

Using the Scanning Electron Microscope for Discovery Based Learning in Undergraduate Courses

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ABSTRACT

Laboratory exercises incorporating a Scanning Electron Microscope (SEM) encourage undergraduate students to explore geologic concepts and methods. A variable pressure (VP) SEM, with attached Energy Dispersive Spectrometer (EDS) and Electron Backscatter Diffractometer (EBSD), has been used in introductory, mineralogy, petrology and structural geology courses to examine sand morphology, quantify mineral chemistry, estimate the temperature of metamorphism, identify microfossils, and pursue student-designed research questions. Student response to using the SEM has been overwhelmingly positive. The undergraduate students, regardless of course level, tend to master the basics of operating the SEM quickly. After a half hour (or so), most students feel confident enough to take control of the SEM and use it to answer questions, or test ideas. Laboratory exercises are designed to allow students some freedom to pursue their own ideas and hypotheses within the framework of a broader geologic question or concept.

INTRODUCTION

Advances in electron-beam technology in the 1960s led to the development of the electron microprobe. This technology changed the way geologists investigate rocks by allowing researchers to image and obtain non-destructive analyses of micron sized samples. More recent advances have led to the development of variable-pressure scanning electron microscopes (SEM) that can be equipped with an energy-dispersive spectrometer (EDS) to analyze chemistry, or an electron backscatter diffraction (EBSD) system to investigate crystal lattice structures and orientations (please refer to Table 1 for list of common acronyms). Results of investigations using electron microprobes and SEMs currently form the foundation of many concepts presented to undergraduate students. Students, thus, benefit from being provided the opportunity to have hands-on experience in inquiry-based laboratory exercises using the technology upon which current geologic research is based.

Hands-on exercises with the SEM meet educational objectives to introduce undergraduate science and non-science majors to current technology, to promote problem solving and communication, and to allow students to discover. These objectives are in line with recent pedagogical studies and reports (c.f. Bonwell and Eison, 1991; Holliday et al., 1996; Ireton et al., 1996) that recommend that undergraduate courses create opportunities for students to "do" science by simulating or engaging in research activities. Specifically, the Shaping the Future report (NSF, 1996) requests that science education "focus on the processes of inquiry and discovery; and rekindle the unique curiosity, the sense of wonder, with which every child is born." When we invite students to investigate a specimen and collect data using the SEM, and we hear them say "look at this" or ask

questions, we have allowed them the freedom to be curious.

EQUIPMENT AND METHODS

Most of the exercises described may be accomplished using any SEM with an attached energy dispersive spectrometer (EDS). The EDS enables qualitative and quantitative chemical analyses and x-ray maps to be obtained. The EDS is used routinely to examine polished thin-sections of rocks (probe-quality polished thin section without a cover slip). The EDS also may be used to collect qualitative chemistry from other non-polished samples (e.g. mineral specimens, sand, ash, stromatolites...). The instrument used in the described exercises is a LEO1450VP equipped with an EDAX EDS that was purchased in 1999 with an NSF-CCLI grant and matching funds from Bowdoin College. The college pays for the yearly service contracts, and funds from external research grants are used to purchase consumables (liquid nitrogen, nitrogen gas, ink, adhesives, gloves...).

Recent SEM models that are controlled by personal computer offer a familiar interface to students, but are not essential. For our set-up, we move the stage with a joystick, and make beam, vacuum, and image adjustments through pulldown menus and mouse movements. Images and data are printed to network printers or to the attached dye sublimation printer, and stored on the hard-drive, on network drives, or written to CDs.

Variable pressure (*aka* low vacuum or environmental) SEMs offer additional flexibility, because non-conductive specimens (e.g. sand, diatoms, silicate minerals) may be examined uncoated under low-vacuum mode. If low-vacuum mode is not an option, or if higher resolution images are desired, then specimens should be coated with carbon or gold to reduce the charging that would otherwise occur under high-vacuum. A 20 angstrom carbon coat may be applied easily using a vacuum sputter coater using carbon rods or threads. The low atomic number of carbon does not interfere with chemical analyses. A gold coat may be preferred for the high magnification and high resolution secondary electron images (SEI) of specimens such as microfossils.

