9-24-2003

MRI: Acquisition Of A Modern Electron Microprobe At The University of Maine

Charles V. Guidotti
Principal Investigator; University of Maine, Orono

Final Report for Period: 09/2001 - 08/2003
Submitted on: 09/24/2003
Principal Investigator: Guidotti, Charles V.
Award ID: 0116235
Organization: University of Maine
Submitted By:

Title:
MRI: Acquisition Of A Modern Electron Microprobe At The University of Maine

Project Participants

Senior Personnel

Name: Guidotti, Charles

Worked for more than 160 Hours: Yes

Contribution to Project:

C.Guidotti acted as Project Director for the acquisition of the microprobe. He coordinated efforts of the co-PIs during the pre-purchase information gathering stage, participated in the selection of the SX-100, and has participated in analytical projects involving the new electron microprobe.

Name: Grew, Edward

Worked for more than 160 Hours: Yes

Contribution to Project:

E.S. Grew contributed in the pre-purchase information gathering stage and selection of the SX-100. He has been active in several analytical projects and has participated in the development of several new UM probe applications.

Name: Lux, Daniel

Worked for more than 160 Hours: Yes

Contribution to Project:

D. Lux contributed in the pre-purchase information gathering stage and selection of the SX-100. He and his students have been active in several analytical projects and have participated in the development of several new UM probe applications.

Name: Johnson, Scott

Worked for more than 160 Hours: Yes

Contribution to Project:

Scott Johnson Lux contributed in the pre-purchase information gathering stage and selection of the SX-100. He has also been very active in the development of monazite age dating techniques using the SX ?100.

Name: Yates, Martin

Worked for more than 160 Hours: Yes

Contribution to Project:

M.Yates as lab manager has had the responsibility to gather technical information on the Cameca and JEOL microprobes and generated the selection criteria. He was responsible for lab preparation and pre-installation purchases. He assisted in the installation for the SX-100 and was trained in the operation and maintenance of the system. He has also coordinated use of the probe by the other PI, co-PIs, and their students and developed lab procedures and techniques for the lab.

Post-doc

Graduate Student

Undergraduate Student

Technician, Programmer

Other Participant

Research Experience for Undergraduates

Organizational Partners

Other Collaborators or Contacts

Activities and Findings

Research and Education Activities:

The NSF-MRI grant EAR - MRI-0116235 titled 'Acquisition of a modern electron microprobe at the University of Maine' provided 70% of the funds necessary to purchase a new electron microprobe and supporting equipment. The acquisition of a new Cameca SX-100 has increased the productivity of faculty and student researchers and has allowed new applications of electron microprobe analysis to on-going UM research.

During the late summer and early fall of 2001, we reviewed detailed instrumental specifications of the JEOL 8200 and Cameca SX-100. Both manufacturers provided M. Yates the opportunity to visit working labs for probe demonstrations. JEOL and Cameca prepared detailed quotes and responded to a series of technical specifications in late October 2001. We used the information provided from the venders and independent reviews from several microprobe lab managers to determine which instrument best suited UM's research needs. In early November 2001, we chose the Cameca SX-100 on the basis of both capabilities and price. Cameca offered UM a substantial educational discount and provided superior WDS, light optics, and more versatile EDS spectrometer options. The new microprobe arrived at UM in the first week of August 2002 and was installed and tested from August 12 to September 9, 2002. The new system has been in continuous operation since September 9, 2002 and has produced a significant quantity of high quality analytical data.

The configuration of UM's SX-100 is designed to balance versatility with high performance. Four of the probe's 5 spectrometers are dual crystal, fitted with high intensity, large area TAP-PET or PET-LiF crystals. Large area crystals have significantly higher count rates than standard crystals and are particularly well suited to minor element analysis. One spectrometer contains a 4 standard-size crystal turret. This spectrometer holds a TAP crystal and 45, 95, and 200 ? synthetic layered crystals that are required for light element analysis. Routine analysis of fluorine through carbon is possible with these crystals. Boron and beryllium can also be analyzed but with greatly reduced detection limits. The probe is also equipped with a R?ntec Xflash 2000 EDS spectrometer. The detector is thermoelectrically cooled and therefore requires no liquid nitrogen. The R?ntec makes use of a silicon drift diode instead of the traditional Si(Li) detector. This new technology allows excellent x-ray spectral resolution even under the high beam current conditions used in many microprobe applications. With the SX-100's pinhole shutter installed in front of the detector useful spectra can be collected at beam currents in excess of 200nA.

