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Acquisition of an Automated Powder X-Ray Diffraction System

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Organization: University of Maine

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Participant Individuals

Senior Personnel

Name: Guidotti, Charles
Worked for more than 160 Hours: Yes
Contribution to Project:

Name: Grew, Edward
Worked for more than 160 Hours: Yes
Contribution to Project:

Name: Yates, Martin
Worked for more than 160 Hours: Yes
Contribution to Project:

Post-doc

Graduate Student

Undergraduate Student

Partner Organizations

Other Collaborators

University of Maine Laboratory for Surface Sciences and Technology

Activities and Findings

Research Activities:
The major accomplishment resulting from the present grant is the purchasing and making operational a multi-user x-ray facility in the newly built Bryand Global Sciences Center, where the Department of Geological Sciences moved to in 1997.

After thoroughly investigating a number of XRD systems, the Scintag, Inc. X2 system was chosen. The decision was made on the basis of features, service, quality, and cost.

The X2 system includes:
4.0 kW High Voltage Generator
Theta/Theta Goniometer
Continuously Adjustable Slits
X2 Diffraction Microprocessor Assembly
Fine Focus, Copper Target X-Ray Tube
DMS/NT Data Acquisition And Analysis Software
Crystallographic Analysis Software

Quantitative Analysis Software
Thermoelectically-Cooled, Solid-State Peltier X-Ray Detector.

Several Scintag options were selected which include:
Half circle pole figure and stress attachment
DMS/NT Stress Analysis Software
DMS/NT Reflectometry Software
DMS/NT Polar 3-D, Texture Analysis Software
Parabolic Incident Beam X-Ray Mirror
Thin Film Grazing Angle Collimation Attachment.
Computer controlled incident beam slits

Several additional third-party components in this XRD facility include:
ICDD Powder Diffraction Database
2 Windows NT Computers
Haskris, Inc. Water-to-Water Chiller.

The University of Maine's Laboratory for Surface Science and Technology (LASST) was included in the original proposal as a strong potential user of this facility. LASST was consulted in the selection of the XRD instrumentation and it has contributed an additional $33,000 to the laboratory to purchase several of the Scintag options listed above.

The system was ordered in the spring of 1998 and was fully operational in mid-December 1998. Since that time the system has been available to the University of Maine campus community and has routinely been used at least 30 hours a week. All of the components have been installed and are operational except for the computer controlled slit system, which will be shipped from the manufacturer shortly.

Research Findings:
Use of the system by researchers and students of the Department of Geological Sciences has been both to identify and to determine the crystallographic properties of minerals.

Dr. Joseph Chernosky uses XRD to characterize chlorite that had been equilibrated in buffered assemblages at various pressures and temperatures in hydrothermal experiments. The composition of chlorites which spontaneously nucleated and grew in a set of experiments initially designed to reverse the reaction tremolite + diopside + spinel = Anorthite + Forsterite + H2O were measured by precisely monitoring the position of the d004 reflection with our Scintag automated diffractometer. Two sigma errors about the mean position of the d004 reflection are about an order of magnitude more precise than measurements made using our manual XRD system. The XRD data indicate that the chlorite composition changes with both T and P. At temperatures substantially below the 'equilibrium curve', the chlorite is enriched in Al2O3 (the higher the P, the higher the Al2O3, up to a maximum of ~23 wt.%) relative to end-member clinochlore (18.34 wt.% Al2O3). As the curve is approached with increasing T, chlorite becomes less aluminous (~20-21 wt.%). The more precise chlorite compositional data obtained with the Scintag XRD will allow determination of activity-composition relations for MgAl-chlorite as a function of P(H2O) and T.

Masters student Thomas Henze used the new system to identify fine-grained, blue-tinted horizons in sediment cores as the uncommon hydrated Fe2+ phosphate vivianite. He also analyzed dark bluish-gray clay within these sediment cores. Mixed quartz, chlorite, illite, and plagioclase proved useful in correlating this clay with a local glacial-marine sediment in the Presumscot Formation. Undergraduate student Anthony Jackson analyzed fine-grained host-rock above the main ore zone at the Cape Rosier volcanogenic massive sulfide deposit as part of his Senior Capstone project. He determined this material to be muscovite and interpreted it to have formed as a result of hydrothermal alteration of volcanogenic sediment shortly after ore formation.

The facility has also been used extensively by other research units on the University of Maine campus, particularly the Laboratory for Surface Science and Technology (LASST). Scott Moulzoft of LASST made extensive use of our XRD facility to characterize thin ZrO2 films. This work constituted a substantial portion of his Ph.D. dissertation titled 'Synthesis and characterization of ZrO2 thin films with controlled Microstructure' and has resulted in two presentations at professional meetings and one submitted journal article.

In adddition to LASST, Ron Davis from the Quaternary Institute has used the system to determine the mineralogy in sediment core in a volcanic terrain. The presence of plagioclase and augite with only minor clay minerals was an important finding because it showed the lack of chemical weathering within the sediment pile.

Research Training:

This XRD facility has provided an opportunity for students and faculty at the University of Maine to use a fully automated, modern x-ray system. The age and disrepair of our previous diffractometer precluded use by students and as a result a generation of students received very little training in this important analytical instrument. Both graduate and undergraduate students are now receiving training in x-ray diffraction. They should be able to identify and analyze minerals with the systems software on their own with minimal assistance. Undergraduate participants in the Department's course 'Modern Analytical Methods' (GES332) were trained in the operation of the XRD system and in interpretation of XRD data. Each of 16 the students prepared, analyzed, and determined the identity of the several unknown minerals. In one assignment the students used XRD data to determine the dimensions of the unit cell of an unknown mineral.

Education and Outreach:

Journal Publications

Moulzolf, S.C. and Lad, R.J., "Diffraction studies of cubic phase stabilization in undoped zirconia thin films", *J. Materials Research*, p. , vol. , ().) Submitted

Books or Other One-time Publications

Web/Internet Sites

URL(s):

Description:

Other Specific Products

Contributions

Contributions within Discipline:
Reported above
Contributions to Other Disciplines:
Reported above
Contributions to Education and Human Resources:
Contribution to completion of one Ph.D. by a LASST student
Contribution to completion of one M.S. by a geology student
Contribution to completion of one geology senior capstone research project

Contributions to Science and Technology Infrastructure:

Beyond Science and Engineering:
Reported above

Categories for which nothing is reported:

Partner Organizations
Activities and Findings: Any Education or Outreach
Any Book
Any Product
Contributions: To Any Science or Technology Infrastructure