Year 2 Annual Progress Report of the Consortium for Materials Properties Research in Earth Sciences (COMPRES)

February, 2004

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A COMPRES YEAR 2: OVERVIEW

A.1 Executive Summary

In 2003, substantial progress has been made in achieving the objectives and goals of the Consortium for Materials Properties Research in Earth Sciences [COMPRES]. Major technological advances at the community facilities operated by COMPRES at national laboratories and the infrastructure development projects sponsored by COMPRES have enabled new scientific research opportunities in the field of high-pressure mineral physics and chemistry.

The management of these community facilities and infrastructure development projects is monitored by Standing Committees elected by the representatives of the member institutions of COMPRES under policies and procedures established by the committees and endorsed by the Executive Committee. As the consequence of a membership campaign, there are now 35 U. S. institutions which are voting members of COMPRES [the Electorate] and another 11 non-voting institutions which have affiliate membership.

COMPRES has sponsored and/or organized many workshops and scientific meetings in the past year. These include the COMPRES Annual Meeting in Santa Cruz, California in June and a special Workshop on "High Presssure Earth and Planetary Sciences: Our Planet from Surface to Center and Beyond" convened in Miami, Florida in March at the request of the NSF Division of Earth Sciences. Other workshops: (1) "Neutrons in Solid State Chemistry and Earth Sciences Today and Tomorrow, Oak Ridge, Tennessee, March 2003; (2) "Workshop on Mantle Composition, Structure, and Phase Transitions", Frejus, France, April 2003; (3) IUCr/COMPRES "High-Pressure Workshop on High Pressure Structure and Reactivity: The Science of Change", Lawrence Berkeley National Laboratory, Berkeley, California, December 2003.

Following an international search process, the Executive Committee appointed Robert Liebermann as President of COMPRES for a four-year term effective September 2003, succeeding Jay Bass who served ably as the first President in 2002-2003. COMPRES communications have evolved during the past year to include an improved website with links to other activities in high-pressure mineral physics and associated geoscience fields, a People database, videoconferencing facilities, a quarterly newsletter, monthly messages from the President to the COMPRES community, and an exhibition booth at the Fall 2003 AGU Meeting in San Francisco.

In this section of the Annual Report for Year #2, we present an overview of the activities of COMPRES. Subsequent sections include detailed reports from each of the Community Facilities operations and Infrastructure Development projects supported by COMPRES. The final section presents the budget plan for Year #3 [May 1, 2004 to April 30, 2005]; detailed budgets and justifications are given in the appendices to this report.

A.2 Research Accomplishments

Here we highlight a few of the accomplishments of the past year, indicating which section in this report describes the item in more detail.

- Discovery of New High Pressure Phase of Ice. The most thoroughly studied compound yields another phase transition to IR investigators. – see DAC at NSLS section.
- High pressure studies of the rheology of olivine suggest surprisingly small pressure dependence. – see multi-anvil facilities at NSLS
- Generation of ultrahigh (200 GPa) pressure using single-crystal CVD diamond anvils – see DAC at NSLS section.
- Breakthrough on Johnson noise thermometry. This tool promises to remove the ambiguity of pressure effect on thermocouple temperature measurement – see the section on absolute pressure and temperature calibration.
- SNAP proposal funded by DOE-BES at $11.8million for 5 years – see the section on COMPRES neutrons.
- Better-cheaper multi-anvil cell assemblies are on the way — see the section on multi-anvil cell assembly development.
- Laser heating design for GSECARS nearing completion – see section on development of the laser-heated diamond anvil cell.
- Technique to measure melt density at high P and T – see multi-anvil facilities at NSLS.
- NSLS investment of $1.44M to build new COMPRES high pressure hutches at NSLS completed – see multi-anvil facilities at NSLS
- ALS development spurred on by new beamline scientist, Martin Kunz, beamline assistant, Katrina Opachich, and visiting scientist, Michael Walter – see West Coast facilities section.
- Pressure scale under review – see section on pressure calibration at high temperatures.

A.3 Meetings and Workshops

The following meetings and workshops were sponsored, at least in part, by COMPRES:

A New Generation of Quantitative Laser-Heating Experiments

Feb. 22nd 2003, at the Advanced Light Source was focused on the community's development of a new generation of quantitative laser-heating experiments. A report on this meeting is available on request from either the ALS or COMPRES

Neutrons In Solid State Chemistry and the Earth Sciences Today and Tomorrow

Co-chairs: Angus Wilkinson (GA Tech) and Nancy Ross (VA Tech), *.March 12-16, 2003,* http://www.sns.gov/jins/NICEST2003/

A. Wilkinson (GeorgiaTech) and N. Ross (Virginia Tech) co-chaired the Joint Institute of Neutron Scattering Workshop: Neutrons In Solid State Chemistry and the Earth Sciences Today and Tomorrow, (March 12-16, 2003). The following attendees received grants from COMPRES:

- Charles Martin (graduate student, Stony Brook)
- Darren Locke (graduate student, Arizona State Univ.)
- Meghan Knapp (graduate student,Ohio State Univ.)
- Kinsom Kam (graduate student, Univ. Calif. Santa Barbara)
- Christopher Holl (graduate student, Univ. Colorado)
- Wendy Mao (graduate student, Univ. Chicago)
- Kim Tait (graduate student, Arizona State Univ.)
- Yang Ding (postdoctoral associate, Geophysical Lab)

R. Angel (Virginia Tech) and B. Toby (NIST) ran a 2-day "hands-on" short course on Rietveld refinement methods directly after the workshop.

High-Pressure Earth & Planetary Sciences in the Future

March 22-23, 2003:

To help define future priorities, a COMPRES workshop on High Pressure Earth and Planetary Sciences: Our Planet from Surface to Center and Beyond, was held on March 22-23 at Miami, Florida. Over 50 scientists from universities, national labs and overseas institutions attended the workshop. Dr. David Lambert from Earth Sciences Division of NSF gave a brief introduction including NSF's expectation from the workshop. Discussions were focused on four major topics: Near Surface Processes, Subduction and the Mantle, the Core and CMB, and Planetary Geology and Impacts. The participants reviewed the scientific advances in these areas in the past decade, significant technologies that enabled the advances, and the current most important issues for future research. Expertise gathered at the workshop extended beyond mineral physics and included seismology and synchrotron x-ray instrumentation. A brief Summary report of the workshop was sent to NSF on May 1, and may be found at http://www.compres.stonybrook.edu/Meetings/2002-03-22/MiamiSummary toNSF-May2003.doc

A brochure on the Future of High Pressure Earth and Planetary Sciences is under preparation in collaboration with Ellen Kappel of GeoProse, Inc..

Mantle Composition, Structure, and Phase Transitions

April 2003: Frejus, France, International Meeting

Workshop on Mantle Composition, Structure, and Phase Transitions, April 2-6, 2003, Frejus, France. http://ens-lyon.fr/LST/WorkShop/frejus

Over 60 scientists from a number of countries discussed topics in geochemistry, mineral physics, geodynamics, and seismology. Presentations included broad state-of-the-field review, and latest research results on a diverse range of subjects including the core-mantle boundary, core-mantle interactions, composition of the lower mantle and transition zone, geochemical reservoirs, seismic structure of the mantle, plumes, mantle convection, high P-T phase transitions, electronic transition in minerals, etc. Invited speakers included Stan Hart, John Brodholdt, Denis Andrault, David Bercovici, Mike Brown, Geoff Davies, Rob van der Hilst, Don Helmberger, Yannick Ricard, Guillaume Fiquet, Don Weidner, Michael Weber, Michael Manga, Kei Hirose, Philippe Gillet, Alex Rocholl, and Renata Wentzcovitch.

The meeting was co-sponsored by COMPRES (USA), CNRS (France), the GeoForschungsZentrum (Potsdam, Germany), and the Japan Ministry of Science & Education. Organizers were Jan Matas, T Katsura, F Schilling, and J Bass.

COMPRES Annual Meeting

June 18-20, 2003: Santa Cruz, California

http://www.compres.stonybrook.edu/Meetings/2002-06-18/index.html

The second COMPRES Annual Meeting was held at Santa Cruz on June 18-20, 2003. Meeting agenda, submitted abstracts and minutes of the meeting are available on the COMPRES web page: http://www.compres.stonybrook.edu/Meetings/2003

-06-18.

The following new members of the current COMPRES committees were elected at the annual meeting:

Executive Committee:

Jay Bass (University of Illinois at Urbana Champaign)

Facilities Committee:

Thomas Duffy (Princeton University)

Charles Prewitt (University of Arizona)

Infrastructure Development Committee:

Kevin Righter (NASA)

Nancy Ross (Virginia Polytechnic Institute and State University)

Our deep appreciations to the rotated-off committee members for their voluntary services for the community. They are Thomas Duffy (Executiv Committee), Quentin Williams and Michael Brown (Facilities Committee), David Walker and Raymond Jeanloz (Infrastructure Development Committee).

High-Pressure Workshop on High Pressure Structure and Reactivity: The Science of Change

IUCr/COMPRES High-Pressure Workshop on High Pressure Structure and Reactivity: The Science of Change, December 4-7, 2003, Lawrence Berkeley National Laboratory, Berkeley, California.

The International Union of Crystallography (IUCr) and the Consortium for Materials Properties Research in Earth Sciences (COMPRES) co-sponsored a workshop on the application of crystallographic techniques to the study of chemical reactions and phase transitions induced by the application of high-pressure. The aim of the Workshop was to review the current state of this field, and to attempt to predict the course of research in the future. The Workshop was arranged into a number of symposia with invited talks, talks selected from abstracts and poster sessions. There was also a Practicum designed for high level instruction in high-pressure techniques, and open to all attendees at the Workshop. There was be a commercial exhibition. The Workshop was be held on December 4-7, 2003, immediately preceding the American Geographical Union Meeting in San Francisco. A large number of bursaries to provide support for students to attend the meeting and practicum were be available.

