Lakehead UNIVERSITY | Centre for Analytical Services

Technical Bulletin

Geological results with the Scanning Electron Microscope (SEM)

How the SEM helps you identify minerals in rock samples

By Colleen Kurcinka

Introduction

The scanning electron microscope (SEM) is a microscope that passes an electron beam over a sample to excite the surface electrons. It then detects electron changes and produces and image. The SEM can view samples at up to 50,000x magnification!

The SEM is an important instrument in geological analysis and the information it provides differs from other instruments.

- Compared to X-Ray Diffraction (XRD) scans which can give you a phase analysis in less than an hour, the SEM is more involved. Doing phase analysis on an SEM entails acquiring spectra for each individual phase. A skilled operator can ID minerals quickly using grey scales to separate individual phases and use elemental analysis to identify them. However, although both can get you to essentially the same approximate conclusions, XRD uses software to perform a semi-quantitative phase analysis on your sample more quickly and easily than the SEM. The SEM can also allow you to infer mineral associations while the XRD cannot.
- Compared to an Inductively Coupled Plasma (ICP) which gives you elemental analysis for the whole rock, the SEM can provide elemental analysis for each phase at a user defined point, which can be as small as 3 microns. This makes the SEM extremely useful for research purposes.
- Compared to an optical microscope, the SEM has a greater depth of field and greater magnification. An electron beam has a smaller wavelength than visible light, so it can view objects in at higher resolution and therefore higher magnification. The disadvantages to SEM over optical include complex sample preparation, high costs of running and maintenance and the fact that samples must be dehydrated. This means living samples cannot be viewed in an SEM as they can under an optical microscope due to the high vacuum in the SEM. Only non-living biological samples coated with gold can be viewed by the SEM.

There are two basic signals produced by an SEM: secondary electrons and backscattered electrons. Secondary electrons are produced when the SEM projects a low energy beam onto a sample and weakly moves an electron from the surface of the sample. A secondary electron detector can receive a signal from these surface electrons and can use this to accurately depict the surface of the sample. The result is a high resolution image that allows the topography of the sample to be shown (Barnes, 2003). Backscattered electrons are of a higher energy state when excited by a beam. Because of this extra energy, the electrons can come from deeper within the sample and therefore is not an ideal method for showing surface topography (University of Georgia, nd). Collisions will occur between electrons and nuclei reflect these back to the detector. An element with a higher atomic number is more likely to have its electrons reach the detector than that of a lighter element, resulting in the creation of a brighter area spot in the image in comparison (Barnes, 2003). Unlike secondary electron imaging, backscattered imaging does not show topography but rather shows the different phases of the sample in grayscale.

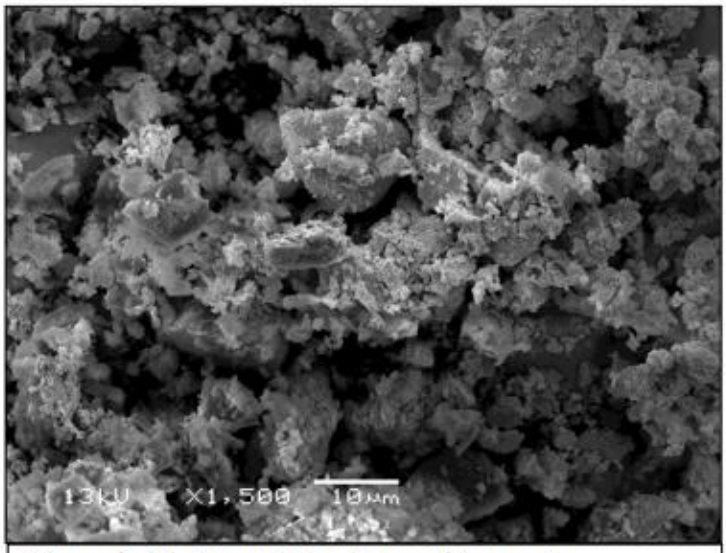


Figure 2: High resolution image of a powder containing zeolites and quartz. Photo by A. MacKenzie

Quantitative Use

Geologists tend to make use of the energy dispersive spectrometer (EDS) to produce X-rays from the sample, which can be used to quantify elemental percentages and identify minerals. By hitting a sample with an electron beam, electrons are removed from a shell orbiting the nucleus. To compensate, electrons from outer levels fall to take their place and by doing so, emit a characteristic X-ray particular to each element (Barnes, 2003). Quantification of elements is possible using this technique and can have 0.2 to 1.0 wt% accuracy. Light element X-rays are absorbed by the detector window and do not actually reach the detector making them difficult to quantify and can only be estimated. Textual relationships between minerals can also be observed as minerals remain in situ (Reed, 2005). Standards are used to ensure quantitative accuracy of the results.

The Instrumentation Laboratory at Lakehead University has a JEOL5900 LV/Oxford Link ISIS and has many metal, silicate, sulphide, PGM and REE standards.

