

## Weeks Hall SEM Lab



In 2006, we added to our microanalytical capabilities with the installation of an Hitachi S-3400 variable pressure scanning electron microscope (SEM), in Room 308.

In addition to normal secondary and backscattered electron detectors for imaging, this SEM has an electron backscattered diffraction (EBSD) detector, cathodoluminescence (CL) detector, and energy dispersive spectrometry (EDS) detector.

It is versatile in being in the new generation of “variable pressure” SEMs, meaning that samples do not necessarily have to be conductively (e.g. carbon or gold) coated to counter charging under the electron beam (that traditional high vacuum with coated samples is preferred where possible). Samples that would be compromised for elemental or isotopic analysis by a coating can now be examined and imaged easily.

## EBSD

Diffraction patterns obtained from minerals in a thin section are a precise record of crystallographic orientation. The patterns

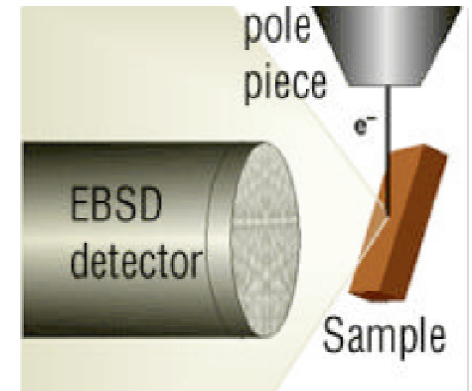
that arise from beam-specimen interactions may take the form of points or bands – Kikuchi lines. The interpretation of Kikuchi line patterns requires only an empirical data set of patterns for crystals of known structure and orientation (and known composition for minerals exhibiting solid solution). A few minerals, e.g. some layer silicates, do not typically produce good Kikuchi lines, but most major rock-forming phases do. There are a variety of reasons to use EBSD to evaluate the crystallographic orientations of mineral grains – lattice preferred orientations – in rocks of interest.

Another usage of EBSD is for identification of different phases that have the same chemistry (e.g. the polymorphs of  $\text{CaCO}_3$  or  $\text{SiO}_2$ ).

As shown in the above illustration, the sample is tilted at  $70^\circ$  to the electron beam, with the EBSD “camera” driven to a very short distance away. The electrons will be diffracted from the top 1–2 layers of atoms into the camera, producing the Kikuchi lines,

which the software then analyses for crystallographic information (atomic layer spacing and orientation), comparing to potential “matches” the user has set up in a library.

EBSD samples need a special final polish to remove all strain induced during grinding and polishing.



Kikuchi lines

## Variable Pressure SEM

Traditional SEMs have operated in high vacuum regimes ( $\sim 10^{-6}$  torr). Most geological materials are insulators, and thus require some conductive coating.

Variable pressure operates by allowing some air to remain in the chamber, which is ionized by the electron beam and lets any charge on the sample to bleed off.

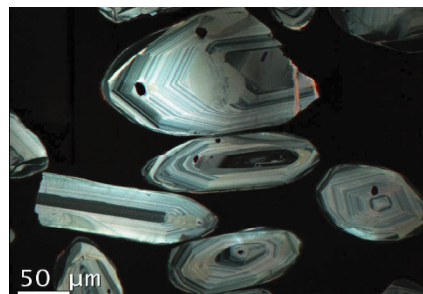
This is of great value in at least two cases:

- 1) EBSD, where coating the non-conductive thin section would prohibit Kikuchi lines from forming; and
- 2) diagnostic appraisal of specimens (e.g., forams) that will later be chemically analyzed, so that a conductive coating is essentially a contaminant. This also saves the time and effort of putting a coating on the samples.

Low vacuum capability also permits rapid SEM operation on materials that might otherwise outgas for a long time (clays, pumices, etc).

## Cathodoluminescence (CL)

Several key rock-forming minerals exhibit cathodoluminescence, which varies in intensity with variations in outer shell electron configuration, which in turn are a function of stress and/or trace element chemistry, providing an effective record of episodes of mineral growth, dissolution, cementation and/or overgrowth. This is very important when you are trying to unravel the history of a rock, and especially when you are using a sensitive



Zircon CL image constructed from 3 images acquired with PanaCL/F + 3

(and expensive) tool such as the ion probe to determine precise isotopic compositions of a population of minerals (e.g. zircons, quartz).

The CL system (Gatan PanaCL/F) has the ability to insert filters, and thus recreate a color CL image, or filter out particular wavelengths. This is useful for CL imaging of carbonates, where the orange-red wavelengths have a dwell causing a smearing of the SEM-CL image. The blue (bandpass) filter yields a useful CL image.

## EDS X-ray Microanalysis

Being geologists, we deal with a variety of minerals, and having EDS is essential, for rapid ID of minerals in a multiphase assemblage. Our EDS detector is a Si(Li) detector with a thin film window, allowing detection of elements down to Z=6 (C). The software has a variety of features: "Point and Shoot" where you collect an image then click on the point of interest, where a spectrum is collected; "Feature Analysis and Chemical Typing" where you have a large area of interest but only a small population of grains you are trying to find ("needle in a haystack"); you set up the imaging so your desired feature stands out in BSE (usually bright on a black background). The software picks out the discrete grains, then acquires EDS spectra (say 2 seconds each) on each. You come back the next day, sort the data and find the 10 or 30 K-feldspar grains that you want for Ar-Ar dating.

For training (usually a few minutes) and scheduling (and discussion of sample particularities), contact John Fournelle, [johnf@geology.wisc.edu](mailto:johnf@geology.wisc.edu) (608) 262-7964