

# Eugene Cameron Electron Microprobe Lab

## Department of Geology and Geophysics

### University of Wisconsin – Madison

#### General Principles

An electron gun shoots focused high energy (15–25 keV) electrons at the specimen, which responds by emitting a variety of signals, which can be captured with various detectors. One interaction that occurs is an “inner shell ionization” where an electron close to the nucleus of an atom is knocked out of the atom and another electron in the atom takes its place, and emits energy of a specific value (either an Auger electron or an x-ray), which can then be detected.

The electron microprobe is a specialized electron microscope which provides precise chemical analyses of micron size regions of a sample (~picograms) with high spatial resolution. Besides providing a table of elemental concentrations, it can map out the distribution of elements in a specimen (“x-ray map”), as well as operate as a scanning electron microscope and give backscattered electron and secondary electron images.

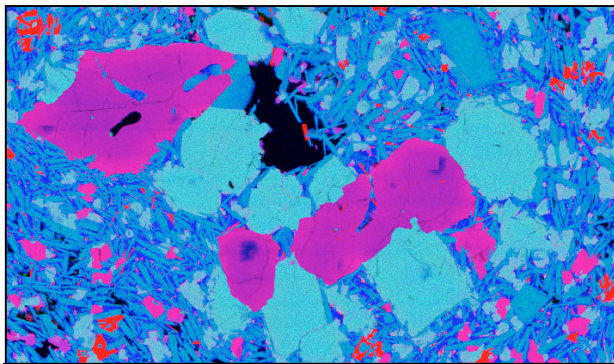
We detect these x-rays with a specialized wavelength dispersive spectrometer (WDS) that utilizes a crystal to diffract the x-rays (Bragg's Law) into a gas-filled tube where an electronic pulse is produced and counted. Electron probe WDS technique uses standards to quantify the elemental abundances in the unknown by ratioing counts from the unknown for an element with those from a known standard, under the same operating conditions. Each element is measured separately, and when summed up, should be between 98–101 wt%, which is one test of how good the analysis is.

Traditionally the electron probe has been used by geologists and material scientists to study rocks, volcanic ash, meteorites, semiconductors, alloys, ceramics, glasses, and superconductors. Other applications have included mapping metals in treated lumber, identifying crystals on cheese, and determining the chemistry of ancient (and modern) bones. Biological tissues may also be analyzed.

WDS differs from EDS (energy dispersive spectrometry) – available on many SEMs – in that EDS typically “normalizes to 100%” and thus gross errors in the process can be obscured. EDS is useful, when used properly – we have an EDS detector on our microprobe for qualitative analysis (quick elemental identification).

Not all materials are analyzable in the electron probe – but most are, if the proper steps are taken. Samples must either have a mirror flat surface, or be mounted in a material (epoxy) and polished to a mirror finish, and they must be small enough to fit in a holder. Most specimens are a fraction of an

inch wide, but it is possible to accommodate some thin samples up to 2 ½” in diameter.



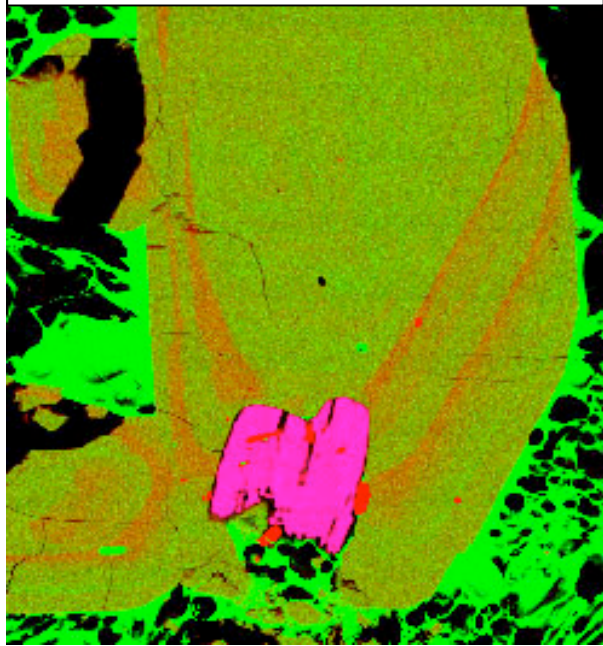
Aleutian Lava: 3 X-ray maps (Fe, Ca, Si) superimposed as RGB composite showing glass (darkest blue), olivine (purple), Fe-oxides (red), pyroxene (light blue) & feldspar laths (mid blue)

