A Web Resource for the Study of Alkali Feldspars and Perthitic Textures Using Light Microscopy, Scanning Electron Microscopy and Energy Dispersive X-ray Spectroscopy

Anne Argast — Deptment of Geosciences, Indiana University-Purdue University Fort Wayne, 2101 E. Coliseum Blvd., Fort Wayne, IN 46805-1499, Argast@ipfw.edu

Clarence F. Tennis III — Deptment of Geosciences, Indiana University-Purdue University Fort Wayne, 2101 E. Coliseum Blvd., Fort Wayne, IN 46805-1499, Tennisc@ipfw.edu

ABSTRACT

A Web site for laboratory activities focused on alkali feldspars and the development of perthitic textures is available at http://www.geosci.ipfw.edu/sem/semedx.html. Backscatter and secondary electron images, plane- and cross-polarized light images, energy dispersive X-ray data (including spectra and results files with data expressed as weight and atomic percents) and X-ray diffraction data are available. Care has been taken to match the areas examined by light and electron methods, allowing students to directly compare the data available from each technique. The information is targeted at undergraduate-level laboratories in mineralogy and petrology.

INTRODUCTION

Research in mineralogy and petrology often relies on data from electron microscopy and microchemical methods. It is important that undergraduates gain a rudimentary understanding of these fundamental techniques if they are to read the literature and critically evaluate the information it contains. Unfortunately, the equipment is expensive and often beyond the resources of smaller, undergraduate-oriented departments. Even when available, electron microscopes do not easily lend themselves to use by the large number of students that would normally populate an undergraduate laboratory in mineralogy or petrology. As a consequence, undergraduates rarely gain meaningful experience with electron microscopy and related methods.

A Web resource at http://www.geosci.ipfw.edu/sem/semedx.html facilitates undergraduate engagement with electron microscopy, energy dispersive X-ray spectroscopy, light microscopy and X-ray diffraction analysis. The site is suitable as an image source for lectures, or as a data source for laboratory exercises. Available information includes backscatter and secondary electron images, plane- and cross-polarized light images, energy dispersive X-ray (EDX) data and X-ray diffraction (XRD) data. The EDX data are presented as the raw spectra, as results files for each of 44 analyzed points (with data expressed as weight and atomic percents) and as a summary file in an Excel database. All images and data are from the identical areas of two analyzed samples, an anorthoclase phenocryst and a perthite, helping students make direct comparisons regarding the information available by light, electron and X-ray methods.

SAMPLES

The analyzed materials are a perthite from Perth, Ontario and an anorthoclase from Mt. Erebus, Antarctica. Both samples were purchased as stock items from Wards Scientific (http://wardsci.com/).

Anorthoclase is an alkali feldspar of composition $Or_{40}Ab_{60}$ to $Or_{10}Ab_{90}$ that forms part of the high-temperature solid solution between sodium-rich and potassium-rich feldspar end members. Upon cooling, the solid solution becomes incomplete with anorthoclase and the sodium-rich sanidines exsolving into two separate Na- and K-rich feldspar phases (albite and either orthoclase or microcline) resulting in the development of perthitic (or antiperthitic) textures.

The particular anorthoclase used here occurs as phenocrysts in phonolite lavas recovered on the slopes of Mt. Erebus. The phenocrysts are zoned and contain inclusions of ulvöspinel, clinopyroxene, apatite, olivine and glass. There have been several published descriptions of the Mt. Erebus anorthoclase phenocrysts (e.g., Kyle, 1977; Mason et al., 1982; Kyle et al., 1992; Caldwell and Kyle, 1994; Dunbar et al., 1994).

The sample of perthite is a characteristically twinned microcline feldspar displaying large exsolution lamellae easily visible in the hand sample and in the petrographic microscope. Standard textbooks routinely provide information on the development of perthitic texture. See Yund (1983) for additional discussion.

METHODS

A scanning electron microscope uses electrons from a filament (typically a heated tungsten wire) accelerated down a column through a voltage potential. The electron beam is rastered over the surface of a sample. Electrons from the sample surface (either secondary electrons or backscattered incident electrons) are detected and an image is rendered.

