11-18-2012

MRI: Acquisition of an SEM-EDS-EBSD-CL Microanalytical System for Solid Earth and Climate Change Research

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Final Report for Period: 09/2011 - 08/2012 **Submitted on:** 11/18/2012
Principal Investigator: Gerbi, Christopher C. **Award ID:** 0820946
Organization: University of Maine
Submitted By:
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Title:
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Project Participants

Senior Personnel

Name: Gerbi, Christopher
Worked for more than 160 Hours: Yes
Contribution to Project:
Instrument proposal development, evaluation; instrument installation and protocol development.

Name: Belknap, Daniel
Worked for more than 160 Hours: No
Contribution to Project:

Name: Grew, Edward
Worked for more than 160 Hours: No
Contribution to Project:

Name: Johnson, Scott
Worked for more than 160 Hours: No
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Name: Koons, Peter
Worked for more than 160 Hours: No
Contribution to Project:

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Worked for more than 160 Hours: Yes
Contribution to Project:
Instrument proposal development, evaluation; instrument installation and protocol development.

Name: Kreutz, Karl
Worked for more than 160 Hours: No
Contribution to Project:

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Contribution to Project:

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Name: Petrie, Meredith
Worked for more than 160 Hours: Yes

Contribution to Project:
Assisted in planning for acquisition of instrumentation.

Name: Alvarado, Cheryl
Worked for more than 160 Hours: Yes
Contribution to Project:
Assisted in technique development.

Name: Shulman, Deborah
Worked for more than 160 Hours: Yes
Contribution to Project:
Managed laboratory and assisted in technique development.

Undergraduate Student

Technician, Programmer

Other Participant

Research Experience for Undergraduates

Organizational Partners

Other Collaborators or Contacts

The electron microscopy lab, by its nature, involves many researchers developing and executing protocols appropriate for their work. Our two principal collaborators outside Earth Sciences are Jon Spender of Chemical Engineering, and Brian Robinson of Anthropology.

Activities and Findings

Research and Education Activities:

Students, educators, and researchers at or visiting the University of Maine have used the instrumentation acquired under this grant in a variety of ways, as described below. Our goals continue to include making the instrumentation as accessible as possible and accommodating many material types. The instrumentation includes a Tescan Vega II XMU scanning electron microscope with low-vacuum capabilities, EDAX/TSL integrated Digiview electron backscatter diffraction and Apollo energy dispersive spectrometry systems, Gatan ChromaCL cathodoluminescence detector, and a Tescan panchromatic cathodoluminescence detector.

Our major research activities fall into the following categories:

[BSE = backscattered electron; SE = secondary electron; CL = cathodoluminescence; EBSD = electron backscatter diffraction; EDS = energy dispersive spectrometry]

? BSE imaging of minerals in rocks to determine identity and microstructure
? BSE imaging of bones from archaeological sites to determine internal structure
? SE imaging for examining quality and structure of lignin nanofibers
? CL imaging of zircon related to ion microprobe dating
? CL imaging of quartz to investigate overgrowths/fractures/structures in metamorphic rocks
? EBSD mapping of rocks to use for modeling seismic anisotropy in the crust
? EBSD analysis to determine deformation mechanisms in amphibolite and granulite facies rocks
? EBSD analysis to determine the degree of metamorphic porphyroblast rotation
? EBSD mapping for mineral grain size analyses
? EDS identification of minerals in rocks

? EDS identification of beach sands to determine provenance
? EDS characterization of atmospheric dust and tephra recovered from snow and ice cores

In addition, we have performed one-time or exploratory work on projects such as identifying particle mineralogy in marine sediment and composition of a colonial bullet mold.

