Theory and Practice of Electron Microprobe Analysis

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F - 304

Introduction

- This course covers the principles, techniques and applications of electron microprobe analysis.
- Topics covered include X-ray theory, principles of electron microprobe analysis, sample preparation techniques, hands-on analysis using the electron microprobe, and techniques of instrument calibration and data reduction.
- The emphasis is on analysis of geological samples, which is required for everyone, who plan to use the electron microprobe as part of their research.

Introduction (cont.)

- The classes will be divided between sample preparation and analysis subjects.
- The class will be geared towards quantitative analysis of geological, mining, and materials samples, and will cover SEM morphological imaging in less detail.

Class Syllabus

- **Class 1:** Theory and history of x-ray analysis. Introduction to instrumentation. Lab session will consist of a detailed tour of the SX-50 facility, and familiarization with the SX-50 instrument.

- **Class 2:** User's introduction to the SX-50. Navigating in Cameca software. What can you do with this instrument, and how?

- **Class 3:** Imaging techniques. The theory behind acquisition of scanning electron, backscatter electron, and X-ray images and maps. The lab will include hands-on collection of electron and X-ray images.

Class Syllabus (cont.)

- **Class 4:** Sample preparation and sample mapping. Lab session will include demonstration of sample mounting and polishing techniques.

- **Class 5:** Electron microprobe calibration, data acquisition, and data reduction techniques. Includes discussion of standards, analytical error and assessment of quality of data. Lab session will include demonstration of calibration procedures, quantitative and semi-quantitative analyses.

- **Class 6:** Data management and presentation. Will include discussion and demonstration of procedures for getting your quantitative and imaging data from the Cameca into your thesis or paper. Familiarization with data output formats and basic techniques of image manipulation.

What is an Electron Microprobe and what can it do?

- ***What is an electron microprobe?***

  An electron microprobe is a high powered microscope that uses electrons, rather than light, to examine a sample. Because electron are charged particles, they can be accelerated towards, and focused onto, a sample surface.

What is an Electron Microprobe and what can it do? (cont.)

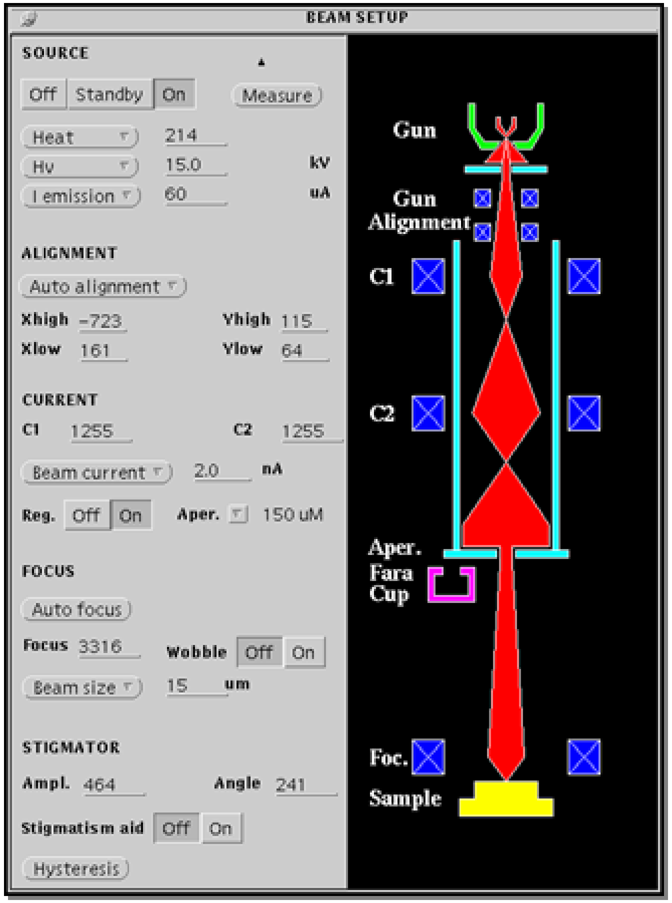
- ***What can an electron microprobe do?***

  Electron microprobes can be used to produce morphological (roughness and shape) or chemical images of a sample surface. The microprobe can also be used to qualitatively or quantitatively determine the chemical composition of a very small spot (1 micron) on a sample surface. The electron microprobe can be used to chemically characterize samples so small that they can be analysed by virtually no other analytical technique, and also allows spatial resolution of chemical variability on a sample surface.