The following workshops and workshops were organized by members of the COMPRES community:

The Center for the Study of Matter at Extreme Conditions

March 24-28, 2003:

SMEC Meeting in Miami

http://www.cesmec.fiu.edu/SMEC/SMEC 2003.htmSMEC Meeting in Miami - March 24-28, 2003

Experimental Rheology at Extreme Conditions

April 6-11, 2003

EGS-AGU-EUG Joint Assembly - Nice, France, April 6-11, 2003
Special Session: MG15: Experimental Rheology at Extreme Conditions in EGS-AGU-EUG Joint Assembly Nice, France, April 6-11,2003

Workshop on High Pressure Mineral Physics using Synchrotron Radiation

May 19-21, 2003

NSLS Users' Meeting - May 19-21, 2003
Includes workshop on High Pressure Mineral Physics using Synchrotron Radiation

Physics and Chemistry of Earth Materials

IUGG General Assembly Sapporo Japan (June 30 - July11)
Symposium JSS05: Physics and Chemistry of Earth Materials

Geosciences and Cosmoscience at High Pressure

Joint 19th AIRAPT - 41th EHPRG International Conference High pressure Science and Technology Bordeaux, France, July 7th - 11th 2003
Symposium S12: Geosciences and Cosmoscience at High Pressure.

Digital Library for Earth System Education

Digital Library for Earth System Education Annual Meeting, Boulder, CO, August 2 to 5, 2003

International Workshop on High Pressure Earth Science at Spring-8

International Workshop on High Pressure Earth Science at Spring-8, September 14-16, 2003

Special Symposium in Honor of Charles T. Prewitt

November 5-8, 2003

MSA/GSA Annual Meeting, Seattle, Washington.

Special Symposium in Honor of Charles T. Prewitt convened by Nancy Ross, Ross Angel and Russell Hemley.

Special Symposium in Honor of Don L. Anderson

December 7-12, 2003

Fall 2003 AGU Meeting, San Francisco, California

Special Symposium in Honor of Don L. Anderson convened by Raymond Jeanloz and Miaki Ishii.

A.4 COMPRES Membership

This consortium, which was founded in May, 2002, is committed to support and advocate research in materials properties of Earth and planetary interiors with a particular emphasis on high-pressure science and technology, and related fields. COMPRES, which derives its primary financial support from the National Science Foundation, is charged with the oversight and guidance of important high-pressure laboratories at several national facilities, such as synchrotrons and neutron sources. These have become vital tools in Earth science research. COMPRES supports the operation of beam lines, the development of new technology for high-pressure research, and advocates for science and educational programs to various funding agencies.

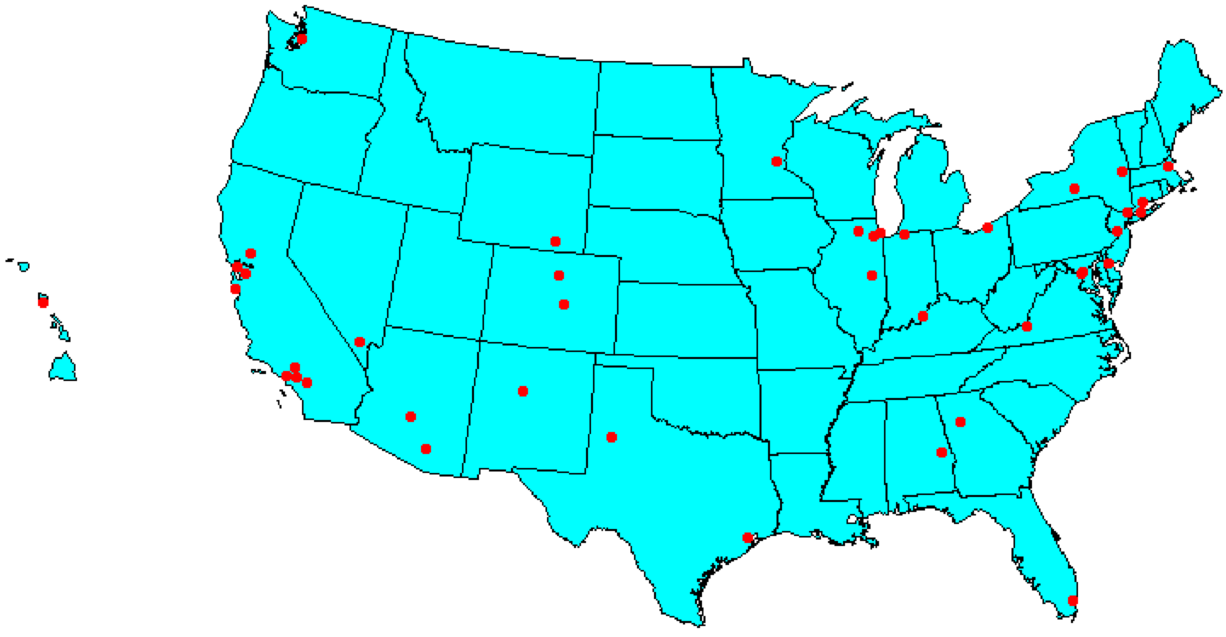
COMPRES is community based. Educational and not-for-profit US Institutions are eligible to become members, and each institution is entitled to one vote in the decision process. The membership defines policy and charts the future of the consortium. Other organizations and non-US institutions are eligible to be affiliated members with a non-voting representative to all COMPRES business meeting.

There are now 35 institutional members and 11 affiliated members of COMPRES. They are listed in the following table and indicated on the maps.

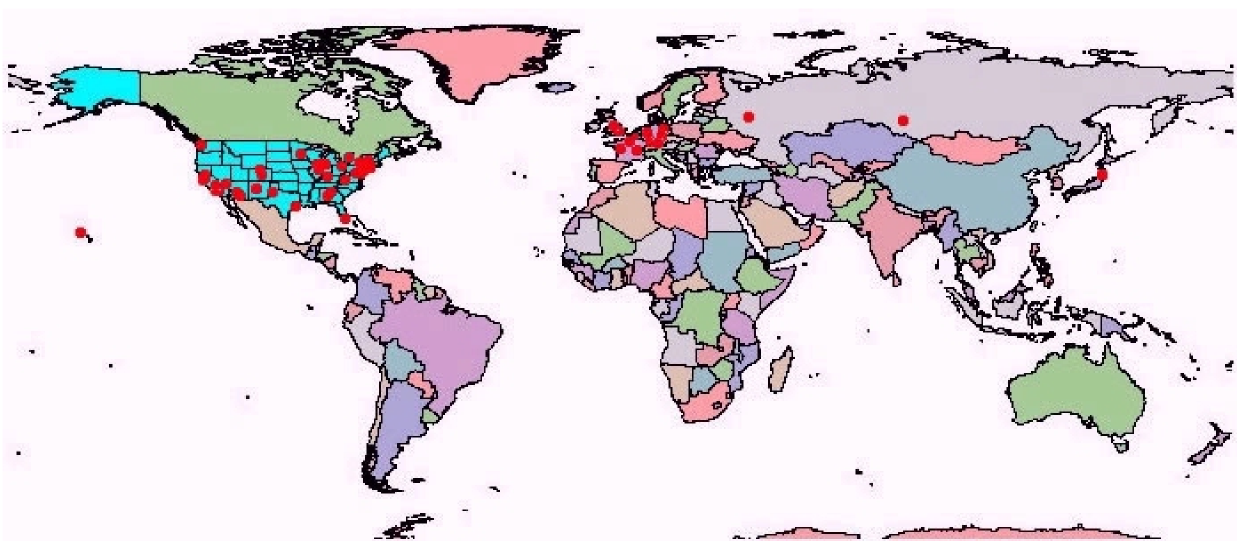
Institution	Elector	Alternate
Argonne National Laboratory	Wolfgang Sturhahn	Marcos Grimsditch
Arizona State University	Thomas Sharp	James Tyburczy
Astromaterials Research and Exploration Science, NASA	Kevin Righter	John Jones
Auburn University	Jianjun Dong	
Azusa Pacific University	Donald G. Isaak	
Carnegie Institution of Washington	Ronald E. Cohen	Yingwe Fei
Case Western Reserve University	James A. Van Orman	Nancy Chabot
Colorado College	Phillip Cervantes	
Columbia University	Dave Walker	Taro Takahashi
Cornell University	Chang-Sheng Zha	William Bassett

Delaware State University	Gabriel Gwanmesia	Al Sameen T. Khan
Ecole Normale Supérieure de Lyon	Jan Matas	
Florida International University	Surendra Saxena	Hexiong Yang
GeoForschungsZentrum Potsdam	Frank R. Schilling	
Georgia State University	Pamela Burnley	
Indiana University at South Bend	Henry P. Scott	Jerry Hinnefeld
Lawrence Livermore National Laboratory	Daniel L. Farber	William B. Durham
Max-Planck Inst. For Solid State Research	Paul S. Balog	
Northern Illinois University	Mark R. Frank	Jonathan Berg
Novosibirsk State University	Elena Boldyreva	
Princeton University	Thomas Duffy	Guust Nolet
Rensselaer Polytechnic Institute	Anurag Sharma	John Schroeder
Russian Academy of Sciences	Yuriy A. Litvin	
Stony Brook University	Michael T. Vaughan	John B. Parise
Texas Tech University	Yanzhang Ma	Valery Levitas
Universite de Poitiers (France)	Jacques Rabier	
Universite des Science et Technologies de Lille	Paul Raterron	
University College, London	David Dobson	
University of Arizona	Robert T. Downs	Michael Drake
University of California at Berkeley	Mark Bukowinski	Raymond Jeanloz
University of California at Davis	Charles E. Lesher	Alexandra Navrotsky
University of California at Los Angeles	Abby Kavner	Donald Isaak

University of California at Riverside	Harry W Green, II	Stephen Park
University of California at Santa Cruz	Quentin Williams	Elise Knittle
University of Chicago	Dion L. Heinz	Andy Campbell
University of Colorado at Boulder	Joseph R. Smyth	Hartmut Spetzler
University of Illinois	Jay D. Bass	Jie Li
University of Hawaii at Manoa	Murli H. Manghnani	Li Chung Ming
University of Louisville	George Lager	
University of Maryland at College Park	William J. Minarik	John A. Tossell
University of Minnesota	Renata Wentzcovitch	David L. Kohlstedt
University of Nevada at Las Vegas	Oliver Tschauner	Malcolm Nicol
University of Washington	Ann Chopelas	J. Michael Brown
University of Wyoming	Jeffrey Yarger	David Anderson
Virginia Polytechnical Institute and State University	Nancy L. Ross	Ross J. Angel
Yale University	Shun-ichiro Karato	David Bercovici



Locations of COMPRES Institutional Members in the United States. Move the mouse pointer over a dot to see the name of the institution. Click to link to the institution's web site.