We prefer to mount thin-sections and small specimens on stubs that are attached to a multi-stub holder that is inserted into the SEM chamber. Thin sections typically are mounted with a double-adhesive tab on 32mm stubs; carbon tape from the top of a coated thin-section to the stub completes the conductive path. Two thin-sections on stubs fit on our multi-stub holder. We mount smaller specimens such as microfossils and mineral grains on 12mm stubs using double-adhesive carbon tape; up to 8 of these stubs fit on the holder. We choose to use the stub-and-tape method of sample mounting because it is simple and effective, and because multiple thin-sections or specimens may be prepared ahead of time.

The first time a student uses the SEM, an instructor introduces the main components of the instrument,

	Definition	Application/Use
BSE	Backscatter Electron (image)	Gray scale atomic contrast images
EBS	Electron Backscatter Diffraction	Crystal lattice patterns and orientation maps
EDS	Energy Dispersive Spectrometry	Chemical composition
SEI	Secondary Electron Image	Topographic image
SEM	Scanning Electron Microscope	
VP	Variable Pressure	Low-vacuum mode (to reduce charging)

Table 1. Common acronyms used in electron microscopy.

Introduction to Physical Geology	Investigation of mineral properties. Analyses of mineral and rock compositions.
Marine Environmental Geology	Imaging, identification and comparison of diatoms.
Environmental Geology and Hydrology	Characterization of beach sands. Imaging, identification and comparison of microfossils.
Earth and Life History	Identification of conodont specimens.
Mineralogy	Determination of mineral chemistry and crystal system.
Igneous and Metamorphic Petrology	Analyses of minerals to be used in pressure-temperature calculations.
Structural Geology	Investigation of crystallographic preferred orientation and other microstructures.

Table 2. Example exercises for geology courses. In addition to the listed exercises, many upper-level courses conclude with a small-group research project using the SEM.

briefly explains how the instrument works from the generation of electrons to the acquisition of images and EDS data, and demonstrates how to move the specimen and how to adjust focus, magnification, brightness and contrast. The introduction typically takes about ten minutes, depending on student questions. After the introduction, students take control of the SEM, review its operation, and begin collecting data. The instructor remains available to answer questions, but allows students to operate the instrument and collect data.

The class set up varies depending on the course and the objective. For introductory courses, we have found it helpful to load the specimen prior to the students arriving, because the pump-down of the SEM chamber to vacuum takes the most time and is the least interesting. Groups of two to six students work well in the lab: one student controls the joystick, one student controls the mouse, and they switch responsibilities during the lab. Groups larger than two seem to promote discussion better than smaller groups. However, groups larger than

six are not ideal, because not all students are actively involved. For larger introductory courses, groups of students are rotated through the SEM lab every 45 minutes to 1 ½ hours depending on the project. This might be done over more than one laboratory period, with students working on other projects when they are not doing the SEM part of the laboratory. For upper-level small-group or individual projects, students typically sign up for three-hour blocks during the week.

EXAMPLE EXERCISES USING THE SEM/EDS IN INTRODUCTORY COURSES

At Bowdoin College, the SEM has been used in multiple geology courses, including three introductory courses: Introduction to Physical Geology, Environmental Geology and Hydrology, and Marine Environmental Geology; and in several upper-level courses: Mineralogy, Igneous and Metamorphic Petrology, Earth and Life History, Sedimentary Geology, and independent studies. This multi-course use allows students to experience different applications of the SEM, and to gain confidence by building on what they have learned in previous courses. An opportunity also opens for students to teach each other techniques based on their experiences in the previous geology courses they have taken. Brief descriptions of exercises are listed in Table 2. Details of example exercises for introductory courses follow. Another source for a SEM/EDS mineralogy exercise is Cheney and Crowley (1997).