The accelerated electrons forming the beam may have sufficient energy to displace inner shell electrons in the target material. This leads to the production of characteristic X-rays that can be used to produce a quantitative chemical analysis. In energy dispersive x-ray spectroscopy, the energy (not to be confused with the intensity) of each individual X-ray arrival is determined and counted toward the development of an energy distribution histogram. With sufficient counts, this histogram becomes an EDX spectrum consisting of background and characteristic X-ray peaks which can be processed to yield a quantitative analysis of an area as small as about 4 μm^2 . Reed (1996) provides an excellent review of electron microscopy, energy dispersive spectroscopy and related analytic methods.

Electron images were gathered with an ISI DS130 SEM operating at 18 to 28 kV. Microchemical data were gathered with a Kevex Sigma EDX spectrometer running Quasar v. 4.9 software, standardless methods and oxygen determined by stoichiometry. X-ray diffraction data were gathered with a Philips APD 3520 diffractometer, 40 kV, 30 mA, 200 steps/degree and 0.5 sec/step. Additional information about these methods is available from the web site.

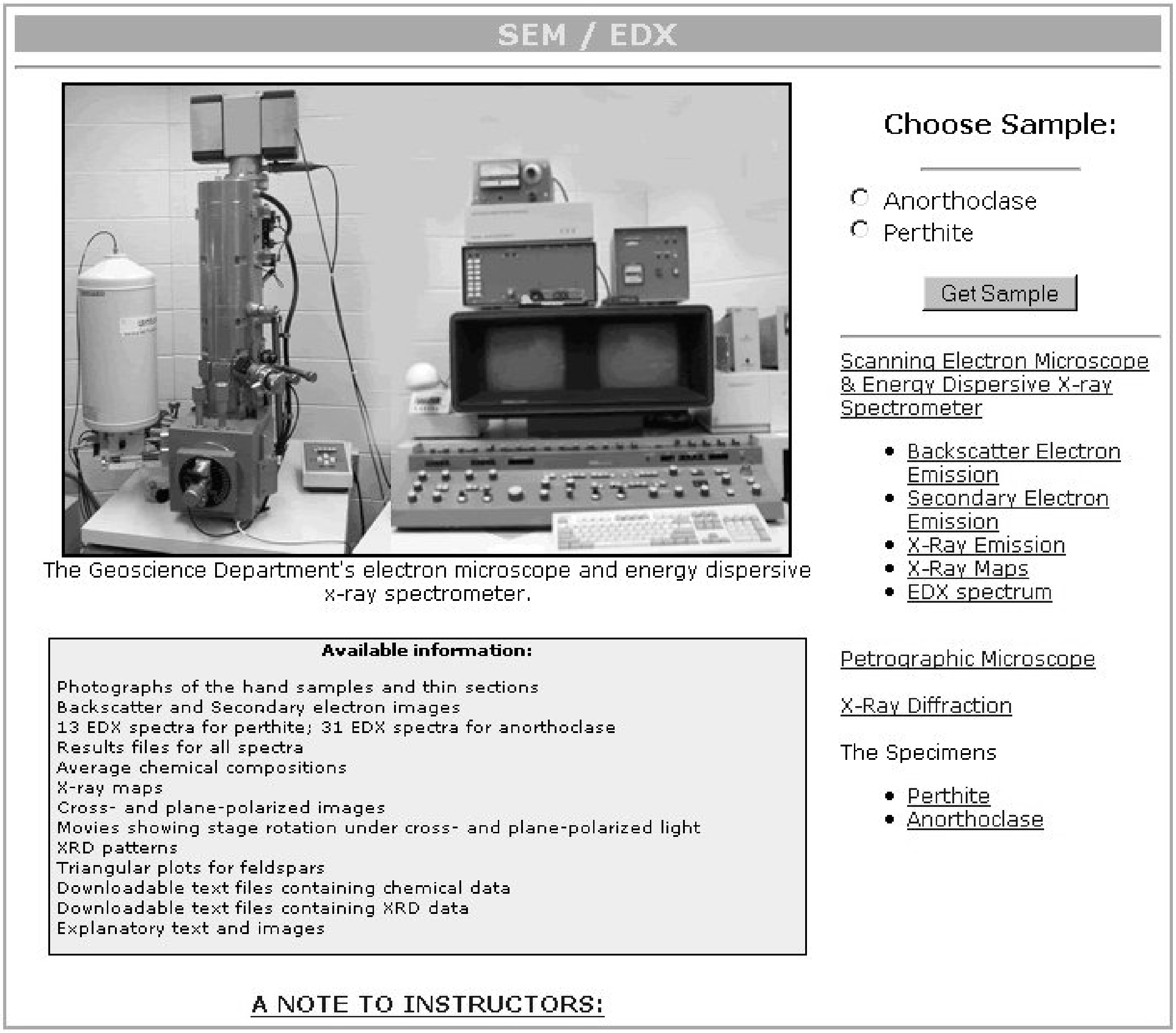

Figure 1. The main page on the Web site provides a summary of available data, links to brief explanatory information and gives the user the choice of an anorthoclase or a perthite sample for further examination.

USING THE WEB SITE

The Web site can be directly accessed at http://www.geosci.ipfw.edu/sem/semedx.html, or from a link at http://www.geosci.ipfw.edu. Upon entering the main page the user is given a list of information available on the site, a set of links to explanatory descriptions of the equipment and methods, and two radio buttons that