Analysis of lead, uranium, and thorium in monazite is vital to several research projects using the University of Maine's electron microprobe. Successful analysis of these minor elements in a complicated matrix of rare earth elements in monazite requires high spectral resolution, high count rates, and extended counting times. The use of long counting times under high beam current conditions leads to deposition of carbon on the analytical spot. The University of Maine's probe is equipped with an oil free turbomolecular secondary vacuum pump, which maintains a cleaner vacuum in the probe column. The probe is also fitted with Cameca's anticontamination attachment. A liquid nitrogen cooled plate above the specimen and a directed oxygen stream work together to reduce the carbon contamination rate by a factor of 10.

UM purchased several supporting systems recommended by Cameca. The power for the SX-100 is supplied through an uninterruptible Liebert 20kVA UPStation. The resulting power is filtered and can be maintained through power outages of short duration. Water circulation is maintained through a Haskris closed loop water-cooled chiller. The lab's carbon evaporator has also been replaced in order to provide faster, more uniform conductive carbon coating of sections to be analyzed. The lab's new Emitech K950X carbon evaporator provides programmable evaporation power and timing and has attachments for both gold and carbon evaporation. The use of gold coating is currently recommended to stabilize minerals for analyses of long duration at high beam current.

In the lab's first year of operation, we have become well acquainted with Cameca's software and service support. The University of Maine negotiated a three-year, full warranty on the system. The initial installation and training was performed by Jean-Michel Peneau from Cameca's Paris office. He proved to have excellent technical and training skills, although his experience with the new PC-based Peak Software was

limited. Several key routines were non-operational in the software that was shipped with the probe. However, we have been able to communicate these software difficulties to Cameca's Paris office and have received appropriate program 'fixes'. During installation and throughout our first year of operation we have experienced an intermittent problem with our vacuum system that proved more difficult to diagnose. The secondary vacuum gate valve would close without apparent cause, interrupting automated analytical sessions. Cameca's USA service group visited the lab 5 times in an effort to fix the problem. The malfunction was finally diagnosed as a faulty secondary vacuum gauge, and we have experienced no further vacuum system failures. Through this experience, we found Cameca's USA service staff to be both professional and knowledgeable.

Once the probe was operational and its functions tested, it was put to use immediately for the research and educational purposes for which it was intended. These activities are described in the other sections of the report.

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Findings:

The University of Maine's microprobe laboratory is designed as a research facility serving the University of Maine and the region. In the first 12 months of operation, the level of research activity can be measured in the numbers of research projects and publications containing UM microprobe data. We have had 20 multi-session probe projects that have involved 10 faculty researchers, 7 undergraduate students, and 7 graduate students. Of these, 3 projects have been initiated by UM researchers outside of the Department of Earth Sciences and 5 have been from outside of the University of Maine. These projects have generated microprobe data that is included in 6 journal articles (either in press or in preparation), 9 published abstracts, 5 abstracts in press, 3 undergraduate theses, and 1 Masters thesis.

Significant advances in science have been made through several of the projects utilizing the new probe:
? E. Grew has combined ion microprobe data on Li, Be and B with electron microprobe data on major constituents in a study of the role of these light elements in ultrahigh-temperature (> 1000 ?C) metamorphism in Napier Complex (Antarctica) metapelites, specifically, the role of metamorphic sapphirine in the development of Late Archean anatectic pegmatites containing Be minerals.
? M. Yates and E. Grew have found aureoles of extraordinary lead enrichment in potassium feldspar adjacent to thorian monazite in the Napier Complex anatectic pegmatites. The distribution of lead between feldspar and monazite has important implications for understanding the diffusion of lead in these minerals and for interpreting ages of crystallization determined with the electron microprobe.
? Under the direction of E. Grew and M. Yates, undergraduate student A. Roy is studying the partitioning of the halogens F and Cl between wagnerite, apatite and biotite in the Napier Complex pegmatites and implications of halogen partitioning for understanding anatexis at ultrahigh temperatures.
? E. Grew and M. Yates in collaboration with scientists in Canada, Sweden and Russia have used the probe to characterize the new mineral makarochkinite, an iron-rich beryllosilicate of the aenigmatite group. Characterization required a full chemical analysis for elements with Z > 8,