Locations of COMPRES Institutional Members Throughout the World.

A.5 Information Technology and Communications

Web Site

Internet technology presents COMPRES with numerous options for implementing organizational services for its members and for developing an attractive and useful interface with the educational and public communities. For the mineral physics community, it can provide a centralized location for information on important events, job openings, detailed information on the organization and management of COMPRES, and streamlined systems for finding information, applying for facilities time and registering for events. It projects our organization to the world and is one of the first impressions we will make on people who are not familiar with COMPRES and its work. In order to realize the benefits that Internet technology makes possible, COMPRES has established a Web site with a new URL link address http://www/compres.us; al of the files related to the COMPRES website are still physically located on the http://www/compres.stonybrook.edu server and are being maintained by Glenn Richard and Michael Vaughan. At present, the COMPRES site provides the following information:

- A general overview of COMPRES
- COMPRES staff contact information
- Contact information for COMPRES the Facilities, Infrastructure Development and Executive Committees.
- Information about institutional and affiliate membership with application forms
- Links to synchrotron and neutron source web sites, including instructions for applications for beam time.
- Links to information on past and upcoming meetings.
- Publication lists for COMPRES and links to list for associated organizations [e.g., GSECARS].
- The quarterly COMPRES Newsletters.
- Education and Outreach.

The COMPRES Central Office envisions the future role of the web site as that of an electronic Central Office that supports all the functionality necessary to enable the Consortium to serve the community's research and educational needs. This includes automation of the entire process needed to apply to perform an experiment at a facility and for reporting on the experiment afterwards as well as the sharing of experimental results.

Other Electronic Information Technology Services

- **List servers:** The initial list server is now operational that reaches hundreds of the members of the COMPRES community. Additional lists will be established during the coming months that serve the broader high pressure community.

- **People database:** Contact information for people involved in COMPRES. This will be made available online through a browser-based form
- **Online Forms for meeting registration:** Plans are in place to offer online registration for meetings and workshops beginning in the summer of 2003.
- **Videoconferencing:** The Central Office has acquired a host bridge to provide support for video conferences of the Executive Committee, the two Standing Committees, and other uses of the COMPRES community.

Quarterly Newsletters

Starting in November 2002, COMPRES publishes a quarterly newsletter with information and announcements of interest to the COMPRES community, in the broadest sense.

The 2003 issues have included special reports from the Community Facilities and Infrastructure Development projects overseen by COMPRES; reports of Workshops, Conferences and the COMPRES Annual Meetings; reports from university laboratories associated with COMPRES activities; and Messages from the COMPRES leadership.

These newsletters are edited by Jiuhua Chen and may be found on the COMPRES web site at http://www.compres.stonybrook.edu/Newsletter/.

In addition to a column in the quarterly COMPRES newsletter, the new President of COMPRES [Robert Liebermann] has sent a Monthly Message to the COMPRES community using the listserve distribution, beginning in October 2003. The purpose of these monthly messages from the President is to keep the COMPRES community informed of recent developments as well as activities of the Executive and Standing Committees. These Monthly Messages are also sent to the Program Directors of the Division of Earth Sciences at the NSF.

COMPRES Exhibition Booth at Fall 2003 AGU Meeting

At the Fall 2003 Meeting of the American Geophysical Union in San Francisco in December 2003, COMPRES had a special booth in the Exhibition Area. This exhibition booth was jointly sponsored by GSECARS and COMPRES, and attracted lots of visitors.. Jiuhua Chen and Ann Lattimore created the materials for the booth based on input provided by the Community Facilities and Infrastructure Development projects. Michael Vaughan helped in staffing the booth, in cooperation with Nancy Lazarz and Mark Rivers of GSECARS.A visual summary of the COMPRES exhibition booth from the Fall 2003 AGU Meeting may be found at:
http://www.compres.stonybrook.edu/Newsletter/V2N4/Newsletter2_4.pdf.

A.6 COMPRES Publications

Papers of COMPRES

Ablett, J. M., Kao, C. C., Shieh, S. R., Mao, H. K., Croft, M. & Tyson, T. A. High-pressure x-ray near-edge absorption study of thallium rhenium oxide up to 10.86 GPa. *High Pressure Res. 23*, 471-476, 2003. – Ruby system, optical

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Kanani K. M. Lee (2003) Exploring Planetary Interiors: Experiments at Extreme Conditions (UC Berkeley, Earth and Planetary Science). advisor Raymond Jeanloz,

Li, Li, (2003) Rheology of olivine at mantle pressure, Stony Brook University (Advisor: Donald Weidner)

Speziale, Sergio (2003) Princeton University (Advisor: Thomas Duffy)

A.7 Education and Outreach

During two past two years, COMPRES has worked with organizations, such as the Digital Library for Earth System Education (DLESE) and EarthScope, in order to promote inquiry-based education and outreach as a nationwide collaboration between scientists, educators, materials developers, government agencies and other stakeholders. As a member of the Planning Committee for the 2002 and 2003 annual meeting of DLESE, Glenn Richard coordinated the organization of a set of skills workshops that are offered during the first day of the meeting. These workshops have been aimed at introducing educators, researchers and materials developers to data and visualization tools that can be used with a diversity of target audiences in a wide variety educational settings. Many of these tools enable students to develop hypotheses about the Earth's systems and test them by working with real data, such as that collected by the EarthScope project.

In order to advance the practice of using digital tools to work with real Earth systems data, COMPRES is promoting the use of geographic information systems (GIS) and other analysis tools in educational settings. The DLESE skills workshops have promoted this strategy on a national level. Glenn Richard is a member of the Advisory Board for the development of a new high school environmental science textbook and CD-ROM by the American Geological Institute. This set of materials, which will be used nationally, is being designed to offer hands-on inquiry-driven activities based largely on the use of USGS data in ArcView GIS. Locally, at Stony Brook, Glenn Richard is co-teaching a graduate-level course on using GIS as a research and educational tool with Dr. Glenn Smith of the Department of Technology and Society.

In collaboration with the Department of Geosciences and the Department of Technology and Society at Stony Brook, COMPRES offers students of the the Brentwood Schools District an Honors Earth Science program modeled after Stony Brook's introductory environmental geology undergraduate course. During the summers, about 15 students from Brentwood engage in a four week residential program, emphasizing scientific methodology, research techniques and data collection in the field. During the following academic year, students work in teams to conduct research projects.

COMPRES, the Department of Geosciences, the Department of Technology and Society and the Center for Environmental Molecular Science at Stony Brook offer an honors Earth science course to students at Sayville High School, equivalent to Stony Brook's undergraduate introduction to physical geology. During the first year of the program, which runs over a two year cycle, lecture and laboratory components of the undergraduate course are incorporated into the honors course at Sayville. During the second year students complete a major research project that is carried out over the duration of the academic year.

Educational networks need to leverage their resources by working with teachers in order to reach large numbers of students. CEN 514: Long Island Geology is a professional development-level course designed for teachers, offered each fall, that explores processes that have governed the geological development of Long Island and other parts of New York State. Topics include mantle processes and their relation to plate tectonics, the tectonic history of New York State, local seismicity, the origin of local rocks, and a brief overview of current research in mineral physics and its relation to processes that have played a role in the geologic history of New York. In the fall of 2003, GEO 514 was taught by Glenn Richard, Steven Englebright, a University Adjunct who is a New York State Assemblyman, and Mirza Beg of the Center for Environmental Molecular Science. Each participant in CEN 514 is required to perform a research project or develop a lesson plan that is designed to familiarize secondary school students with Earth science as an investigative process.

Future Plans

Through its partnership with EarthScope, the Earth Science Educational Resource Center (ESERC), which performs education and outreach for COMPRES, plans to develop an Earth Materials Virtual Laboratory over the next two years, designed as a collection of interactive tools and activities that will enable students to explore the materials and processes of the Earth's interior and how they affect our planet's surface. This will include curriculum materials that utilize data being collected by EarthScope's Plate Boundary Observatory. On both a local and national level, COMPRES will continue to promote the use of GIS and other visualization and analysis tools as a means for students to explore phenomena that characterize the Earth's interior and surface.

A.8 Management and Organization

Executive Committee

Elected by Electorate

Members and affiliation (term of service)

- Donald Weidner, Chair, Stony Brook University (2002-04)
- Jay Bass, University of Illinoise (2003-06)
- Russell Hemley, Carnegie Institution of Washington (2002-04)
- Shun-ichiro Karato, Yale University (2002-05)
- Robert Liebermann, President (Current appointment 2003-07)

Previous Members:

- Thomas Duffy, Princeton University (2002-03)

Facilities Committee

Elected by Electorate

Members and affiliation (term of service)

- Mark Rivers, Chair, University of Chicago (2002-05). Chair (2003-05)
- Thomas Duffy, Princeton University (2003-06)
- Yingwei Fei, Carnegie Institution of Washington (2002-05)
- Harry Green, University of California at Riverside (2002-04)
- Charles Prewitt, University of Arizona (2003-06)

Previous Members:

- Michael Brown, University of Washington (2002-2003)
- Quentin Williams (Chair), University of California at Santa Cruz (2002-2003)

Infrastructure Development Committee

Elected by Electorate

Members and affiliation (term of service)

- James Tyburczy, Chair (2002-04)
- Pamela Burnley, Georgia State University (2002-05)
- Kevin Righter, NASA Astromaterials Laboratory (2003-06)
- Nancy Ross, Virginia Tech University (2003-06)
- Yanbin Wang, University of Chicago (2002-05)

Previous Members

- David Walker, Columbia University (2002 – 2003)

Raymond Jeanloz, University of California at Berkeley (2002 – 2003)

ByLaws Committee

At the COMPRES Annual Meeting in Santa Cruz in June 2003, the Electorate passed the following resolution:

> "A by-laws committee will be established to recommend changes in the by-laws. Timetable will be Oct 10 to Nov 1 for circulating to the COMPRES community. Target decision date is the 2003 AGU."

The following members were elected at 2003 Annual Meeting in Santa Cruz

Members

Ronald Cohen, Carnegie Institution of Washington

Bruce Buffett, University of Chicago

Charles Prewitt, University of Arizona

Joseph Smyth, University of Colorado

Lars Stixrude, University of Michigan

In October 2003, the Executive Committee asked that the ByLaws Committee self-organize and elect a Chair and begin its work; the ByLaws Committee subsequently elected Ronald Cohen as Chair.   The ByLaws Committee was encouraged to consult widely the COMPRES community in formulating their recommendations for possible changes in the ByLaws, especially in view of Article XI, which stipulates that changes to the ByLaws requre the affirmative vote of two-thirds of the entire Electorate. The report of the ByLaws Committee is anticipated Feb 2004.

