.5X for coarse-grained rocks and adjust for features of interest. Remember that the printed image of the slide should be at the same magnification that you will use to “see” the minerals on the SEM. In order to find the selected area on the SEM, the area should be marked with dots from a black “Sharpie” pen on the carbon-coated thin section. You should make a sketch of the thin section showing the location of the selected area on the slide.

B. Backscattered Electron Image

Backscattered electrons are high energy electrons from the primary electron beam that are backscattered or repelled by the negative charges associated with the electrons of atoms comprising the mineral. The higher the electron density of the sample the greater the number of “gun” electrons that are backscattered. The higher the average atomic number of the atoms comprising the mineral the greater the electron density. The backscattered detector images these repulsed electrons.

1. Using a low magnification (~10x), locate the area of interest using the “Sharpie Dots” as a guide. With the magnification of the SEM set the same as for your optical photo, take a photo of the backscattered electron image (BSI) of the same area.

2. Describe the BSI photo of the sample or the BSI image on the monitor. Specifically, describe in what ways the BSI photo is similar to the “optical image”. Pay particular attention to the shapes, sizes and habits of the constituents.
3. Does the mosaic of constituents resemble the distribution of minerals in the “optical image” of the slide, explain.
4. Can you associate the objects in the backscattered electron image with specific minerals? How? What observations and/or properties allow you to determine this relationship?
5. How does the “backscattered electron” image differ from the “optical” image. Consider for example, color and/or brightness. Which mineral are bright and which minerals are dark. Make a list of the minerals in order of increasing brightness. What chemical property do the different bright minerals have in common? In what ways are the different darker mineral similar in composition?

C. Secondary Electron Image

Secondary electrons are the electrons from the mineral that are “knocked” off the surface of the mineral by beam electrons. Secondary electrons (SE) are imaged by a detector located above and to one side of the sample. Sample surfaces inclined toward the detector have a higher SE contrast than those surfaces inclined away from the detector. Secondary or low energy electron imaging of the surface of the sample is particularly effective for studying 3-dimensional topography at high magnification, such as in biological samples like the eye of a fly.

1. Describe the SE “picture of the sample” on the monitor. Specifically, in what ways is the image similar to the “optical” image and the “backscattered electron” image. Pay particular attention to the shapes, sizes and habits of the constituents.
2. Can you think of uses for secondary electron imaging in the study of minerals in polished thin-section? In polished thin section, the best use of the secondary electron image is to find holes in the slide, dust particles, and locate areas that are charging due to poor carbon coating. How about the study of mineral grains or powders?

D. Qualitative Chemical Analysis

In addition to being backscattered and generating secondary electrons, the BEAM electrons penetrate the mineral and displace inner shell electrons of the constituent atoms. As outer-shell electrons back-fill the inner-shell vacancies they lose energy by the emission of X-rays. The energy level (or wavelength) of the X-rays is characteristic of the element excited and the amount of energy is proportional to the number of atoms of that element in the small excitation volume. An energy dispersive X-ray spectrometer collects the X-rays for a fixed period of time (typically 30-120 seconds) at all energy levels simultaneously and an on-line computer is used to convert the data to a spectrum of X-ray intensity (“counts”) versus energy level. The resulting graph is thus a sequence of “peaks” at energy levels corresponding to specific type of atoms. The software will label the peaks with specific elements. The energy level of the K transition peaks increases with increasing atomic number so that the left to right sequence of peaks is Na, Mg, Al, Si, K, Ca, Ti, Mn, Fe, Zn etc.

1. Now we can do some chemical petrography. Begin by obtaining a spectrum on one of the brightest minerals in the backscattered image. List the elements that make up the mineral. Suggest one or more possible identities of the mineral. How could you differentiate among these possibilities?
2. Repeat the above experiment on the other minerals in the area of interest in your slide. Can you now look at the BSI photo of the and predict the composition and name of each grain in the photo? If so why? If not, why not? Please explain.

E. Quantitative Chemical analyses

The computer has software that converts the X-ray spectrum to a quantitative analysis of the mineral by comparing the spectrum obtained from a mineral in your sample to a set of standards stored in the computer memory. This data is typically presented both as Wt% oxide and moles of atoms per formula unit of oxygen in a chemical formula such as Mg_2SiO_4 or $Mg_{0.37}Fe_{0.63}SiO_4$.

1. Determine the chemical composition of one of the minerals in the rock. How many experiments must you perform in order to do this? What are some of the reasons that the analyses are not all the "same"? When are two different analyses the same? How might you determine when two different numbers are the same or when they are different?
2. How helpful is a working knowledge of mineral compositions for using the SEM/EDS to do petrography?
3. Compare and contrast the SEM/EDS and the petrographic microscope as a tool for identifying minerals and determining the composition of the minerals. How would you tell the difference between kyanite and sillimanite - with the petrographic scope or the SEM?