fully within the capabilities of the Cameca SX 100, which was also used to characterize minerals associated with makarochkinite. The new mineral has been approved by the Commission of New Minerals and Mineral Names, International Mineralogical Association.
? D.Lux has used various techniques (elemental maps, traverses and spot analyses) to study zoning in plagioclase phenocrysts and accessory minerals in order to assess changing P-T conditions during magma emplacement and crystallization. He and his students have also used the Al-in-hornblende geobarometer to investigate the depth of intrusion. They have also characterized the chemical composition of major and accessory minerals from numerous granite bodies.
? S. Johnson and his students have made use of the probe to determine pressure-temperature conditions of tectonic events in the Appalachian orogen in western Maine. EMP analysis of rocks from the pre-Silurian bedrock of west-central Maine proved critical to unraveling several persistent problems related to the earliest stages of Appalachian orogenesis in the area. Specifically, in Chain Lakes massif and the adjacent ultramafic Boil Mountain Complex, we determined the composition of several metamorphic phases, including muscovite, biotite, plagioclase, and hornblende, in order to constrain the metamorphic conditions. In addition, we used the EMP to measure and calculate chemical ages for monazite from several samples within the Chain Lakes massif.
? S. Johnson has been investigating the micro-chemical zonation of minerals in tectonic fabrics in metamorphic rocks in order to characterize the processes active along grain boundaries during strain-induced recrystallization.

Training and Development:

The SX-100 microprobe has served as a training ground for students in electron beam microanalysis and in mineralogy and petrology. Seven undergraduate and seven graduate students have used the microprobe as part of research projects. In addition, the microprobe has been used for laboratory demonstrations in UM's introductory geology course given mostly to non-geology majors. In every case, students become acquainted with the features of the electron microprobe, the operating principles, and applications. Students using the probe for research are instructed in the underlying theory of electron microanalysis and are trained in probe operation..

Outreach Activities:

The electron microprobe lab has hosted K-12 classes on four occasions in the last year. The microprobe was also used as a laboratory demonstration during the Annual High School Teachers Workshop, which is held annually by the Department of Earth Sciences. The lab is designed with an area in front of the console sufficiently large for 15 students or teachers to view SEM images and x-ray spectra. During each demonstration, individuals are shown how to locate analytical points on a mounted sample, collect an x-ray spectrum with the EDS, and to identify minerals from the characteristic x-ray spectra. This hands-on experience has generated enthusiasm for science, and participants have asked very thoughtful questions about mineralogy, petrology, and electron beam microanalysis.

Journal Publications

Barbier, J., Britten, J., Grew, E.S., Yates, M.G., Polyakov, V.O., Shcherbakova, E.P., H?lenius, U. and Shearer, C.K., "Makarochkinite, a new beryllosilicate member of the aenigmatite group and the Ti-dominant analogue of h?gtuvaite [Data set is approaching completeness.", American Minerologist, p. , vol. , ( ). in Preperation,

Christy, A.G. and Grew. E.S., "Synthesis of beryllian sapphirine in the system MgO-BeO-Al2O3-SiO2-H2O and comparison with naturally occurring beryllian sapphirine and khmaralite. Part 2: a chemographic study of Be content as a function of P, T, assemblage and FeMg-1 exchange.", American Mineralogist, p. 1, vol. , ( ). Accepted,

Grew, E.S., Yates, M.G., Shearer, C.K., Hagerty, J.J., Sheraton, J.W., and Sandiford, M., "Beryllium in pelitic granulites and anatectic pegmatites of the ultrahigh-temperature Napier Complex, Enderby Land, East Antarctica: The role of sapphirine.", Journal of Petrology, p. 1, vol. , ( ). in Preparation,