Following discussions and consideration of the proposed changes by the COMPRES community, the Electorate will be asked to vote (section by section) on the proposed changes, via a confidential balloting system using double sealed envelope, at the 2004 Annual Meeting [which we hope to hold in June]. For those Electors [or their Alternates] who will not be attending the 2004 Annual Meeting, they will be able to submit absentee ballots using the double-sealed envelope balloting system in advance of the Annual Meeting.

Advisory Committee

Members and affiliation (term of service)

Bruce Buffett, University of Chicago

Chi-chang Kao, Brookhaven National Laboratory

Guy Masters, University of California at San Diego

Richard O'Connell, Harvard University

Paul Silver, Carnegie Institution of Washington

Held informal meetings in Miami and Santa Cruz

Plan separate meeting in Spring 2004

Presidential Search

A search committee consisting of:

David Walker (Chair)

Raymond Jeanloz

Charles Prewitt

Nancy Ross

Donald Weidner

Conducted an international search, with advertisements in Science and EOS. They solicited applications, gathered and filtered the applications and transmitted two applications to the Executive Committee. The Executive Committee interviewed the two candidates and selected Robert Liebermann for the position, whose appointment began on September 1, 2003 for a term of four years.

We would like to take this opportunity to thank Jay Bass for his exemplary leadership and fine service that he rendered to COMPRES as its first President in 2002 – 2003.

Relationship to National Facilities

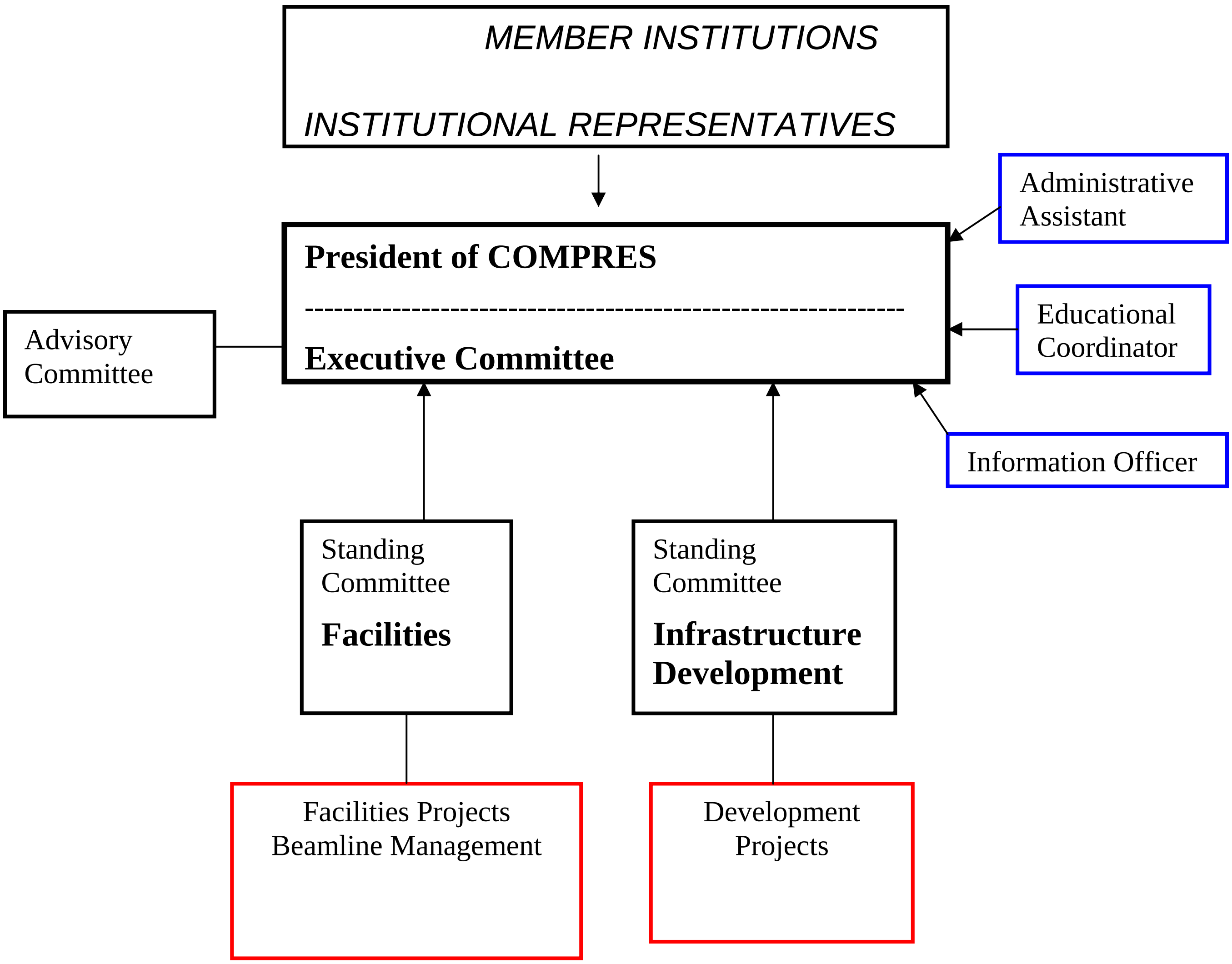
GSECARS COMPRES will review the high pressure facilities and assure highest service to the user community.

NSLS COMPRES funds Multi-anvil and Diamond-anvil facilities at NSLS. COMPRES will review the high pressure facilities and assure highest service to the user community.

ALS COMPRES funds Diamond-anvil facilities at ALS. COMPRES will review the high pressure facilities and assure highest service to the user community.

ORNL The COMPRES community has succeeded in obtaining DOE funding to build a high pressure facility at the Spallation Neutron Source that is now under construction. COMPRES will work to build the user community and assure access to this facility.

Organization of COMPRES Office



Operation of the COMPRES Central office:

The Central office of COMPRES is located at Stony Brook University in the ESS Building, along with the Mineral Physics Institute [MPI], which is directed by Donald Weidner.

The Central office staff includes Robert Liebermann, the President of COMPRES (from September 1, 2003) and Ann Lattimore, Administrative Assistant, both of whom are supported by the COMPRES Collaborative Agreement with the NSF.

The administrative operation of COMPRES is also supported by the following personnel who are employees of the Mineral Physics Institute of Stony Brook University: Jiuhua Chen, Research Associate Professor. COMPRES role: Editor of Newsletter Glenn Richard, Educational Coordinator: COMPRES role: Web Manager and Education/Outreach activities. Michael Vaughan, Research Associate Professor: COMPRES role: Manager of listserve and database.

Administrative Assistant for MPI: In February 2004, a new staff member will join the MPI as an Administrative Assistant to the Director. This person will work in collaboration with Ms. Lattimore to provide administrative support to COMPRES activities.

In addition to the MPI staff contributions, we have profited from the advice and logistical support of two staff members of the Department of Geosciences at Stony Brook: Owen Evans, Director of Laboratories, and Claire Ondrovic, Assistant to the Chair.

President's Narrative:

On September 1, 2003, I took up my new duties as President of the Consortium for Materials Properties Research in Earth Sciences [COMPRES]. In the first 16 months of its existence, COMPRES made major strides in achieving its objectives. I look forward to working with the Executive Committee and the COMPRES community to build on this initial progress and to continue to develop a shared vision and nurture existing programs and new initiatives over the next few years.

As part of of my first four months on the job, I paid visits all of the institutions which are responsible for overseeing or operating Community Facilities for COMPRES or where Infrastructure Development (ID) projects are being conducted under the auspices of COMPRES. The purpose of these visits was to learn about the current state of these operations and to discuss their future plans. I also included visits to related facilities operations [e.g., GSECARS at the Advanced Photon Source] and other experimental laboratories in mineral physics and chemistry.

Following is a list of these visits

Sept. 30: GeoSoilEnviro CARS [GSECARS] at the Advanced Photon Source (APS).

M. Rivers, S. Sutton, Y. Wang, G. Shen, P. Eng and colleagues.

Oct. 8: Diamond-anvil cell facilities at the National Synchrotron Light Source (NSLS).

H-k. Mao and R. Hemley.

Oct. 16: Geophysical Laboratory (Pressure calibration at high temperature project)

Y. Fei

Oct. 20-21: Virginia Tech (Neutron studies program and labs of N. Ross and R. Angel)

N. Ross for neutron studies.

Oct. 28: Princeton University (Laser-heating in DAC project)

T. Duffy.

Oct. 29: Multi-anvil facilities at the NSLS.

D, Weidner and M. Vaughan.

Oct. 30: Yale University-lab of S. Karato

Nov 3: University of Washington—labs of M. Brown and A. Chopelas.

Nov. 4: Synchrotron facilities-Advanced Light Source of the Lawrence Berkeley Lab

R. Jeanloz, E. Knittle, S. Clark

Nov. 17: University of Illinois at Urbana-Champaign (Brillouin spectroscopy project)

J. Bass

Nov. 19: University of Colorado (Absolute P & T project)

I. Getting

Nov. 20: Arizona State University (Multi-anvil cell development project)

K. Leinenweber and J. Tyburczy

On September 23, Don Weidner, Chair of the Executive Committee of COMPRES, and I visited the NSF Division of Earth Sciences [EAR] consult with Herman Zimmerman, Director of EAR and David Lambert, Program Director for Instrumentation and Facilties [IF], and other EAR Program Directors to discuss the current status and future plans for COMPRES.

On Oct. 17, Charlie Prewitt and I represented the Center for High Pressure Research [CHiPR] at a ceremony at the NSF honoring the directors of the Science and Technology Centers Class of 1991. In a brief presentation, we were able to show how the NSF investment in technological developments in the 1990s has led to the formation of the new consortium COMPRES. During that visit to NSF, I met with Dr. Hugh Van Horn, Director of National Facilities in the Division of Materials Research [DMR] to find out more details about a new program for major instrumentation projects within DMR. I had been alerted to this new funding opportunity by David Lambert during our visit to EAR on September 23.