branch to specific data for the anorthoclase and perthite samples (Figure 1). The Web site is not intended as a replacement for information otherwise available in text books and other generally available reference materials. Consequently the explanatory information is brief and fundamental. Also, the website does not provide prepared laboratory exercises, although our suggestions are described below.

Using the "Get Sample" button (Figure 1) leads to a page containing six index frames along the left border (Figure 2). Selecting an index frame reproduces the image in the larger window on the right. Plane- and cross-polarized light images, backscatter and secondary electron images, a composite X-ray map, and an electron image showing points with available EDX information are displayed.

Along the top border (Figure 2), links bring forward additional information available on the specimen. This includes a set of movies (in animated .gif and .avi formats) that show the effects of rotation on the cross-polarized image, a photograph of the hand sample and the thin section, an atomic K-Na-Ca plot for all feldspar analyses, an X-ray diffraction pattern of the bulk sample, a downloadable Excel file that contains the raw and smoothed X-ray diffraction data that can be used with standard plotting software, and downloadable text files in Excel and tab-delimited formats containing all chemical data for the specimen. Tips for importing and plotting these data are contained in the headers of the text files.

Individual points on the 'EDX Analysis Points' image become hot links when displayed in the main window on the right-side of the page (Figure 2). Clicking on any point produces a labeled EDX spectrum obtained from that point on the specimen and the results file produced by the Kevex Sigma software (Figure 3).