Lux, D.L. and Yates, M.G.,, "Plagioclase zoning and magmatic processes in the Deer Isle Magma Chamber", Atlantic Geology, p. , vol. , ( ). in Preparation,

Lux, D.L., Hooks, B.P., Gibson, D., and Hogan, J, "Evolution of the Deer Isle Pluton", Journal of Geology, p. , vol. , ( ). in Preparation,

Vernon, R.H., Johnson, S.E. and Melis, E.A., "Transition from magmatic to solid-state flow in the marginal deformation of an intruding pluton: the San Jose tonalite, Baja California, Mexico", Journal of Structural Geology, p. , vol. , ( ). Submitted,

Choate, M. A. and Gibson, D., "Distribution, petrography and chemistry of rapakivi feldspars in the Mt. Waldo pluton, coastal Maine", Geological Society of America 2003 Abstract with Programs, p. 36, vol. 35, (2003). Published,

Day, K. J. and Gibson, D., "Origin of garnets from the peraluminous North Jay pluton, west-central Maine. Magmatic or xenocrystic", Geological Society of America 2003 Abstract with Programs, p. 35, vol. 35, (2003). Published,

Dupee, M.E., Johnson, S.E., and Guidotti, C.V., "Relations between deformation and metamorphism in the aureole of the Mooselookmeguntic pluton, western Maine", Geological Society of America Abstracts with Programs, p. 13, vol. 35, (2003). Published,

Gerbi, C., Johnson, S.E., "The Chain Lakes massif, Maine and Quebec: A product of extension?", Geological Society of America Abstracts with Programs, p. 29, vol. 35, (2003). Published,

Gontz, A.M., Belknap, D.F., and Kelley, J.T., "The relationship between packmarks, gas-enhanced reflectors and acoustic wipeout in an active estuarine pockmark field, Penobscot Bay, Maine", Geological Society of America Abstracts with Programs, p. 7, vol. 35, (2003). Published,

Grew, E.S., Yates, M.G., Shearer, C.K., Hagerty, J.J., Sheraton, J.W., and Sandiford, M., "Beryllium in pelitic granulites and anatectic pegmatites of the ultrahigh-temperature Napier Complex, Enderby Land, East Antarctica: The role of sapphirine.", Geological Association of Canada, Mineralogical Association of Canada, Joint Annual Meeting, Abstracts, p. 1, vol. 28, (2003). Published,

Lipfert, G., "Arsenic distribution and geochemical relationships in a fractured-bedrock aquifer", Geological Society of America 2003 Abstract with Programs, p. 78, vol. 35, (2003). Published,

Lux, D.R. and Yates, M.G., "Reading the plagioclase record", Geological Society of America 2003 Abstract with Programs, p. 36, vol. 35, (2003). Published,

Reusch, D. N., Van Staal, C., Barr, S.M., and Yates, M.G., "Ultramafic and sedimentary dikes, Penobscot Bay: Cambrian extension and Ordovician accretion along southeastern Ganderia", Geological Society of America 2003 Abstract with Programs, p. 78, vol. 35, (2003). Published,

Johnson, S.E., Vernon, R.H., Upton, P., Melis, E.A., "Evidence for fast magma chamber construction: the deformed carapace of the San Jose tonalite pluton, Mexico", Geological Society of America Abstracts with Programs, p. , vol. 35, (2003). Accepted,

Lipfert, G., Reeve, A., "Fracture surface chemistry in an arsenic-rich bedrock aquifer", Geological Society of America Abstracts with Programs, p. , vol. 35, (2003). Accepted,

Roy, A., Grew, E.S., and Yates, M.G., "Wagnerite, apatite and biotite in granulite facies rocks of the Napier Complex, Enderby Land, Antarctica; the role of Halogens", Geological Society of America Abstracts with Programs, p. , vol. 35, (2003). Accepted,