On Nov 13, Don Weidner and I visited the Basic Energy Sciences offices of the DOE to meet with Nick Woodward, Helen Kerch and Pedro Montano, at the suggestion of David Lambert. We discussed the new changes in how the DOE proposes to operate their synchrotron and neutron facilities.

Within the past few months, many institutions have been approved by the Executive Committee for membership in COMPRES. We now have 38 U.S. institutions and 8 foreign affiliates. A complete list can be found on the COMPRES website: http://www.compres.stonybrook.edu, as well as in the Membership section [A4] of this report, where there also are US and world maps showing the location of these member institutions.

On Nov 1-5, I attended the Annual Meeting of the GSA and MSA in Seattle, Washington. Highlights included a symposium in honor of Charlie Prewitt organized by Nancy Ross, Ross Angel and Russell Hemley, which was a great success.

During the meeting, we all took pride in the award of special honors to our colleagues from Mineral Physics and Chemistry, including:
Charles Prewitt—Roebling Medal of the MSA

Guillaume Fiquet—MSA Award

George Harlow—Distinguished Service Award of the MSA

Michael Manga—Donath Medal of the GSA

In addition, at the Seattle meeting, Michael Carpenter of the University of Cambridge was installed as the new President of the Mineralogical Society of America.

The ByLaws Committee [Bruce Buffett, Ronald Cohen, Joseph Smyth, Charles Prewitt, and Lars Stixrude] has elected Ronald Cohen as its chair and has begun its work on behalf of the COMPRES community. As indicated earlier, the ByLaws Committee was encouraged to consult widely the COMPRES community in formulating their recommendations for possible changes in the ByLaws, especially in view of Article XI, which stipulates that changes to the ByLaws require the affirmative vote of two-thirds of the entire Electorate. Following discussions and consideration of the proposed changes by the COMPRES community, the Electorate will be asked to vote (section by section) on the proposed changes, via a confidential balloting system using double sealed envelope, at the 2004 Annual Meeting.

The major event of December 2003 for COMPRES was surely the Fall AGU Meeting in San Francisco. Highlights included:

Symposium in honor of Don Anderson's 70th birthday organized by Raymond Jeanloz and Miaki Ishii, which included many interesting oral and poster presentations. I had the pleasure of giving a talk on Don's role in nurturing and encouraging research in mineral physics.

Exhibition booth jointly sponsored by GSECARS and COMPRES, which attracted lots of visitors [despite its awkward location] and seemed to be appreciated. My thanks to Jiuhua Chen, Michael Vaughan and Ann Lattimore for their invaluable work in creating and staffing the booth, as well as to Nancy Lazarz and Mark Rivers of GSECARS for their cooperation.

We all took pride in the award of special honors to our colleagues in Mineral and Rock Physics (MRP), including:

David Kohlstedt—Harry Hess Medal

New AGU Fellows: William Durham, Eiji Ito, Ian Jackson and Taro Takahashi (who was honored for his contributions to ocean chemistry, but whom many know as one of the pioneers in mineral physics research using the diamond-anvil cell).

The Physical Properties of Earth Materials group once again organized a fantastic dinner celebration at Destino's, the critically acclaimed Latin American restaurant in the Castro district. Our congratulations to Brian Bonner and Bill Durham for discovering such a wonderful venue for this special evening.

The Committee on Mineral and Rock Physics hosted a wine and cheese reception, during which Annette Kleppe of Oxford University received the Outstanding Student Award in Mineral and Rock Physics for 2003.

We also had meetings with David Lambert, the NSF Program Director who oversees the COMPRES initiative; one of these meetings included Harry Green to discuss a proposed plan to acquire and operate a focused ion-beam microscope for the rock and mineral physics community.

The COMPRES Standing Committees held luncheon meetings to discuss the annual reports on the Infrastructure Development projects and Community Facilities. The Executive Committee met for breakfast on Dec 11 to begin the planning process leading to the submission of the Annual Program Plan and Budget request to the NSF on February 1, 2004. See details of this process in Section A9 of this report.

2004 Annual Meeting

The dates for the 2004 Annual Meeting have been tentatively set for June 20-21-22 [Sun-Mon-Tues]; alternative dates are June 17-18-19 [Thurs-Fri-Sat]. We have deliberately tried to avoid conflicts with other known meetings of potential interest to members of the COMPRES community [e.g., the IRIS annual meeting, the Gordon Research Conference on High Pressure, etc]. Details on the site for this meeting will soon be announced to the COMPRES community and associated organizations and colleagues.

A.9 Annual Program Plan and Budget

In preparation for the submission of the Annual Progress Report and Annual Program Plan and Budget to NSF in February, 2004, the Executive Committee developed a process that involved the COMPRES community and the two elected Standing Committees for Community Facilities and Infrastructure Development Projects.

In September 2003, the two Standing Committees asked the project directors of each of the subawards to submit annual progress reports for Year #2 and budget requests for Year #3 by November 15, 2003. The Infrastructure Development Committee also issued a call to the COMPRES community for proposed new initiatives for technological projects that would contribute to the COMPRES mission, with a deadline of November 1, 2003.

Following receipt of the requested information, the Standing Committees evaluated the progress reports and budget requests via a series of email exchanges and teleconferences, culminating in meetings of the Committees at the Fall AGU Meeting in San Francisco. Each of the Standing Committees gave oral reports on their deliberations to the Executive Committee at the Fall AGU Meeting, and then submitted their written report, with evaluations of progress and recommendations for funding in Year #3, to the Executive Committee. In the case of the Infrastructure Committee, this report included recommendations for initial funding of new projects and community workshops.

In January 2004, the Executive Committee met via video and teleconference on three occasions to discuss the reports of the Standing Committees and to formulate recommendations for an Annual Program Plan and Budget for Year #3. Following these meetings, the President prepared a budget plan which was discussed, revised, and approved by a majority of the Executive Committee.

B COMMUNITY FACILITIES

B.1 Diamond-anvil cell facilities at the National Synchrotron Light Source

[H-k. Mao, R. Hemley, Carnegie Institution of Washington]

Scientific highlights

Discovery of Two New Post-Spinel Minerals in a Shock-Metamorphosed Chromite Grain in Suizhou Meteorite -- Forty years ago, in search for denser polymorphs of the then newly discovered silicate spinel (ringwoodite) and modified spinel (wadsleyite) that are stable at the pressure and temperature (*P-T*) conditions of the Earth's transition zone, Ringwood proposed orthorhombic $CaFe_2O_4$-type (CF) and $CaTi_2O_4$-type (CT) structures as the top candidates for "post-spinel" transitions in the Earth's mantle. Although ferromagnesian silicate spinels were later found to break down to simple oxides or stishovite plus perovskite, several post-spinel oxides convert to a single phase with the CF or CT structure. Neither dense post-spinel polymorphs nor silicate perovskites, however, have been discovered in nature. Using the x-ray diffraction (XRD) microprobe technique at X17C, Chen *et al*.discovered a CF-type [*Chen et al.*, 2003c] and a CT-type polymorphs [*Chen et al.*, 2003b] of chromite composition in a shock-metamorphosed chromite in Suizhou meteorite. Using laser-heated diamond-anvil cell and XRD at X17C, they demonstrated that both CF and CT are indeed quenchable polymorphs of chromite formed above 12.5 and 20 GPa, respectively. The two post spinels and the unaltered chromite show an astonishing example of three polymorphic zones spanning a very wide pressure range (equivalent to the conditions of upper mantle, transition zone and lower mantle) all within a single chromite grain. With the ubiquitous presence of chromite, the CF and CT phases may be among the important index minerals for natural transition sequence in mantle rocks and meteorites.

Elastic Anisotropy and Rheology of Hydrous and Anhydrous Ringwoodite --Abby Kavner performed radial X-ray diffraction experiments with OH-bearing (hydrous) ringwoodite compressed uniaxially in a diamond anvil cell. The X-ray beam was 10 µm x 15 µm. The result supports a differential stress that increases from 2.9 to 4.5 GPa over the pressure range of 6.7-13.2 GPa at room temperature. This result suggests a significant water weakening effect when compared with results from similar experiments on the anhydrous counterpart [*Kavner and Duffy*, *Geophys. Res. Lett.* **28**, 2691 (2001)]. The elastic anisotropy ($=2C_{44}/(C_{11}-C_{12})$) of hydrous ringwoodite is measured to be 0.87(7) throughout this pressure range, similar to measured values for anhydrous ringwoodite. These results suggest that hydrous minerals in the upper mantle and transition zone may have higher ductile strain rates for a fixed shear stress at high temperature, resulting in stronger preferred lattice orientation. This, in turn, may be seismically detectable, which opens the possibility of using seismic anisotropy as a marker for local volatile-containing areas within the upper mantle and transition zone.

High-Pressure Phase Transition and Hydrogen Disordering in Gibbsite --

Haozhe Liu *et al*. studied gibbsite $Al(OH)_3$ with XRD at room temperature up to 53 GPa, respectively. A phase transition was confirmed at about 2.5 GPa. The high- pressure phase is indexed as an orthorhombic structure, rather than a triclinic structure as reported in previous studies. It is quenchable to ambient conditions, and the unit cell parameters of the new quenched phase was a = 8.690 Å, b = 5.044 Å, c = 9.500 Å, and its unit cell volume was 416.4 $Å^3$, which was about 2 % smaller than the unit cell volume of gibbsite at ambient conditions. The second order Birch EoS fitting for the high pressure phase yields bulk modulus of 75 ± 2 GPa based on the assumption of K_0' = 4. The high-pressure phase also showed partial disordering as diffraction peaks broadened. To understanding the broadening, they also performed *in-situ* high-pressure infrared absorption spectra experiments at beamline U2A. From its broadened IR vibrational modes above 15 GPa, while the Al-O substructure still kept stable from the corresponding XRD data. A gradual disordering of hydrogen substructure above 15 GPa in a quasi-hydrostatic compressing is suggested. The disordering of hydrogen may induce a small amount of local disordering in the Al-O basic structure, but is insufficient to drive the system to complete amorphization under compression. The further broadening of XRD patterns above 30 GPa demonstrated some extent the disorder distribution of the Al-O substructure, but they still remained the “crystalline” instead of complete amorphization within the experimental range up to 53 GPa.