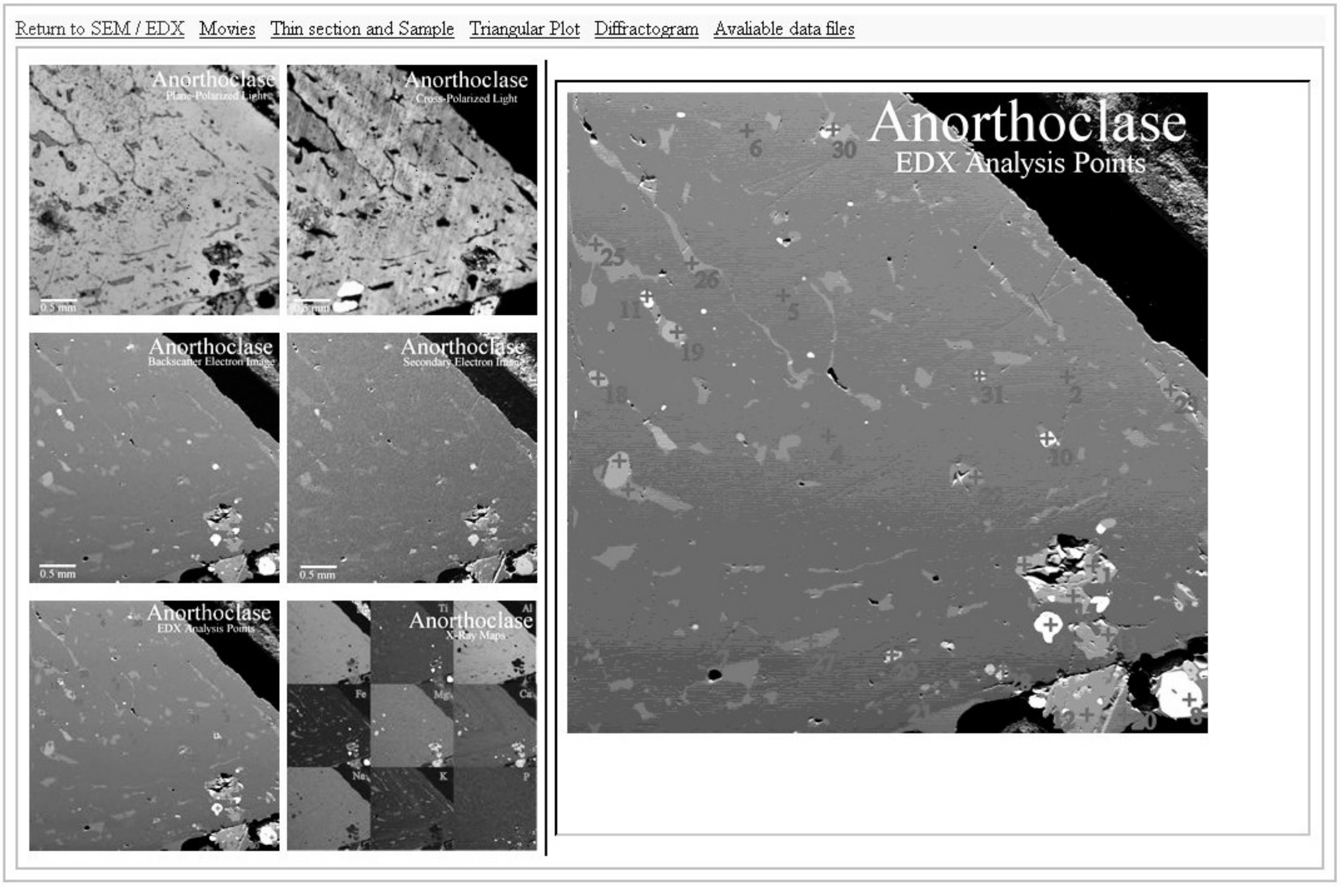

Figure 2. After choosing a sample, the user can see light, electron and X-ray images of the specimen, any one of which can be displayed at a larger size in the window at the right side of the monitor; in this case an image showing the analyzed points on the anorthoclase specimen. Links along the top border lead to other information available on the sample. The actual Web page is in color and of larger size.

TECHNICAL ISSUES

Every effort has been made to compress files without an excessive loss of quality. However, users will find value in a broad-band connection when using this site.

Layouts work best at screen resolutions of 1024 x 768 and higher. With lower resolutions the need to use scroll bars will prove a frustrating limitation.

The site has been tested using Netscape, Mozilla, Opera, Safari and Internet Explorer. Different browsers produce some differences in functionality. This will be most apparent when viewing movies in the .avi format. Web-browsers running under the Macintosh OS produce a seamless window with an attached bar that allows the user to pause or step through the stage rotation. Windows-based machines spawn a separate program such as Windows Media Player which must be previously loaded on the user's computer. The animated .gif movies contain the same information as the .avi-formatted movies and will run without additional software from all browsers and all platforms. However, the animated .gif format does not allow the user to pause the presentation.

POSSIBLE ACTIVITIES

Different instructors will find different ways to use the available information. The following examples briefly illustrate some possibilities that are suitable for a sophomore-level course in mineralogy or petrology.

Activity 1 - Count the number of different phases in the perthite sample using a combination of light and electron images and the X-ray map. Assign each of the 13 chemical analyses to one or the other phase. Give a name to each phase and recast the formulas.

Goal - The perthite sample is simple, containing albite (analytic points 1-6) and microcline (7-13). Students will gain familiarity with electron and light microscope images, learn to recognize differences in appearance, and gain practice in using chemical data to calculate chemical formulae.

Activity 2 - Repeat activity 1 for the anorthoclase.