Short, H.A., Upton, P., Koons, P.O., Johnson, S.E., "The exhumation Of a high metamorphic grade terrane within a transpressional orogen: constraints from numerical modeling in central coastal Maine", Geological Society of America Abstracts with Programs, p. , vol. 35, (2003). Accepted,

Yates, M.G. and Grew, E.S., Wilson, C.J.L., "Lead aureoles in alkali feldspar around thorian monazite-(Ce) in Late Archean granulite-facies pegmatites of the Napier Complex, Khmara Bay, East Antarctica", Geological Society of America Abstracts with Programs, p. , vol. 35, (2003). Accepted,

Books or Other One-time Publications

Larkin, R., "Electron microprobe age dating of monazite from the Bretton Woods granite, Presidential Range, New Hampshire", (2003). Thesis, Published
Bibliography: Senior Honors Thesis, Bates College, Lewiston, Maine, 106 p.

Hooks, B.P., "Petrology of the Deer Isle Pluton: A complex mixed magma system", (2003). Thesis, Published
Bibliography: Masters Thesis, University of Maine, Orono, 185p

Web/Internet Site

Other Specific Products

Product Type:

New Mineral

Product Description:

The chair of the Commission of New Minerals and Mineral Names, International Mineralogical Association, has informed us that makarochkinite is approved as a valid new mineral species.

Sharing Information:

This new mineral discovery will be the subject of a journal which is currently in preparation

Contributions

Contributions within Discipline:

The Cameca has been used to analyze a wide variety of minerals. Some of these have required development of special protocols and unusual element lists not generally encountered in the earth sciences. In addition to analyses of the major rock-forming silicate minerals using standard element lists (Na, Mg, Al, Si, K, Ca, Ti, Cr, Mn, Fe), we have developed protocols and element lists for the following:
? Halogens in biotite and hornblende. F is particularly difficult because of low count rates and interferences with Fe and other heavier elements
? P and F in the phosphate minerals wagnerite and apatite
? Nb, Ta, W, Th, rare earths, Y, Zr, Hf, etc. in complex oxides in ultrahigh-temperature granulites (Napier Complex) and pegmatitic minerals including makarochkinite and its associates
? Rare earths, Y, Th, U, Pb, etc. in monazite-huttonite solid solutions both for chemical characterization and dating

The Cameca has allowed us to create element profiles and maps of zoned minerals, not only of major constituents in common rock-forming minerals such as plagioclase, orthopyroxene and garnet, but also of trace and minor elements such as Pb in monazite and K-feldspar. The cathodoluminescence capability has allowed visualization of zoning patterns not readily observable by other means.

Contributions to Other Disciplines:

The University of Maine's electron microprobe lab is available for non-earth science scientific and engineering applications. While most of the use of the probe in the last year has involved earth science applications, several projects have shown the potential of the system to serve a broader community. The probe has the ability to image chemical heterogenieties in a wide variety of materials. The University of Maine has an active wood technology group that is developing technologies that will allow renewable wood resources to be used as high strength construction materials. Back-scattered electron images of fiberglass-wood laminates collected on our probe have proved useful in determining the causes of structural failure under load. UM's laboratory of surface science and technology is involved with the development of sensor technology. They have found the probe useful in characterizing materials used in development. The presence of a full function, modern electron microprobe facility on campus at the University of Maine will continue to inspire researchers to discover new applications of electron microbeam analysis. The ability of researchers to quickly test ideas using our system facilitates scientific inquiry and will lead to innovation and discovery.

Contributions to Human Resource Development:

The electron microprobe facility at the University of Maine has and will continue to serve as a training site for students and faculty researchers in electron microbeam analysis. The uniquely visual interface and control of the system engages students and makes the complex operational characteristics of the microprobe easier to understand. The microprobe is also an excellent tool for teaching mineralogy and petrology. Classroom discussions of mineral chemistry, mineral chemical variations, and mineral interactions are easily demonstrated with the probe. In operating the probe, students make discoveries in mineralogy and petrology on their own. Learning use of the microprobe and applying it in research enhances an undergraduate student's prospects in getting into graduate school and an undergraduate or graduate student's prospects for employment.