Discovery of New High Pressure Phase of Ice by Far-IR Spectroscopy --New far-IR measurements were carried out by Klug *et al*. to test proposed pressure-induced phase transformations in H_2O and D_2O ice VIII at low temperatures up to 20 GPa and compared with the results of a series of first-principle studies for this phase transformation. The beamline capability expanded into the new far-IR region at low-T and high-pressure enables us to study the pressure and temperature dependence of the expected far-IR active mode and complement the *ab initio* linear response phonon studies. Surprisingly, both low-T high-pressure far-IR and theoretical calculation results give a different interpretation regarding the phase transformation compared with neutron scattering studied at high pressure. Further studies of various ices continue with new phases being uncovered by these methods at extreme conditions such as far-IR studies above 100 GPa at variable temperatures.

High P-T Phase Diagram of Nitrogen -- The existence and characterization of a distinct class of molecular phases of solid nitrogen at high pressures and temperatures have been studied by Raman scattering, IR spectroscopy, and x-ray diffraction methods by Gregoryanz *et al*.. The most remarkable change is a phase (*q*), which is characterized by strong intermolecular interactions and infrared vibron absorption. A second lower-pressure phase (*i*) is diatomic with orientationally equivalent molecules. Both phases can be quenched to room temperature and are observed over a wide *P-T* range from 20 to 100 GPa and 30 to 1000 K. The results suggest a major revision of the phase relations of nitrogen at high pressures and temperatures.

High Pressure Phase Transition and Partial Dehydration of Gypsum -- Gypsum ($CaSO_4$–$2H_2O$) is one of the most common sulfate minerals, forming in a variety of environments including hydrothermal vents near mid-ocean ridges, diagenetically altered marine sediments, and evaporate deposits. It contains both molecular water and molecular-like sulfate groups ionically bounded to calcium ions. Recent high-pressure Raman and IR studies give different interpretations compared with synchrotron energy dispersive x-ray diffraction (EDXD) studies regarding the phase transition ~5 GPa as well as the pressure-induced evolution above 5 GPa. Liu *et al.* used different techniques including synchrotron far-IR spectroscopy and Raman scattering at U2A beamline, EDXD and angle dispersive x-ray diffraction (ADXD) at X17C beamline to study the phase transitions. The synchrotron far-IR spectra confirmed the pressure-induced phase transition in gypsum around 5 GPa based on the changes of IR lattice vibrational modes and frequency discontinuities of these modes with increasing pressure. ADXD studies further confirmed this phase transition from monoclinic to orthorhombic symmetry around 5GPa. Both IR and ADXD showed that this high-pressure is fully reversible. It also revealed that the white synchrotron radiation induced dehydration due to the intrinsic features of this high-pressure phase in gypsum during the EDXD measurements. This quenchable high-pressure phase has been studied by IR and Raman as well and is determined as following: CaSO4□2H2O? high pressure phase (orthorhombic ~5GPa)? CaSO4□0.8H2O

High-Pressure Infrared Absorption Spectroscopy of Katoite Hydrogarnet – Lager *et al.* studied a D-rich katoite hydrogarnet, $Ca_3Al_2(O_4D_4)_3$ up to 10 GPa by infrared absorption. Discontinuities in both O-H and O-D vibrational frequencies at ~5 GPa suggest a pressure-induced phase transition that is in excellent agreement with the single crystal x-ray diffraction studies at high pressure. Pressure dependence of the frequencies related to the O-H stretching vibrational modes is consistent with the calculated results and indicates that deuteration does not significantly affect the pressure of the transition..

Development of Moissanite Anvil High-Pressure Cells – J. Xu, H. Scott and co-workers performed synchrotron IR studies of SiC-Moissanite to assess the utility of the moissanite (6H-SiC) anvils for high-pressure infrared spectroscopic measurements. A maximum pressure of 48.2 GPa was achieved by using moissanite anvil cells (MAC) and KBr pressure medium. In the region of characteristic phonon absorption of diamond, the signal to noise ratio measured with 4 mm thick moissanite anvils is 5~10 times better than that of diamond anvils. No significant change of the transmission spectra through the moissanite anvils was observed up to 48 GPa. This opens a new window for many studies in which the optical region of interest is normally obscured by diamond anvils.

Numerous Research Projects Carried out by Different Groups at U2A beamline

- Synchrotron IR (at both U2A and U2B beamlines) and Raman scattering studies of metamorphic microdiamonds (Harry Green's group, University of California, Riverside).
- New phases of N_2O_4 , and N_2O at high pressures and high temperatures were characterized by synchrotron IR and Raman spectroscopy (Y. Song et. al, Harvard University and Geophysical Laboratory)
- Characterization and imaging studies of CVD diamonds (Yan, Geophysical Lab.)

- High-pressure synchrotron IR and Raman spectroscopy of TiO_2 nanotubes (D. He et. al., LANL).
- New phase of aluminum hydroxide studied by synchrotron x-ray diffraction, IR and Raman spectroscopy (H. Liu et. al, SUNY, Stony Brook);
- FTIR technique detection of trace amount of H_2O and hydroxyl in a number of hydrous minerals such as olivine, $MgSiO_3$ perovskite and Al-perovskite samples quenched from high pressure and high temperature (groups from SUNY, Stony Brook, and University of Illinois, Urbana-Champaign).
- Pressure-induced structural changes in silicate glasses and glass-ceramic composite (K. Lipinska-Kalita, Univ. of Wyoming; M. Kruger, University of Missouri, Kansas City).
- Chemical reactions in simple planetary fluids were studied at high pressures and temperatures by infrared spectroscopy (J. Zaug and A. Goncharov, Livermore).
- Pressure-induced transitions in metal carbonyls (with R. Angel and B. Hanson, Virginia Tech).
- Far IR spectra of lawsonite and talc were measured under pressure to constrain thermodynamic properties (H. Scott, now at Indiana University South Bend).

X17B3 -- New Station Development Milestones:

The structure of X17B3 hutch has been completed.

The Goniometer, which was used in X17B1, was removed into the new hutch X17B3.

1. Apr. -- May of 2003:

- Established VME electronic system instead of the E500 system, which was used in X17B1 hutch.
- More than 50 hutch's cable was made, and later testing results showed that the shielding cable was better for avoid the cross-talk between motors drivers.

2. June of 2003:

- With Mark Rivers's help, the epics control software system was set up
- A Linux Server was connected to X17B3 as an operating computer.

3. July of 2003:

- Energy dispersive x-ray diffraction (EDXD) spectra of Au and Pt were collected for the first time in the new station.
- A used YAG laser heating system was moved from Geophysical Lab, Washington, DC to BNL.

4. August of 2003:

- A new beam slit was ordered from Advanced Design Consulting Company.
- The top optical table was setup for the YAG laser heating system for the two level's design, and the x-ray slits table was strengthen by adding two more legs.

5. Sept. – Oct. of 2003:

- Glass KB mirrors were tested for the white x-ray beam focusing. The mirrors were borrowed from Peter Eng, APS, since our Si mirrors is not ready yet.
- With a pair of the glass KB mirror the x-ray spot size can be focused as 20_ µm x 20 µm from the 70 µm vertical beam and 150 µm horizontal beam respectively. And the intensity of x-ray at the focusing point is 10 times more than the one without mirrors.

- The beamline of X17B3 is ready for users to perform their EDXD experiments.
- The optic mirrors of the YAG laser-heating system has been setup and ready for the optical element alignment. We are working for the laser safety approval with NSLS safety officers.

Users (including Collaborating Groups)

X17C:

U. Alabama --N. Velisavievic Caltech-- I. Halevy, A. Papandrew, A. Yue Carnegie Institution-- J. Lin, J. Shu, Y. Song, J. Xu Chinese Academy of Sciences--M. Chen Columbia U.-- D. Walker Harvard U.-- I. Silvera, S. Rekhi, A. Chijioke Lawrence Livermore Nat. Lab.--H. Cynn, M. Lipp, B. Baer, W. Evans, J. PattersonLos Alamos Nat. Lab.--D. He, G. Chesnut, D. Dattelbaum, B. Streetman U. Missouri Kansas City-- M. Kruger U. Wyoming – K. Lipinska-Kalita National Synchrotron Light Source-- Q. Guo, J. Hu, H. Liu, Z. Liu Princeton U.-- S. Shieh, S. Spezials, T. Duffy, B. Keifer, A. Batchelor SUNY at Stony Brook-- H. Liu, J. Chen, J. Kung Texas Tech--Y. Ma

U2A:

Albert Einstein College of Medicine –N. S. Marinkovic
R. Jackson-- Bruker Optics UCLA –A. Kavner, M. Weinberger (rescheduled) UC Riverside—H. Green, L. Dobrzinetskaya Carnegie Institution—Y. Song, E. Gregoryanz, J. Xu Colorado College --P. Cervantes, K. Chynoweth, T. Atkinson University of Illinois –J. Bass, J. Jackson University of Indiana South Bend – Henry Scott University of Louisville-- George A. Lager University of Michigan-- Wendy Panero (rescheduled) University of Wyoming—K. Lipinska-Kalita Livermore National Laboratory – J. Zaug, A. Goncharov National Research Council of Canada --D. Klug, J. Tse, W. Davidson National Synchrotron Light Source--L. Miller, M. Ruppel SUNY at Stony Brook--L. Li, L. Wang, D. Weidner, H. Liu, J. Kung Virginia Tech – C. Slebodnick, J. Zhao, R. Angel, B.E. Hanson

Future Beamline Upgrade Plans (not included in present budget)

X17B3:

To upgrading YAG laser and it's temperature measurement.
To establish angle dispersive x-ray diffraction (ADXD) facility.
To establish micro x-ray diffraction facility.

U2A:

In order to further improve the far-IR performance, we are going to install a beam compressor into the new pipe system. This will be done together with NSLS.

The current vacuum IR microscope system has been used for several years but limited to far-IR transmission experiment. We designed and will build a new Raman/vacuum IR microscope system with multi-functions covered the whole IR range. With the new system, IR transmission/reflection as well as Raman scattering can be easily done for same sample at same pressure. The water vapor absorption will be removed completely and the S/N ratio in these particular regions can be improved by factor of 2-5. All key functions will be controlled through a computer and user friendly.