Mineral Properties - This exercise uses the SEI to examine properties of minerals, such as cleavage and habit, and uses EDS to determine the qualitative chemical composition of the mineral. For this exercise, small mineral specimens are mounted on multi-stub holder. Common minerals are selected that have a range of properties and compositions, for example: quartz, galena, asbestos-form serpentine, pyroxene, garnet, and feldspar. The first question posed in the exercise familiarizes students with the components of the SEM and with mineral properties: "Examine the topography of all the mineral specimens using the SEM. Then, select one mineral to photograph, and determine the composition of this mineral using the EDS." This question frequently leads to discussion of the perthitic texture of the feldspar that is apparent in the BSE image, and also to discussion of mineral end-member compositions during chemical analysis of the pyroxene and garnet samples. Students often seem eager to analyze the composition of additional specimens and are encouraged to do so as time allows. The second question invites students to explore other applications: "If you could choose any sample to analyze with the SEM/EDS, then what would you choose to analyze and why?" Some students follow through with their ideas in later laboratory sessions.

Beach Sand - Within a laboratory section, groups of four students pose questions that can be answered using the SEM and sand they collected from beach profiles. The assignment reads, "We collected sand samples as part of our Reid Beach profiles. Ask a question, or construct a hypothesis, that can be tested using the sand samples and the SEM/EDS. What are your observations and conclusions?" Questions posed by students include: "do mineral proportions change along the width of the

beach?" and "does grain morphology change along the profile?" To answer their questions, the students select a few sand samples from the fifteen beach profile samples they took, and mount them on carbon tape on a small stub. The sand is uncoated and viewed in VP mode. One group evaluating whether mineral portions changed along the width of the beach set up a sampling grid using the SEM "Annotation" tool and analyzed the composition of every grain at the grid intersections for each of their samples. They discovered that the sample at one end of the beach had higher concentrations of garnet than the other samples and attributed the source of the garnets to the metamorphic rock that was exposed at that end of the beach. In addition to answering their questions and testing their hypotheses, the exercise introduces students to mineral compositions and potential sampling biases.

Microfossils - Students collect glaciomarine muds of the local Presumpscot Formation from coastal exposures near campus. Dried and sieved mud samples are mounted on carbon tape and either viewed uncoated in VP mode. Students identify the microfossils using the SEM and explore microfossil variations relative to sample locations and stratigraphic depth. They present their findings through a series of labeled SEI images of the microfossils.

Igneous Compositions - Students work with carbon-coated thin sections from an igneous dike that they examined during the previous week's field lab. The first question posed to students is to determine the average chemical composition of the rock using the SEM/EDS. This type of analysis usually is done using an ICP-MS or XRF during petrologic studies, but the accessibility of the SEM/EDS and the immediacy of the result make this approach attractive for an introductory course. The analysis is done in a beam-scan mode, rather than the typical spot mode used to examine mineral composition, and yields a quantitative analysis in weight-percent oxides. Using the chemical composition, students are asked to identify the magma source as felsic, mafic, or ultramafic and to evaluate whether the composition is compatible with the igneous rock type that they identified in the field. As time permits, students may find, analyze the compositions of, and identify several minerals in the sample using qualitative EDS analyses (spot mode). Discussions with students in class and during office hours revealed that an unanticipated outcome of this laboratory exercise was that students left with a clearer understanding of Bowen's reaction series, and what minerals are found in what rock types and why.

Research Project - The Introduction to Physical Geology course and several of the upper-level geology courses include a research project component. The first step in using the SEM in these projects is to identify the minerals in the sample using BSE images and EDS analyses. The next steps depend on the interests of the students. One introductory class project involved field and laboratory work on a series of granitic intrusions: one group of students discovered that tourmaline had replaced biotite over the scale of a thin section in a schist that was adjacent to a granite dike, and another group became interested in the zoning patterns of plagioclase

and completed a series x-ray maps. In mineralogy classes, students have elected to identify asbestos fibers in samples of commercial vermiculite and examine the formation of sector garnets. Some students need guidance in selecting a project, but most choose projects that follow through with something that they have observed in the field or while using the petrographic microscope or SEM.

ELECTRON BACKSCATTER DIFFRACTION

An Electron Backscatter Diffraction (EBSD) system may be attached to a scanning electron microscope (SEM) to allow in-situ determinations of crystal lattice structures and orientations. I address EBSD applications to undergraduate education in this separate section because only a few - although an increasing number of - institutions currently have this technology. Over the past ten years, geologists have begun publishing research applying EBSD techniques to problems such as the effects of microstructures on intragrain argon isotope ages (Reddy et al., 1999), the development of garnet porphyroblasts by multiple nucleation (Speiss et al., 2001) and plastic deformation of metamorphic pyrite (Boyle et al., 1998). The capabilities of EBSD systems allow researchers to test existing nucleation, growth, and deformation models and to develop new ones; and, these same capabilities introduce new possibilities for teaching crystallography in mineralogy courses and micro-scale deformation in structural geology courses.