Goal - The anorthoclase is considerably more complex. In addition to feldspar (analytic points 1-7), the anorthoclase phenocryst contains ulvöspinel (8-11), clinopyroxene (12-19), glass 20-27, apatite (28-29) and olivine (30-31). This activity provides more practice in recognition and chemical recasting. The presence of glass reinforces the idea that 'phase' and 'mineral' are not identical concepts. Recognizing the glass will be especially challenging, and students may need to be helped a bit by having them make close comparisons between the plane and cross-polarized images with appropriate prompts to consider the effect of isotropy on birefringence.

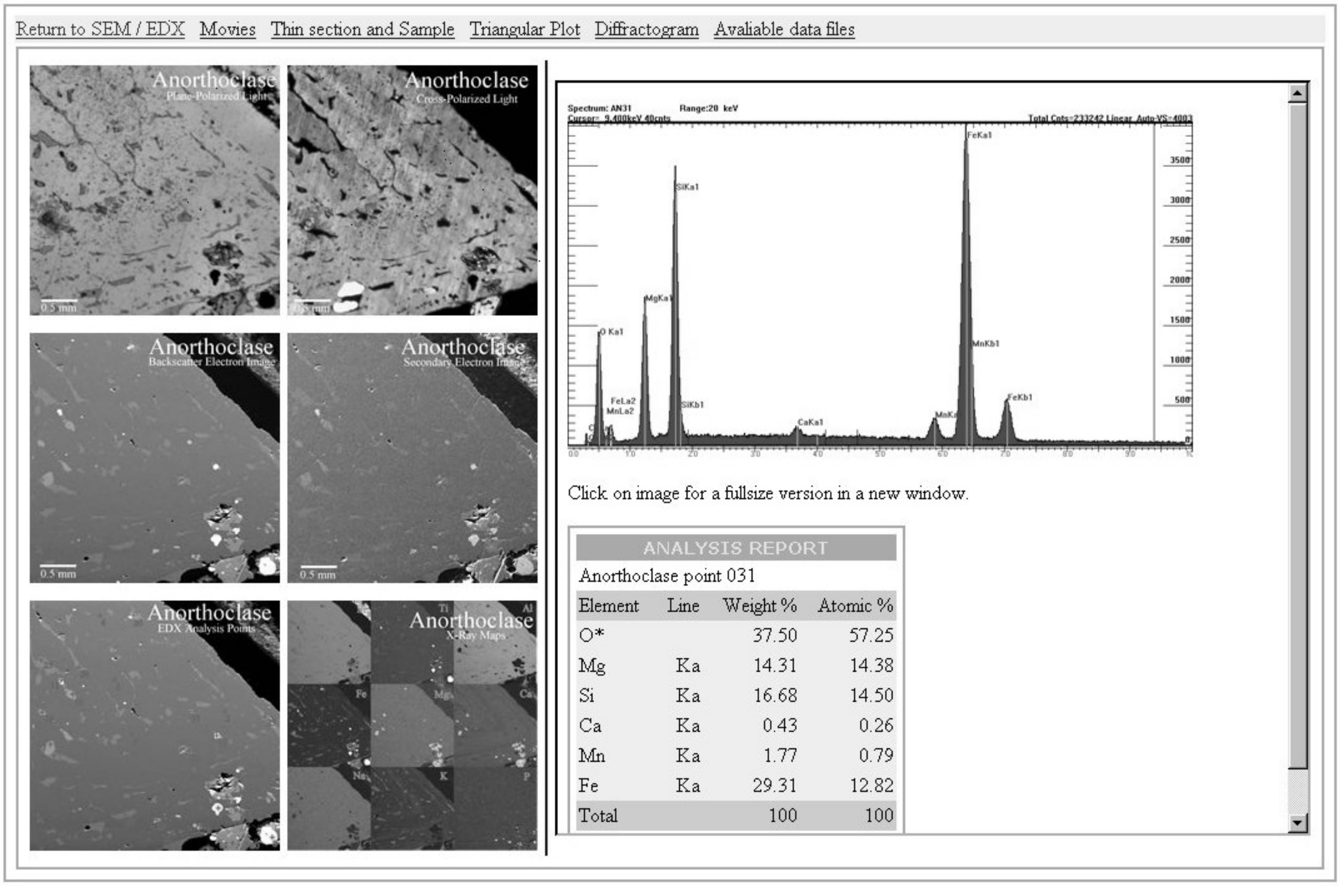

Figure 3. Choosing a point (here point 31) places the EDX spectrum and an analysis report in the right-hand window. This image can be expanded for easier examination.