We propose to build a side station close to the U2 port. The distance from the spot of synchrotron source to the IR system will be only about 3 meters. This will avoid any potential beam divergence and image distortion. Significant improvement of spatial resolution is expected (as U10B) and experiments at extremely high pressure with very small sample (5 micron) can be done at this side station. We also propose to develop high pressure and high temperature FTIR capability with this new side station. As first step, we are going to build an external heating system for symmetric DAC or MAC up to 1000 K. Taking the advantage of the synchrotron bright source, an aperture with typical size of 100200 μm^2 will be used to get decent FTIR spectra while the thermal radiation signal from the sample can be dramatically reduced by the aperture. As second step, we will develop combined laser heating and IR emission technique at high pressure and temperature above 1000 K.

2003 NSLD-DAC Publications

(The following publications are resulted, solely or partially, from using the x-ray, IR, or optical systems of the NSLS-DAC facility as noted.)

Ablett, J. M., Kao, C. C., Shieh, S. R., Mao, H. K., Croft, M. & Tyson, T. A. High-pressure x-ray near-edge absorption study of thallium rhenium oxide up to 10.86 GPa. *High Pressure Res. 23*, 471-476, 2003. – Ruby system, optical

Chen, B., D. Penwell, J.H. Nguyen, and M.B. Kruger, High Pressure X-ray Diffraction Study of Fe_2B, *Solid State Comm.*, in press, 2003. –X17C

Chen, M., J. Shu, H.K. Mao, X. Xie, and R.J. Hemley, Natural occurrence and synthesis of two new post-spinel polymorphs of chromite, *Proc. Natl. Acad. Sci. 100*, 14651-14654, 2003. –X17C

Chen, M., J. Shu, X. Xie, and H.K. Mao, Natural $CaTi_2O_4$-structured $FeCr_2O_4$ polymorph in the Suizhou meteorite and its significance in mantle mineralogy, *Geochim. Cosmochim. Acta 67*, 3937-3942, 2003. –X17C

Chestnut, G.N., B.D. Streetman, D. Schiferl, R.S. Hixson, W.M. Anderson, M. Nicol, and Y. Meng, Static X-ray diffraction study of cerium: The standard approach & the magic-angle approach, in *13th APS Conference on Shock-Compression of Condensed Matter*, edited by M.D. Furnish, American Institute of Physics, Argonne, IL, 2003. –X17C

Cui, C., T.A. Tyson, Z. Zhong, J.P. Carlo, and Y. Qin, Effects of pressure on electron transport and atomic structure of manganites: Low to high pressure regimes, *Phys. Rev. B 67*, 104107, 2003. –ruby pressure calibration system, optical

Errandonea, D., M. Somayazulu, and D. Hausermann, Phase transitions and amorphization of $CaWO_4$ at high pressure, *Phys. Stat. Sol. 235*, 162-169, 2003. –X17C

Gregoryanz, E., A.F. Goncharov, R.J. Hemley, H.K. Mao, M. Somayazulu, and G.Y. Shen, Raman, infrared, and x-ray evidence for new phases of nitrogen at high pressures and temperatures, *Phys. Rev. B 66*, 224108, 2002. --U2A, X17C

Guo, Q., H.K. Mao, J. Hu, J. Shu, and R.J. Hemley, The phase transitions of CoO under static pressure to 104 GPa, *J. Phys.: Condens. Matter 14*, 11369-11374, 2002. –X17C

He, D., Y. Zhao, T.D. Sheng, R.B. Schwartz, J. Qian, K.A. Lokshin, S. Bobev, L.L. Daemen, H.K. Mao, J.Z. Hu, J. Shu, and J. Xu, Bulk metallic glass gasket for high pressure, in situ x-ray diffraction, *Rev. Sci. Instrum 74*, 3012-3016, 2003. –X17C

He, D., Y. Zhao, H. Xu, Y. Zhang, Z. Liu, H.K. Mao, J. Shu, J. Hu, and R.J. Hemley, Behavior of TiO2 nanotubes under high pressure, *Second COMPRES Annual Meeting, Santa Cruz*, 2003. --U2A, X17C

Hemley, R.J., Z. Liu, E. Gregoryanz, and H.K. Mao, Infrared and Raman microspectroscopy of minerals under pressure, *Microscro. Microanal. 9*, 1098, 2003. --U2A

Hemley, R.J., Z. Liu, and H.K. Mao, New developments in high-pressure synchrotron Infrared spectroscopy, in *Proceedings of the International Workshop on Infrared Microscopy and Spectroscopy with Accelerator Based Source*, Lake Tahoe, 2003. --U2A

Hemley, R.J., and H.K. Mao, New windows on earth and planetary interiors, *Mineral. Mag. 66*, 791-811, 2002. --X17, U2A, optical

Hemley, R.J., and H.K. Mao, Overview of static high pressure science, in *High Pressure Phenomena (Enrico Fermi Course CXLVII)*, edited by R.J. Hemley, pp. 3-40, IOS Press, 2002. --U2A, X17C, optical

Hemley, R. J. and H. K. Mao, New findings in static high-pressure science, in *Shock Compression in Condensed Matter – 2003*, edited by M. Furnish *et al*., in press. U2A, X17C, optical

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B.2 Multi-anvil Cell Facilities at the National Synchrotron Light Source

[D. Weidner, M. Vaughan-Stony Brook University]

The 2003 year has been an exciting year at the multianvil facility at the NSLS. The new X-ray hutches were constructed, all plumbing, electrical wiring, interlock systems were installed. Then the press was installed with a new hydraulic pumping system and interfaced to the X-ray system. Our last beamtime in the old hutch was in October, 2002. We began new experiments in the new hutch in June, 2003. First, they were development experiments, but now we are fully operational. The new hutch construction for the B2 (COMPRES multianvil hutch) and B3 (COMPRES diamond anvil hutch) was a joint effort by Brookhaven National Lab – NSLS and CHiPR (Stony Brook and Geophysical Lab). This project was funded as follows:

Stony Brook University:	$50,000
CHiPR:	$160,000
NSLS:	$1,440,000

We have developed the associated floor space outside of the hutch. This included the operations region, just outside the hutch, attachment of room 142 on the floor plan below, a half lab across the hall but near the operations region, and an office space in a building next door to the NSLS. Room 142 is for sample assembly, the room across the hall is for sample cell building, and the office is to provide a place for visitors to work on data reduction.

We have begun a new operations mode that is facilitated by the hutch design. While the B3 hutch – the Diamond Cell hutch – is in operation, we can operate in 'parasitic' mode. We have installed a shutter between the two hutches. Thus, they can close the shutter and enter their hutch without interrupting our beam. However, we cannot enter our hutch without shutting down the diamond cell beam. During parasitic mode, we agree to not enter our hutch except during beam refill time at 7:00 am and 7:00 pm. Other entries are with permission and designed as to not interfere with their experiments. This mode is much more restrictive than standard mode where we have control of the up stream shutter and should be reserved for routine experiments and experiments where we are willing to wait for a entry time should something go wrong. This should be mostly used by local researchers, who do not have strict airplane schedules to keep. Nonetheless, this mode seems to be working and effectively doubles the usable beam time for the multianvil system.

This year we have changed our review process for beamtime allocation. Now all experiments must be submitted to NSLS review panel using the NSLS review process for general users. NSLS then assigns up to 90% of the standard mode beamtime on the basis of their review. We reserve 10% for beamline development. We schedule the standard mode time and the parasitic mode time with consideration of the NSLS assignment and the special considerations for the parasitic time.

In June of 2003, we switched to a completely proposal based assignment of beamtime. All users were asked to submit proposals to the NSLS Proposal Review Committee (PRC). For the two scheduling periods covered (May - August and September-November), a total of 21 proposals or beamtime requests were received, of which 16 were unique and 5 were renewals. Of these, 9 were granted times ranging from 1 to 7 days, with a total of 27 days assigned. 37 days of beamtime were assigned to X17B2, so we scheduled the 27 days worth of general users' projects to those 37 days. An additional 16 days were assigned to X17B3, during which time we can use the beam in parasitic mode, as described above. We were therefore able to assign 26 days (16 shared B3 days plus 10 B2 days) to the 7 proposals which were not given time by the PRC.

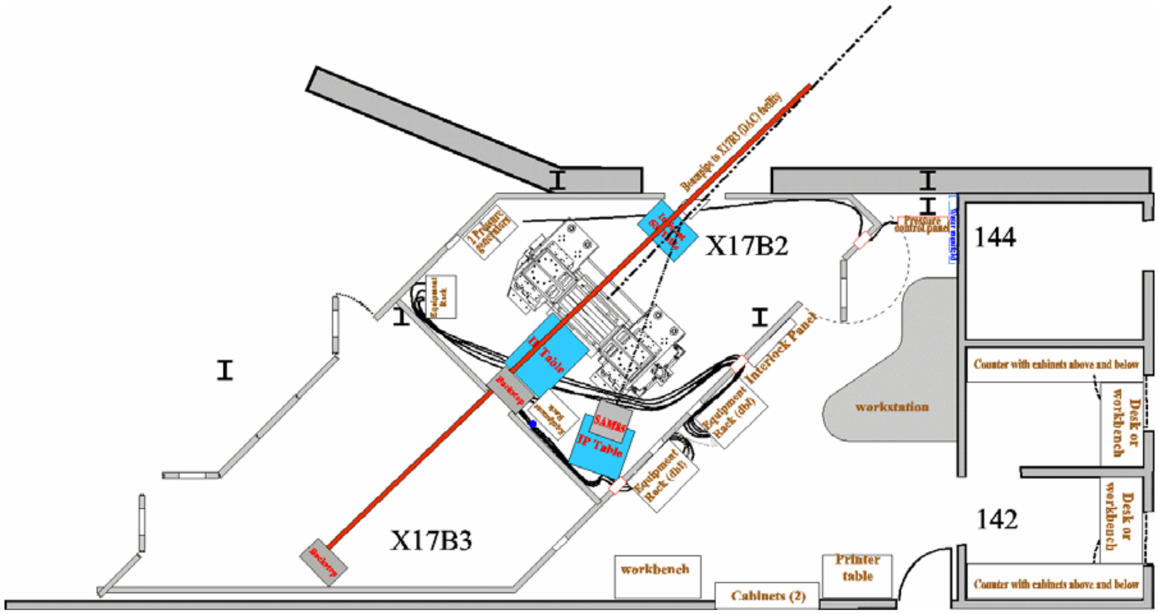
Because the August blackout shut down the Light Source for several days during one of our runs, we lost some part of one run, and all of another. As a result, one user lost his entire assigned time and another's time was reduced from 2 days to 22 hours. The first user was unable to come back at another time. The second user's experiment was at high pressure at 4:09 PM when the blackout occurred.