High Resolution Imaging

High resolution imaging can be useful for structural analysis of a sample. It can show interesting features not visible at lower magnification, such as sheet structures and needle structures. Powders and their mineral grains can be imaged by mounting them on to a 1 cm stub and then coating them with gold. This is the same way that we obtain detailed information about micro-organisms, such as cells, fossils, and semi-conductors and other examples in which it is necessary to view surface topography.

Elemental Maps

Elemental maps are created to show the spatial relationship of elements or phases to each other in an image. These are used when trying to analyze textures or to determine chemical zonation of a crystal, for example. Elemental maps are particularly useful in areas where the

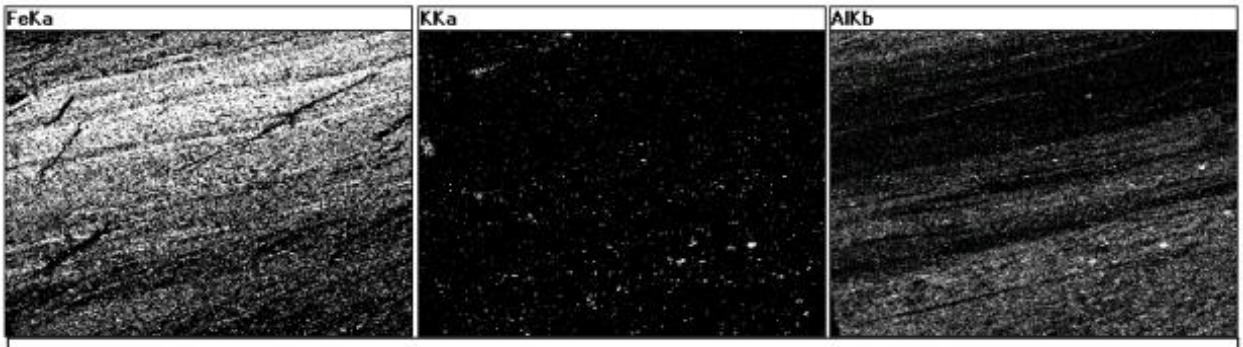
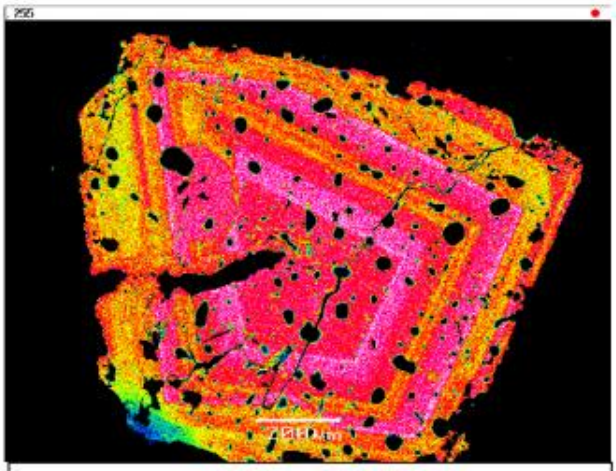


Figure 3: From left to right, shows an elemental map of iron, potassium and aluminum in a sedimentary rock. Photo by P. Fralick

average overall colour is uniform so you can distinguish between phases (Goodge, 2003). To do this, an electron beam will scan the area of interest at a chosen resolution and determine elemental concentration at each point (Goodge, 2003).

The above grayscale photo shows sedimentary layers as shown by an elemental map. White represents high concentrations whereas dark represents low concentrations. The spatial relationship in this area shows that iron concentrations are high where aluminum concentrations are low, and vice versa. Potassium concentrations are consistently low throughout the area in view.

False colour can be used in element mapping to make the presentation of the map more appealing and easy to read. The above photo shows oscillatory-zoned uranoan pyrochlore. The compositional banding is easy to distinguish using this type of image.



A false colour element map of uranoan pyrochlore. Photo from Zurevinski and Mitchell, 2004.

Overall advantages of the SEM

- BSE techniques allow for the identification of multiple phases in a sample due to a difference in grayscale colour (Pye and Krinsley, 1984).
- Rare earth minerals, zircons and other small inclusions can be detected (Pye and Krinsley, 1984).
- Non-destructive
- Quantitative analysis to ±1% for major elements
- High spatial resolution
- Textural relationships can be observed (Reed, 2005)

Scanning Electron Microscope (SEM) Applications

The use of SEM in analytical techniques has many versatile applications.

- Mining and exploration companies interested in mineral identification
- To identify zoning in crystals
- Modal analysis of minerals in a sample and particle sizing
- Studies of fossil preservation in sedimentary rocks
- View microscopic details of a sample

How can we do this?

Once a sample is received, a thin section is made in which the back side is polished until no surface topography remains. They are then carbon coated and ready to be analyzed. Although the sample is physically manipulated, this process is classified as non-destructive because there is no loss of the sample after being used by the SEM.

References

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