Activity 3 - Plot the 13 perthite analyses on a properly labeled K-Na-Ca ternary diagram. Add the point for the average perthite composition. Use a second piece of graph paper and do the same for the subset of feldspar analyses from the anorthoclase phenocryst, again plotting the average analysis. Have the students develop an appropriate metric (e.g., standard deviation, histogram or even measured distances on the plot, depending on class experience) to describe the variation around the sample average and give a reason to explain the different distributions.

Goal - Students gain practice in plotting data on a ternary diagram. The different chemical distributions (bipolar with a central average far from any single analysis in the case of the perthite, a single cluster plotting near the average in the case of the anorthoclase) provide graphic evidence for the chemical demixing that characterizes perthite development.

Activity 4 - Have students determine the arithmetic average of the 13 perthite analyses and compare that result to an area-weighted average. Areas can be determined by printing the backscatter image, cutting out dark and light areas, and weighing on a balance. Alternately, areas can be determined using appropriate image processing software such as ImageJ, public domain software from the National Institutes of Health (free download from http://rsb.info.nih.gov/ij/download.html).

Goal - The 13 points in the perthite analysis were not randomly distributed and a simple arithmetic average is not a fair measure of the average composition. Students learn that automatic and unthinking application of statistics can produce misleading results.

Activity 5 - There are eight glass analyses scattered in different parts of the anorthoclase phenocryst. Have the students determine a relative age sequence based on position relative to the core and rim. Compare position/age to glass composition. Have the students explain the significance of glass in the anorthoclase and exsolution textures in the perthite as they relate to determination of sample cooling rates.

Goal - Students will gain experience with zoned crystals and become more familiar with the idea that crystals grow from a central core and with the role kinetics plays in the development of a final phase assemblage. There is some disagreement in the literature about the relative composition of glass from cores and rims in the Mt. Erebus phenocrysts. Kyle (1977) reports an increase in Al_2O_3 and a decrease in total FeO from core to rim. Mason and others (1982) report that glass from rims and cores are identical. The anorthoclase crystal discussed here is consistent with the results reported by Mason. Advanced students can be asked to explain the significance of this result as it relates to magma evolution on Mt. Erebus.

Activity 6 - Once the students have identified all phases and formulae in the anorthoclase (activity 2) have them estimate the brightness of each phase in the backscatter image. This can be done qualitatively by estimating the brightness on an arbitrary scale, or quantitatively by determining the average gray scale value using imaging software such as ImageJ, described above. Plot brightness against average atomic number of each phase.

Goal - Backscattering efficiency is a strong function of atomic number. Students will learn that electron images can be used to estimate the chemical composition of a target material.

Activity 7 - Have the students identify an analysis with high iron content (e.g., olivine sample 31 in the anorthoclase phenocryst), identify the FeKα, FeKβ and FeLα peaks on the spectrum, determine the energy of each peak, and describe the electron transition responsible for the production of each peak.

Goal - Students will see a practical application to the theory they have probably already learned related to electron shells, quantum designation and energy-level diagrams.

DISCUSSION

There is no doubt that technology, computers and the World Wide Web have had a significant positive impact on teaching in the geosciences (Durbin, 2002). Over the past decade, there have been numerous descriptions of Web-related resources for use in the geology classroom. For example, Argast and Corey (1998) describe a Web-accessible diffractometer and Hluchy (1999) establishes the pedagogical value of teaching diffraction methods in an undergraduate course. There are Web-based resources to support teaching across the broad spectrum of the earth sciences including planetary geology, economic geology, meteorolgy and in many other areas (Exton, 1998a, 1998b, 1999; Francek, 2002). This paper and the associated Web site adds to this existing inventory by describing a new resource for use in undergraduate mineralogy and petrology laboratories and classes. This paper also describes a few possible activities based on this resource. Some additional comments:

It is easy to assume that methods such as electron microscopy are not accessible to undergraduate students because the technology is too complex, or the laboratory spaces are too small for undergraduate classes. In reality, meaningful exposure to these methods is possible, and exposing undergraduates to specialized equipment reaps rewards in the form of broader experience, increased comprehension, interest and classroom excitement.