The electron microprobe lab has also developed into a gathering place of students and faculty. The lab is often full of student and faculty earth scientists discussing probe results and new analytical ideas. Student researchers from neighboring colleges and universities have the opportunity to interact with members of UM's earth sciences faculty just as visiting researchers interact with our students.

UM's microprobe lab also has welcomed every opportunity to showcase its capabilities to pre-college students, K-12 teachers, and the public. We have hosted several tours of third graders and two high school groups. The probe was used in a workshop for high school science teachers. The participants are given the opportunity to operate a few of the controls of the microprobe and leave with a better understanding on how this advanced technology is used to gain a better understanding of geological processes.

Contributions to Resources for Research and Education:

Although the UM has had a functional electron microprobe lab since the mid-1980's, the acquisition of the new Cameca SX-100 probe represents a major improvement in analytical capability for the University and the region. No comparable system exists in the state of Maine. We have served and will continue to serve this region's analytical needs.

Contributions Beyond Science and Engineering:

UM's probe facility has the potential to be applied in assessing environmental risks posed by manufactured and natural contaminants. Ground water and surface water are increasingly invaded by man-made pollutants. Use of new ground water reservoirs has the potential to release natural toxic chemicals into drinking water. A UM student has been using the probe to evaluate bedrock as a potential source of arsenic measured in drinking water in Bayside, Maine. Samples of drill core from this area have shown that one possible source of arsenic is the mineral arsenopyrite present in the bedrock through which ground water tapped by wells has percolated.

Conference Proceedings

Categories for which nothing is reported:

Organizational Partners

Any Web/Internet Site

Any Conference

NSF Annual Report

Award No. EAR-0116235 to the University of Maine

"MRI: Acquisition of a Modern Electron Microprobe at the University of Maine"

This report summarizes progress on behalf of the PI and co-PI's toward acquisition of a new electron microprobe at the University of Maine. Insufficient funds available in FY2001 require the funds to be made available by NSF in two installments of $237,000, one in FY2001 and the second in FY2002. The first was made available in September, 2001. The second is expected after October 1, 2001.

On receipt of preliminary notification from NSF that our proposal would probably be funded (June 25, 2001), the PI and co-PI's began a concerted effort to investigate the relative merits of the latest models of electron microprobe systems, that is the Cameca SX-100 and the JEOL 8200. We have requested from the two manufacturers detailed specifications of hardware and software systems and options that are required for our purposes. We have contacted numerous electron microprobe laboratories having similar systems, and have requested information on instrument quality, quality of service, and the relative merits of different options and configurations. We visited the Cameca SX-50 electron microprobe laboratory (the immediate predecessor to the SX-100) at the University of Massachusetts, Amherst. During that visit we had discussions with Michael Williams and Michael Jercinovic regarding optimum probe configurations for analysis of monazite for purposes of age determination. We also had the opportunity to meet with two representatives from Cameca and witness a demonstration of the new Cameca PC-based software.

In August and September, co-PI Martin Yates participated in demonstrations of the JEOL 8200 electron microprobe system at the University of Calgary in Calgary, Alberta and of the Cameca SX-100 in Paris, France. In the course of these demonstrations, a battery of tests was performed on both the JEOL 8200 and Cameca SX-100 microprobes using

samples from current research projects at the University of Maine. Test analyses performed on these systems are as follows:

- analyses of the beryllium oxides chrysoberyl and musgravite to test the instrument's capacity to analyze for the ultra-light element Be and elements not routinely sought (Ga, Zn, Cr)
- analysis of sillimanite to test overall quality of analysis
- analysis and chemical mapping of zoned plagioclase
- WDS scan of rutile to search for trace Nb, Ta, W
- Chemical mapping and analysis of monazite

These tests proved to be very instructive, and will figure prominently in our decision on which system to acquire.

We are currently preparing a "Request for Proposal" for the University of Maine to submit to JEOL and Cameca as a form of bidding. This formal request will outline our instrumental and service requirements. The response from JEOL and Cameca to that request will determine which system we will acquire. Our objective is to place an order by the end of October, 2001, and thus we expect to have our new electron microprobe system installed and operational by mid-Summer, 2002.