  The capabilities of an electron microprobe are fundamental to many types of research, including many aspects of geology, such ore deposition, environmental geochemistry, hydrology, petroleum research, igneous and metamorphic geochemistry, soil science, and sedimentology, as well as various aspects of biology and metallurgy.

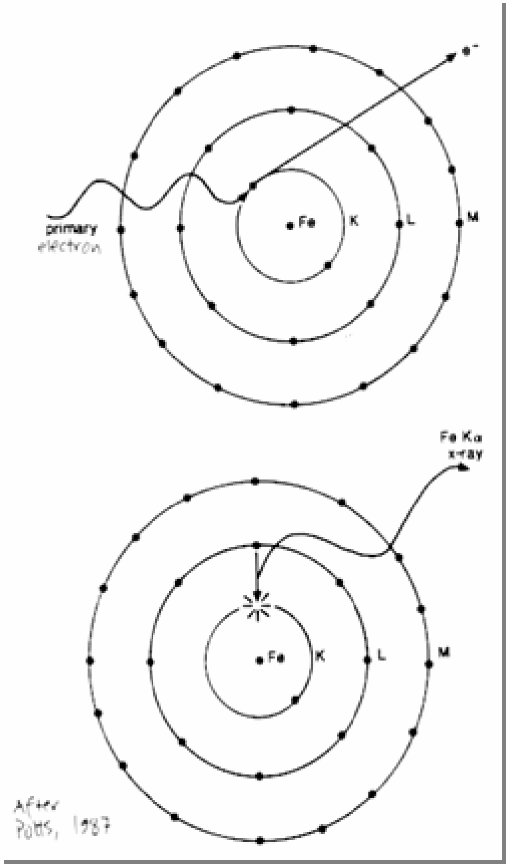
How does an electron microprobe work?

- Electrons are produced by heating a tungsten filament to high temperatures (in the gun) and those electrons are accelerated away from the filament and gun towards the sample surface. Because the electrons are charged particles, they can be directed and focused using electronstatic lenses (gun alignment, C1, C2, foc, see left).
- The electron beam is directed onto the sample surface, and can produce secondary electrons, backscattered electrons, cathodoluminescence, or X-rays that can be analysed to determine the sample's composition.



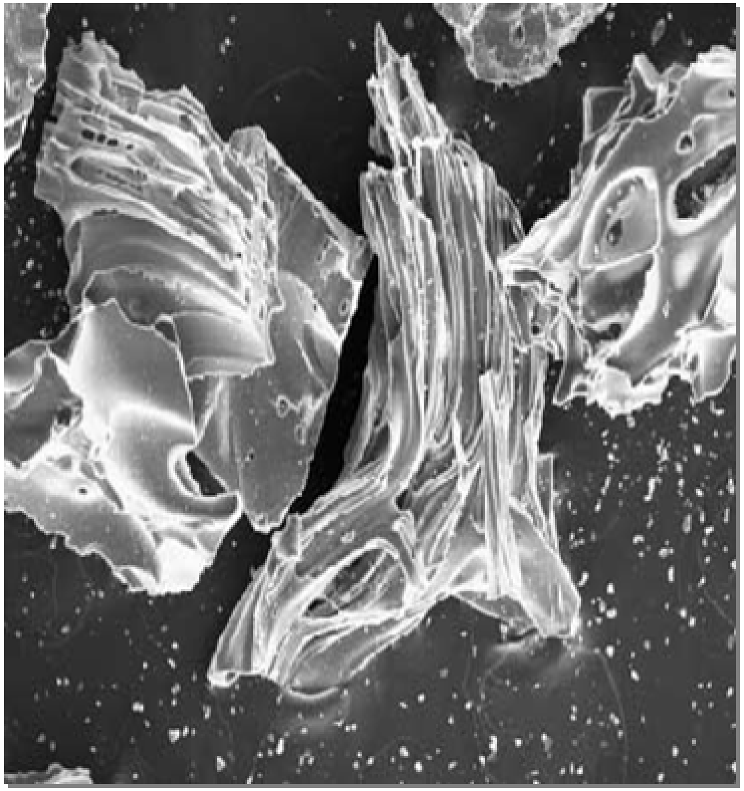
How does an electron microprobe work? (cont.)

- As electrons from the primary beam hit the sample, they produce a number of secondary particles.
- **These include:**
  - Secondary electrons
  - Backscattered electron
  - Auger electrons
  - Cathodoluminescnce
  - Continuous X-rays
- Interaction between the electron beam and the sample also produces characteristic X-rays, that have a wavelength and energy proportional to the atom from which they are produced. An electron from the primary beam causes an electron from the target atom to be ejected from its orbital shell. In response to the empty energy shell, another electron will cascade down to fill the lower energy position, in the process releasing energy in the form of a characteristic X-ray.
- These characteristic X-rays can be detected by spectrometers to determined something about the sample composition.