Our education activities include:
Training undergraduate and graduate students in analytical methods. As indicated in our original proposal, graduate students have largely taken on the role of maintaining the laboratory, assisting new users, and developing operational protocols (such as sample preparation, analysis parameters, and post-processing algorithms). Senior Personnel Yates developed a graduate-level course in analytical methods to provide more formal theoretical training for students. To date, thirteen graduate students and two undergraduates have used the SEM and related instrumentation as part of their thesis work. Five additional undergraduates and high school students have worked in the lab acquiring and processing data.
Inclusion of instrumentation in classes. PI Gerbi has used the instrumentation in Mineralogy for student projects, gathering data for laboratory exercises, and demonstrating how minerals vary in composition and structure. Yates has used the instrumentation in Ore Deposits for phase identification.
Most visitors, including administrators and prospective students and their parents, who tour the School of Earth and Climate Sciences view our instrumentation capabilities in an effort to improve knowledge in the general public about how scientists gather and use microscale images and data. Most years, several middle and high school classes visit specifically to learn about instrumentation in the Earth and Climate Sciences community. During those visits, students operate the electron microscope and analyze various materials.

We explored using large-scale SEM-derived visualization and remote operation of the instrument in classes. Although those remain possible pathways for the future, we did not find them a natural fit at present.

Findings:

Over the past two years, many of the projects initiated at the time of instrument installation have produced reportable results. Naturally, we have begun many new projects that are ongoing, but we can report here the following results supported by the instrumentation. We describe these at the project level. Discussion of the significance and transferability of these results can be found in following sections.

Vorticity gauges in sheared rocks rely on certain assumptions about the internal mechanics of the system. Application of the rigid-clast rotation gauge requires knowing the coupling between the clast and the matrix. Johnson et al. (2009a,b) described how lubrication at the clast boundary by mica or other factors has a strong influence on the stable position of clasts and that mica is likely self-lubricating. This is quantitively evident when vorticity in a single rock is measured using different techniques. These findings indicate that clast-based vorticity measurements may be underestimates.

Building off that study, 2012 M.S. graduate Ben Frieman (Frieman et al., in review) documented the 3D rotational behavior of porphyroblasts in a micaceous schist. He used the observed phase distributions as the basis for numerical models investigating clast rotation as a function of the proximity of mica concentrations to the porphyroblasts. His results support and complement those of Johnson et al. (2009a,b), indicating that porphyroblast orientation is an imperfect tool for relating strain to an external reference frame.

Ph.D. graduate (2012) Nancy Price investigated several aspects of rocks that lay at the base of a Paleozoic strike slip fault that is considered to be the best ancient analog for the modern San Andreas Fault. First (Price et al., 2012), she exhaustively documented the transition from pseudotachylite to an ultramylonite texture due to grain coarsening. This allowed her to predict a much higher degree of pseudotachylite than previously recognized in these and other rocks, indicating a mechanism for maintaining the localization of slip at this crustal level. Subsequently, she documented the inheritance of quartz crystallographic orientations, even at high strain, indicating the need for caution in interpreting crystallographic data in mylonitic rocks. Quartz microstructures also seem to preserve evidence for transient stress loading, a feature we would expect in seismically active regions. Finally, she proposed parallels between the across-strike fault structure at the surface and that at depth. The divisions at depth, including a damage zone around the principal slip planes, are based on microstructural criteria.

The mechanical properties of rocks throughout the crust evolve due to many factors, including metamorphic reactions, fluid presence, and textural change. Bulk weakening commonly leads to localization, which can have a profound effect on the strain patterns throughout the crust. We have explored the Twelve Mile Bay shear zone in the Ontario segment of the Grenville Province, Ontario, Canada, in an effort to determine the processes that lead to localization in this km-scale structure (Marsh et al., 2011, 2012a). Our principle conclusion is that hydration of the granulitic protolith along pegmatite-filled fractures caused the weakening and consequent localization. The primary use of the instrumentation in this project was to image zircon in preparation for SHRIMP dating and trace element analysis. The cathodoluminescence imaging was critical in identifying internal zoning and therefore targets for dating. Zircon from pegmatites in the Parry Sound domain, Grenville Province, Ontario, have igneous morphologies and a uniform age along the Twelve Mile Bay shear zone. This supports our hypothesis that pegmatite intrusion along the length of this 20+ km-long structure occurred geologically simultaneously. We are currently exploring how general this pegmatite-induced hydration weakening can be.