Many undergraduate programs do not have routine access to electron microscopes, X-ray diffractometers and similar devices. The World Wide Web is a viable medium for spreading accessibility across a broad spectrum of institutions and users. This requires a commitment of time and effort by those fortunate enough to have local access to specialized equipment. Whether through the Web, by opening facilities to guest users or in other ways, it is hoped that those who have will find ways to share with those who don't.

In this paper we present coherent data detailing compositional and textural qualities of exsolved and unexsolved alkali feldspars. All the data are interrelated and overlapping. Students can move smoothly from plane-polarized images, to cross-polarized images to electron microscope images to X-ray maps and EDX spectra. As a consequence, a student can bring multiple skills and modes of understanding to bear on a single problem. For example, a student who has trouble understanding the significance of a light microscope image may understand better if she sees an X-ray map showing compositions across the same area. Presenting information in this way can take a bit more work, but can be rewarded with faster and more complete comprehension by your students.

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REFERENCES

Argast, S., and Corey, T., 1998, Using the World Wide Web for interactive control of an X-ray diffractometer, Computers & Geosciences, v. 24, p. 633-640.

Caldwell, D. A., and Kyle, P. R., 1994, Mineralogy and geochemistry of ejecta erupted from Mount Erebus, Antarctica, between 1972 and 1986, Volcanological and Environmental Studies of Mount Erebus, Antarctica, Antarctic Research Series, v. 66, p. 147-162.

Dunbar, N. W., Cashman K. V., and Dupré, R., 1994, Crystallization processes of anorthoclase phenocrysts in the Mount Erebus magmatic system: evidence from crystal composition, crystal size distributions, and volatile contents of melt inclusions, Volcanological and Environmental Studies of Mount Erebus, Antarctica, Antarctic Research Series, v. 66, p. 129-146.

Durbin, J. M., 2002, The benefits of combining computer technology and traditional teaching methods in large enrollment geoscience classes, Journal of Geoscience Education, v. 50, p. 56-63.

Exton, B. J., 1998a, Exploring geology on the World Wide Web - planetary geology, asteroids, comets and meteorites, Journal of Geoscience Education, v. 46, p. 285-287.

Exton, B. J., 1998b, Exploring geology on the World Wide Web - economic geology, Journal of Geoscience Education, v. 46, p. 398-401.

Exton, B. J., 1999, Exploring geology on the World Wide Web - meteorology, Journal of Geoscience Education, v. 47, p. 278-279.

Francek, M., 2002, Web resources: Weather on the Web, Journal of Geoscience Education, v. 50, p. 469-470.

Hluchy, M. M., 1999, The value of teaching X-ray techniques and clay mineralogy to undergraduates, Journal of Geoscience Education, v. 47, p. 236-240.

Kyle, P. R., 1977, Mineralogy and glass chemistry of recent volcanic ejecta from Mt. Erebus, Ross Island, Antarctica, N.Z. Journal of Geology and Geophysics, v. 20, p. 1123-1146.

Kyle, P. R., Moore, J. A., and Thirlwall, M. F., 1992, Petrologic evolution of anorthoclase phonolite lavas at Mount Erebus, Ross Island, Antarctica, Journal of Petrology, v. 33, p. 849-875.

Mason, R. A., Smith, J. V., Dawson, J. B., and Treves, S. B., 1982, A reconnaissance of trace elements in anorthoclase megacrysts, Mineralogical Magazine, v. 46, p. 7-11.

Reed, S. J. B., 1996, Electron microprobe analysis and scanning electron microscopy in geology, Cambridge, Cambridge University Press, 201 p.

Yund, R. A., 1983, Microstructure, kinetics and mechanisms of alkali feldspar exsolution, in Ribbe, P. H. (editor), Feldspar Mineralogy, second edition, Reviews in Mineralogy, v. 2, Washington, D.C., Mineralogical Society of America, p. 177-202.