Secondary Electron Images

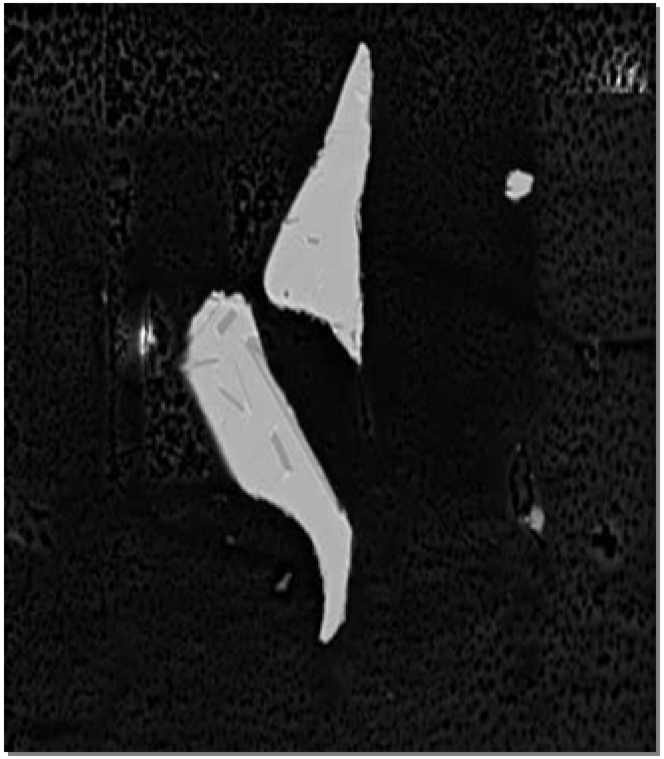
- Secondary electrons are produced when the microprobe electron beam interacts with the sample surface, producing low energy secondary electrons that are detected by the secondary electron detector. A secondary electron image appears 3-dimensional. A well-tuned instrument can produce secondary electron images at magnifications of 100,000x.



Volcanic ash shards from Mt Erebus, Antarctica

Backscattered Electron Images

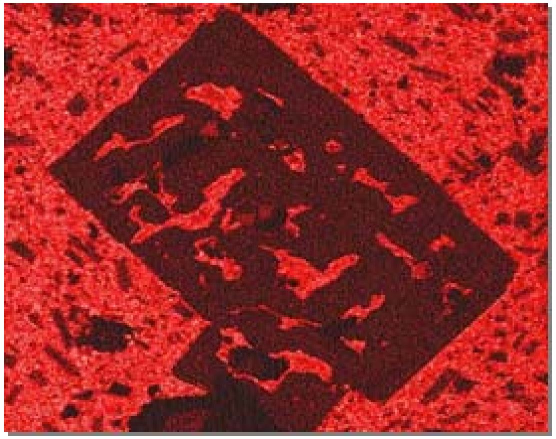
- Backscattered electron images can be generated from samples with relatively smooth surfaces, as opposed to secondary electron image that rely on sample roughness to produce an image. Backscattered electrons are high-energy electrons that rebound from the sample surface.
- The quantity of electrons backscattered from a given surface is proportional to the mean atomic number (Z) of the sample material. Hence, a high mean Z material will produce more backscattered electrons than a low mean Z one. In the sample shown below, the brightest areas are magnetite, a high Z Fe oxide. The next brightest areas are pyroxene crystals that contain Fe, but also lower Z elements, such as Si, the medium grey areas are volcanic glass, and the darkest grey areas are feldspar.

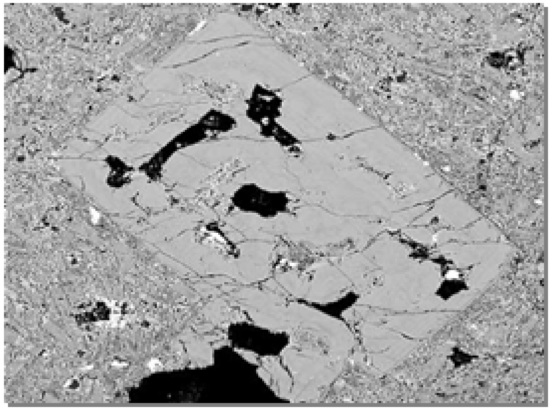


Volcanic ash shards from the Siple Dome icecore (Antarctica) Field of view = 100 microns

Map of potassium distribution in altered feldspar

- Chemical maps of sample surfaces can be produced by detecting X-rays generated from the sample surface. The following image shows the potassium (K) distribution in an altered volcanic feldspar, and the surrounding glassy matrix.
- The intensity of red color indicates the amount of K present on the sample surface. A backscattered image of the same feldspar can be seen below the K map.