Other work in the Parry Sound domain of the Central Gneiss belt in Ontario has been to characterize the conditions leading to the formation of a new species of garnet, menzerite-(Y) (Grew et al., 2010; Marsh et al., 2012b). This is a high Y+HREE garnet whose formation required high temperatures and low pressures, outside the stability field of almandine. Menzerite-(Y) formed during prograde metamorphism and broke down with further heating and burial, eventually becoming armored and therefore preserved by Y+HREE-rich almandine. The instrumentation was used primarily to image the microstructural relationships among the phases in the rock as well as zircon used to date different components of the PT path.

The bulk strength of a rock depends on many factors, including the mineralogy, temperature, fluid presence, and topology of the phases. One way to coarsely characterize the phase distribution is the position of the bulk strength between the theoretical isostress and isostrain-rate bounds (Gerbi et al., 2011). Using the EDS capabilities of the instrument, we have mapped out the phase distributions in selected rocks. We find that many rocks do not move substantially closer to the isostress bound with increasing strain, suggesting that weakening associated with localization in natural systems is due more to chemical (metamorphic) changes than to textural changes.

One of the main tools we have for investigating the deep structure of the Earth, particularly in active orogens, is passive and active seismology. Interpretation of the increasingly finer crustal and mantle structures of interest depends in large part on an accurate understanding of the seismic properties of the rocks through which the waves pass. We have used the instrumentation to characterize the seismic signature of (a) schists with a range of development of crenulation cleavage, one of the most common microstructures in the middle crust (Naus-Thijssen et al., 2011), and (b) lower crustal shear zones. We find that the development of the crenulation cleavage mutes what is otherwise a very strong seismic anisotropy. The development of shear zones at high angles to preexisting foliation affects the bulk anisotropy of lower crustal rocks similarly to that of the crenulation cleavage, but viewed individually, shear zones may not have substantially different seismic anisotropies than their protoliths. This conclusion complements the conclusion about textural weakening described above. However, the orientation of anisotropy in both crenulated and sheared rocks will change from the precursor, which can help identify the presence of that structure deep in the crust.

Accurate quantification of rock deformation requires an accurate characterization of the operative deformation mechanisms. Dissolution-precipitation creep is a recognized deformation mechanism, but its importance is difficult to evaluate due to the challenges of establishing whether it operated. Naus-Thijssen (2011 Ph.D. thesis) used cathodoluminescence imaging to identify precipitation-related overgrowths on quartz grains that formed during crenulation cleavage development. Thus we can confidently state that dissolution-precipitation creep can be an important factor in deformation of schists.

Continued work on the mineral werdingite from a pegmatite from Norway is consistent with an earlier suggestion that werdingite and the boron-bearing mineral boralsilite exhibit limited solid solution (Grew et al., 2011). This limited solid solution makes exsolution of one from the other possible.

Rates of chemical weathering depend on several factors, including the mineralogy, grain size, local microbiology, and environmental conditions. Studies (Bodkin and Negrich M.S. theses) of a ultramafic rocks from coastal Maine suggest that pyroxene is the first mineral to weather in this system. Pyroxene reactivity controls the weathering of the rock with depth by controlling water access to the entire system.

Due to the condition of archaeological bone, species differentiation can be difficult. However, studies have demonstrated that histology (microstructure) is preserved in calcined archaeological bone and can provide valuable information in these cases. Heller (2011 M.S. thesis) contributed to this growing field by identifying histological differences among three important large mammal species - black bear (Ursus americanus), white-tailed deer (Odocoileus virginianus), and caribou (Rangifer tarandus) - and applying these differences in the analysis of calcined bone from the Bull Brook and Morrill Point Mound sites in Massachusetts (10,400 to 3,900 radiocarbon years B.P). Histological analysis of the Bull Brook archaeological samples provided evidence consistent with identified characteristics of bear and caribou. Of the two Morrill Point Mound bone tools, the analysis led to a confident identification of a projectile point as bear right femur. The detailed treatment of each sample emphasized the need for an understanding of how biological processes during life affect microstructural features observed after death and the recognition that groups of characteristics rather than single universal identifiers are required for species identification.