#### Instrument Capabilities

The only electron microprobe in Wisconsin is located in our lab. The Cameca SX51 has 5 automated wavelength spectrometers and with 12 crystals it can cover the periodic table from Be through U. Anti-contamination devices are present to facilitate light element analysis. With precise autofocusing, samples can be left to run unattended overnight. Backscattered and secondary electron as well as cathodoluminescence images are routinely collected. X-ray mapping is

1966–2006: 40 years of electron probing in Madison

RGB X-ray maps (Ca, Si, S) of Volcanic ash from 1991 Mt. Pinatubo eruption: sulfate (pink) trapped by growing zoned feldspar (olive green with red zones), surrounded by bubble-rich volcanic glass (bright green). (Apatite=small bright red)



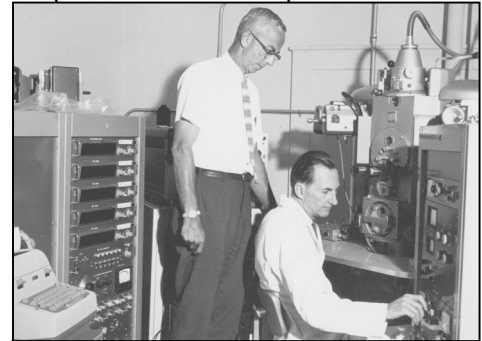
also a routine procedure. An attached EDS detector can be operated in to provide rapid phase identification.

Analytical error (precision) is a function of statistics, and that means count rates. Thus, higher precision is possible for major elements, and lesser for minor ones. Trace elements may be

measured, but longer counting times are generally required. A normal 8 element silicate mineral analysis takes a little over a minute, and analytical error varies, from 0.4 % for SiO<sub>2</sub> at 50 wt%, to 1.3% for Al<sub>2</sub>O<sub>3</sub> at 4.8 wt%, to 33% for MnO at 0.10 wt%.

Rates

Internal EPMA/SEM 9AM-9PM	\$35/hr
EPMA/SEM 9PM-9AM	\$25/hr
Overnight Mapping	\$15/hr
External SEM	\$250/hr
EPMA	\$300/hr



Gene Cameron (L) and Everett Glover (R) with EMX

Raymond Castaing had invented the electron probe at the University of Paris in 1951, and by 1956 the first commercial electron microprobe (Cameca) was available. Our Department's Gene Cameron had developed the science of reflected light microscopy to study ore minerals and knew of the additional power of the microprobe.

In 1966, with \$50K from WARF and \$50K from NSF, Gene acquired an Applied Research Laboratories (ARL) 'EMX' electron microprobe. Soon thereafter Everett Glover was employed to run the probe lab, which he did until retiring in 1992. The probe was at 917 University Avenue, and ran until

December 1980. Some of the first lunar samples from the 1968-71 Apollo missions were analyzed with this microprobe.

By 1978, the limitations of the first generation electron microprobes were clear, and Gene Cameron developed a new proposal with the assistance of Professor Charlie Guidotti for an automated ARL-SEM-Q (SEM Quantometer). It was purchased with \$167K from NSF, \$78K from the Graduate School and WARF, \$15K from Letters and Science, and \$43K from the new Weeks Hall building fund. It was installed in Weeks Hall in February 1981, and users paid \$5 per hour (by 1992 the charge was \$15, and it is \$35 today.) A new generation of microprobers cut their teeth on this 9 spectrometer instrument.

The SEM-Q was showing its age by 1990 (ARL had folded by 1983) and there was new interest in a state-of-the-art electron microprobe that could combine SEM features — high quality imaging (BSE, CL, x-ray maps) — with spot quantitative analyses, and measure light elements with a new generation of pseudocrystal diffractors.

John Valley took the lead in 1991 in developing a proposal to NSF for funding our third electron microprobe. A new Cameca SX51 was installed in September 1993, thanks to the financial contributions of NSF (\$320K), UW-Madison Graduate School (\$150K), UW-Madison College of Engineering (\$75K), UW-Madison College of Letters and Science (\$65K) and AMOCO (\$8K). Gene Cameron cut the ribbon at the official opening of the new probe lab in late 1993.

[www.geology.wisc.edu/~johnf/sx51.html](http://www.geology.wisc.edu/~johnf/sx51.html) (608) 262-7964