The EBSD system uses backscattered electrons (BSE) emitted from a specimen to form a diffraction pattern that is imaged on a phosphor screen. The system may be used to identify the crystal system of a mineral, to measure crystallographic preferred orientation (CPO) measurements of minerals, to map orientation of grains in a specimen, and to plot the crystallographic orientations on a stereonet. Further details about EBSD methods and applications can be found in Schwartz et al. (2000) and Prior et al. (1999), and on the following websites: <http://www.tsl-oim.com> and <http://www.hkltechnology.com>.

I have used an SEM/EBSD set-up in undergraduate mineralogy and structural geology courses. For the mineralogy course, I selected six small (mm-scale) mineral specimens (quartz, pyrite, plagioclase, muscovite, amphibole, and garnet) and placed them on a multi-stub holder. Students used SEM images to identify habit, cleavage, and fracture on the specimens, then obtained EBSD patterns to determine to what crystal system the mineral belonged. They then combined these data to identify the minerals. This lab followed labs introducing mineral properties and crystallography at the beginning of the course, and served to reinforce these concepts and to emphasize the lattice structure of mineral crystals. Later in the course, students completed mini-projects for which they could opt to use the SEM/EBSD and/or SEM/EDS.

For the structural geology course, we used four samples from a local granite pluton that had been cut by a fault. The samples, collected from a transect across the fault zone, showed variations in strain. Polished thin sections of the samples were further polished using colloidal silica on a vibratory polisher to remove any defects introduced during the mechanical polishing. Using the SEM/EBSD, students collected orientation maps to observe and interpret variations in CPOs for the

four samples. This multi-week lab followed an introduction to microstructures using petrographic microscopes.

EVALUATION AND DISCUSSION

Three objectives for this project were 1) to acquire an analytical instrument that could be readily used by novices, 2) to introduce science and non-science majors to current SEM technology, and 3) to engage undergraduates in scientific discovery.

1) We find that undergraduate science and non-science users readily learn to use the SEM. Observations of students in the SEM lab show that the PC-based observation of the SEM makes it easy for students to use, allowing them to focus their attentions on the science - not only on the techniques. After three years of having introductory geology students in the SEM lab, we have learned that most students are competent (and confident!) enough to take complete control of the SEM, without instructor direction, in half hour or less. Having students work in groups of four is helpful for increased confidence and because they can remind each other how to operate the instrument. Acquiring and interpreting EDS data tends to require more instructor guidance. A "cheat sheet" of common mineral formulas helps introductory students interpret the chemical spectra they obtain.

2) Through the project, science and non-science majors have been introduced to current analytical technology. During the first three years of the project, 278 undergraduates used the SEM in their courses (Bowdoin College has a student population of 1600). Of these students, nearly fifty percent (134 students) used the SEM while taking an introductory geology course. Comments on evaluation forms show that students taking introductory courses respond favorably to the perceived newness of the SEM technology; for example, students answered that the best aspect was "Becoming familiar with high tech scientific equipment" and "Using the new technology." In addition to course use, thirteen undergraduates pursued independent studies using the SEM and ten of these students presented their results at regional geology meetings during the first three years of the project.

3) Comments on student evaluation forms and our observations of students using the SEM show that the undergraduates are enthusiastically engaged. A recent evaluation of a research project in an introductory course asked students "What was the best aspect of the research project?": nine of sixteen responses mentioned the SEM (other responses cited the field component, doing real research, and working in a group). We find that our most valuable feedback comes from student remarks when using the SEM. These remarks show that they are actively engaged and learning geology: the students make observations, ask questions, draw conclusions, discuss geology with each other, and take projects in their own directions. The remarks also show that some students become frustrated when there are operational problems with the SEM, or when they do not understand the goal of the lab or research project. We note that in designing labs using the SEM, there needs to be a balance between providing enough structure to focus the lab, and allowing sufficient freedom for students to explore and make their own discoveries.

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