In recent years, recognition of the role of nanostructures in material properties had greatly increased. The imaging capabilities of our instrumentation have assisted in the development of methodologies for controlling the production of certain carbon nanostructures from an organic polymer liquid (Nievandt, 2010).

Training and Development:

Work with the scanning electron microscope has opened up new pathways for observation. For students, the microscope has made observation of microstructure more accessible (i.e., not having to master light optics) and allowed them to better understand the two main components of minerals - structure and composition. For researchers, answering certain basic questions (e.g., related to petrography) has become easier. For everyone, work with the scanning electron microscope has enhanced skills in quantitative analysis.

Outreach Activities:

We have:

? given several tours of the laboratory to school groups
? identified the mineralogy of samples brought in by curious members of the general public
? involved two high school students for two summers each performing research with ERS-affiliated faculty

Journal Publications

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Other Specific Products

Contributions

Contributions within Discipline:

Most of the research using the electron microscope and the associated detectors has centered around crustal rheology. We have investigated multiple crustal levels, with an emphasis on rocks at granulite-upper amphibolite facies conditions and those at midcrustal levels (schists and rocks within the frictional-viscous transition). Taken together, the findings listed above contribute to a deeper understanding of the mechanical structure of the crust at tectonic and seismic timescales. In particular, we can propose that zones of weakness in continental crust result primarily from metamorphic processes. This is not a novel idea, but one that has awaited the testing made possible by this instrumentation. One of the greatest contributions of the instrumentation is the ability use otherwise inaccessible observations of natural systems as the basis for exploring mechanical processes through numerical models. As one example, our research group has developed software, soon to become publicly available, for calculating the bulk properties of elastic, viscous, and power-law materials; although not directly supported by this grant, our progress hinged on EDS- and EBSD-based data. From this work, we strongly emphasize the importance of microstructure on bulk mechanical properties of a polyphase material. On-going work using catholuminescence imaging to document otherwise invisible quartz microstructures has the potential to greatly add to our understanding of the deformation mechanisms, and therefore mechanical properties, of one of the most abundant crustal minerals.

Contributions to Other Disciplines:

One of the most visible contributions of the instrumentation is the characterization of rocks bearing the newly discovered garnet species, menzerite-(Y). The electron microscope has also opened new pathways for comparing bone structure among archaeological finds, assessing nanostructure engineering methods, and analyzing atmospheric dust from snow and ice cores. We have spent considerable time on developing techniques for analyzing particles from ice cores. The work is not yet at a point where we can report findings, but the techniques will allow us to query data from the climate record in ways we have not been able to before.

Contributions to Human Resource Development:

Analytical investigation is a major component in fields within and outside Earth science. Therefore, any training provides our graduates with marketable skills. Even if they do not apply those skills directly, recognition of the power and limitations of analytical instrumentation is part of a scientifically literate society. We cannot quantitatively measure the impact of analytical training on the future pathways of our graduates, but at least three of our former students continue to use analytical instrumentation in their research.

Contributions to Resources for Research and Education:

The laboratory serves as a focal point for visitors (visiting scientists, prospective students, school groups, administrators, and the general public) and has allowed us to engage high school students in summer research projects. As alluded to earlier, the instrumentation has a ripple effect through our program and across campus. The lab's existence has leveraged collaboration with anthropologists, mechanical engineers, and chemical engineers on campus, and Earth scientists from several different institutions. Prospective undergraduate and graduate students are drawn to the way we integrate the instrumentation into our curriculum and the opportunities we provide for research access. Thus, on the whole, the instrumentation has greatly enhanced our research and educational infrastructure.

Contributions Beyond Science and Engineering:

The principal public benefit of the instrumentation is in identifying materials curious members of the general public bring to us. Most are questions related to potential value of rocks and minerals, and we can demonstrate the composition and morphology of their samples with the electron microscope. The Earth and climate science research and education otherwise has only indirect effects on public welfare. The greatest impact we see is in increasing scientific literacy related to microanalysis. The imaging capabilities of the instrument have, however, contributed to a patent application for the production of carbon nanofibers.

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