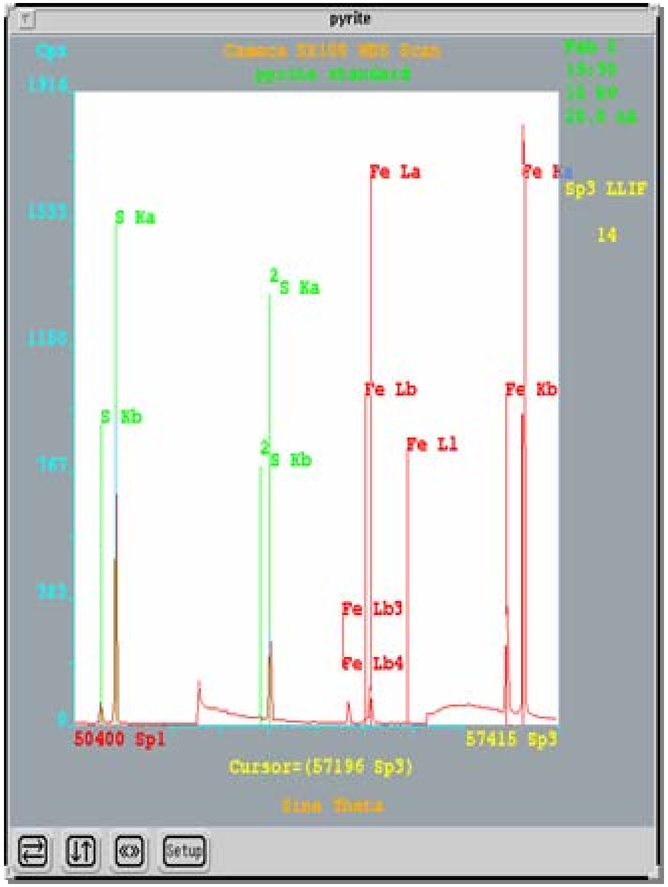




Field of view is 400 microns

Qualitative Analysis

- The elements present in an unknown sample can be determined qualitatively by analyzing the characteristic X-ray production from the sample. The following diagram shows a characteristic X-ray scan collected on a sample of pyrite, a mineral that contains Fe and S. X-ray peaks corresponding to those elements are labelled on the scan.



Quantitative Analysis

- The electron microprobe can be used to make accurate determinations of the chemical composition of a very small (1 micron) spot on a sample surface. Quantitative analysis is accomplished by comparing the number of characteristic X-rays for a given element generated by an unknown sample with the number generated by a standard of known composition. An average geochemical analysis takes between 2 and 10 minutes to complete.

Quanti Report

Point #3 an-3

	Concen.	Cations
SiO2	44.183	2.048
Al2O3	35.447	1.937
CaO	19.223	0.955
FeO	0.380	0.015
SrO	0.105	0.003
BaO	0.005	0.000
Na2O	0.551	0.049
K2O	0.017	0.001
Total:	99.911	5.008

This is an example of a quantitative analysis of a plagioclase feldspar. The analysis is recalculated in terms of oxide weight percent (first column) and also in terms of cations for 8 oxygen atoms.

Selected References

Some of these books, along with a number of others, are ideal reference and few are available in the microprobe laboratory.

- **Cameca SX- 50: Operations Manual – Maintenance and Troubleshooting**
- **Cameca SX- 50: Operations Manual – Reference Guide**
- **Cameca SX- 50: Operations Manual – User's Guide**
- **Potts, P.J., 1987. *A Handbook of Silicate Rock Analysis*. Blackie: Glasgow**
- **Potts, P.J., Bowles, J.F.W., Reed, S.J.B., and Cave, M.R., 1995. *Microprobe Techniques in Earth Sciences*. Chapman and Hall: London**
- **Reed, S.J.B., 1993. *Electron Microprobe Analysis*. Cambridge University Press: Cambridge**
- **Reed, S.J.B., 1996. *Electron Microprobe and Scanning Electron Microscopy in Geology*. Cambridge University Press: Cambridge**