When power came back on, the wiggler had to be restarted, and then the beam was given to X17B1 which had general users scheduled. We were unable to gain access to the beam to continue the run until September 9. The press was kept at high pressure that entire time, losing only about 20% of its oil pressure!

Plans for the future include building a side station within the B2 hutch using a single bounce monochromator to send a side beam to a second press. We envision this press to be a light weight press equipped with a T-10 cell assembly. It will be useful to have a full time monochromatic station running simultaneous with the white beam system. We are now seeking funding for this system.

With the completion of the side station and the full commissioning of the parasitic mode, we expect that we will effect a net growth of the multianvil beam time from 25% of the total synchrotron operations to 100%.

We will soon take possession of a DDIA that will be used for deformation experiments. This system is part of our long term push to include rheology experiments at synchrotron facilities. We feel that this opens the facility to a community that has not had a history using the synchrotron.



hutch layout

Science Highlights

- ***Deformation experimental technique breakthrough and scientific research:*** A new high pressure deformation apparatus D-DIA has been married to the synchrotron x-ray source. The new apparatus has typical cubic-anvil geometry with independent control of top and bottom rams. Therefore under high pressure and temperature, the top and bottom ram can advance or retreat independently to deform the sample. In conjunction with synchrotron x-ray, the sample stress and strain can be measured by x-ray diffraction and radiograph imaging. Multiple x-ray diffractions along different direction relative to the principal stress axis yield an accurate measurement of stress in the sample to 10-100 MPa, and correlation of strain-mark images on the radiograph provides a precise strain measurement to 10^{-4} – 10^{-3}. More remarkably, the technique avoids the uncertainty introduced during the deformation and friction modeling of the pistons in the high pressure cell..
- ***High pressure Rheology of olivine:*** Li Li defended her PhD thesis reporting the results of the DDIA. Olivine flow laws were studied at high pressure. The activation volume appears to be much lower than conventional wisdom had surmised. Agreeing with the extant diffusion literature, an activation volume less than 5 cc/mol appears to be the most likely. This has a big impact on our understanding of the flow processes within the mantle.
- ***Polycrystalline stress field:*** MgO data in the DDIA give new insights into the distribution of stress among the grain subpopulations. Understanding the manner that a polycrystal supports a complex stress field can give many insights into earth studies and material studies.
- ***Ultrasonic measurements of non-quenchable phases at high pressure and temperatures:*** Study of acoustic velocity in minerals has move forward to

measure lower mantle minerals and non-quenchable phases at the beamline. Challenges have been made to obtain acoustic data of $MgSiO_3$ perovskite, Al doping $MgSiO_3$ perovskite, non-quenchable $CaSiO_3$ perovskite and high pressure pyroxene phase. Weakening effect of Al in perovskite has been confirmed. Data on the non-quenchable phases supply important information for understanding the tectonic structure of mantle.

- ***Melt property study at high pressure:*** A technique has been developed to measure the melt density at high pressures using x-ray radiograph and absorption simulation. Measurements have been carried out on tin and promise to be quite interesting.

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B.3 West Coast Synchrotron Facilities

[R. Jeanloz, University of California at Berkeley]

Report on COMPRES activities at the West Coast Beamlines for 2003-2004 Simon Clark, Elise Knittle and Raymond Jeanloz on behalf of the West Coast COMPRES committee.

Executive summary

Good progress has been made towards achieving our primary objective of establishing the infrastructure for COMPRES use of the West Coast Beamlines. Reductions in proposed funding has resulted in slower progress than planned, and the focus has been on ALS because of the SPEAR3 upgrade at SSRL. A Beamline Scientist and Associate Beamline Scientist have been appointed, and basic infrastructure including facilities for high-pressure powder diffraction and infra-red is now in place. The dedicated laser-heated diamond-cell beamline will come on line during the next calendar year; given sufficient levels of funding, a dedicated infra-red endstation can also be established. The focus for next year is completing the development of facilities and building the COMPRES user base at the ALS and SSRL.

Introduction

The principle objectives for this year, 2003-2004, in order of priority were:

1. Appoint two COMPRES staff, one as a Science Specialist (corresponding LBNL level: Beam line scientist) and one as a helper (corresponding LBNL level: Associate beam line scientist). These people will support COMPRES use of West Coast facilities, at LBNL's ALS and SLAC's SSRL.
2. Establish powder x-ray diffraction and infra-red facilities at the ALS for COMPRES users.
3. Support the community's development of quantitative laser-heating capabilities.
4. Develop a general infrastructure for COMPRES use of the West Coast Beamlines.
5. Contribute to the development of a dedicated high-pressure facility at the ALS.
6. Develop the COMPRES user community at the ALS and SSRL.
7. Explore the use of the ALS for high-pressure single-crystal x-ray diffraction studies.

Progress has been made in all of these areas. Recruitment of personnel has taken longer than expected but that has been somewhat offset by the ALS and other funding sources supporting more manpower for the new beamline development than previously anticipated.

Here follows: a point by point summary of activity this year, a list of objectives for next year, a summary of expenditure for this year, a budget request for next year, lists of COMPRES user groups, publications and dissertations and a list of COMPRES West Coast committee members.

A. Activities this year: 2003-2004

1. Appointment of COMPRES personnel. After an international search, we have succeeded in appointing Dr. Martin Kunz as a Science Specialist for the high-pressure effort based at ALS, and he has started work at the beginning of November, 2003. The fact that an expert of his caliber and international reputation could be attracted to leave a secure position in Europe is an indication of the impact that COMPRES is viewed as potentially bringing to high-pressure research worldwide. Dr. Kunz will focus on the x-ray beamline dedicated for high-pressure research at ALS, but will also support high-pressure experimental activities at the infrared beamlines at ALS as well as at the general-user beamlines at SSRL.

After a national search, we also appointed Katrina Opachich as a helper. She has been working since May, 2003 in helping to set up the high-pressure laboratory at ALS. The two positions are fully supported by COMPRES, with the Science Specialist position being considered a long-term appointment. We anticipate regular rotation of younger staff through the helper position. To support the COMPRES-funded staff, we have obtained separate funding for additional positions. In particular, we obtained support for Dr. Michael Walter, on leave from his faculty position at Okayama University in Japan, to help develop the laser-heating system at ALS. Dr. Simon Clark, who is fully supported by ALS, has donated approximately 33% of his time to COMPRES-related activities, and Professors Elise Knittle and Raymond Jeanloz have each donated more than 5% of their time to COMPRES activities.

2 . Establishment of powder x-ray and infrared facilities. During the construction phase of the dedicated high-pressure x-ray beamline, 12.2.2, access has been provided to the COMPRES community to x-ray diffraction at beamlines 7.3.3 and 11.3.1. At the same time, access is also being provided to the infrared beamline 1.4.4, with the anticipation that equipment dedicated to high-pressure experiments – specifically, optics accommodating a wide range of high-pressure cells – will be established there in the near future.

The primary effort this year is in commissioning and beginning use of the new beamline dedicated to high-pressure research. This involves an investment of $35,000 for end-station equipment, including basic optical benches and alignment equipment, slits, beam position monitors and controls, that will allow collection of x-ray diffraction data as early as the beginning of 2004. We are also investing in associated high-pressure infrastructure based on the program approved in the original proposal, modified by community requests made at our high-pressure workshop. Thus, we are investing about 10% of the total estimated cost for the development of the hardware required for upgrading the infrared facilities at beamline 1.4 for high-pressure experiments. We are also acquiring the cryo-loading system, that was due to be purchased in the first year but was carried over to the second year.

3 We emphasize that this year's funding for the endstation represents the first of two major investments that are needed in order to establish beamline 12.2.2 as a dedicated high-pressure facility. It is thus crucial that the second-year increment be provided in order to ensure that the COMPRES facility at ALS be successfully established. Because of the higher-than anticipated personnel costs and the new initiatives recommended by the community, we have had to make the tough decision to remove

both the Raman system and the laser-feedback system from the current budget. Though useful, neither the Raman system nor the laser-feedback system are absolutely essential for making the beamline operational. Also, it is evident that optimal laser stabilization is still an area of study, as emphasized by several speakers at our first laser-heating workshop, so we feel it is best to hold off on that purchase until the best approach becomes clearer.

Finally, we point out that COMPRES applied for and has been granted recognition as an Approved Program at the ALS. This means that COMPRES users have guaranteed access for up to 20% of the available beamtime on beamline 11.3, for diffraction measurements, and beamline 1.4, for infra-red measurements, in addition to the beamtime available to COMPRES on the dedicated high-pressure beamline 12.2.2.

4. Determination of community priorities.

Two workshops were held this year. The first, on Dec. 11th 2002 addressed the general needs of the west coast COMPRES community. More than 2 dozen colleagues attended from the U. S., Asia and Europe. The presentations can be viewed at: http://www-library.lbl.gov/teid/tmVideo/aboutus/VideoDefault.htm# The second workshop, on Feb. 22nd 2003, was focused on the community's development of a new generation of quantitative laser-heating experiments. A report on this meeting is available on request from either the ALS or COMPRES.

Key outcomes of these workshops include the definition of several research efforts required to enhance laser and external heating of diamond cells, as well as temperature measurements by spectroradiometry and other methods. In addition, two new recommendations were made by the attendees in response to the developments they witnessed at the ALS: 1) ALS should expand and enhance its capabilities for high-pressure infrared absorption and reflectance spectroscopy; and 2) ALS should develop capabilities in single-crystal x-ray diffraction at high pressures. In both cases, the recommendations stemmed from special capabilities evident at ALS and LBNL, as well as from particularly strong interest across the COMPRES research community.

5. Development of general infrastructure.

Leveraging off the CALIPSO effort dedicated to high-pressure research at ALS, the West Coast COMPRES group has established a structure of committees for supporting Earth scientists wishing to conduct high-pressure experiments at SSRL or ALS. The responsibilities of these committees is to 1) advocate for high-pressure research facilities on the West Coast, representing the COMPRES community's needs to the major facilities; 2) develop the relevant community of facilities users; 3) raise funds for and establish supporting experimental capabilities at the major West Coast facilities; and 4) help ensure top-quality science being conducted by the COMPRES community at the facilities. We have been successful on all these counts, including raising funds from DOE, University of California and elsewhere, and using that funding to develop new initiatives in high-pressure infrared spectroscopy and single-crystal x-ray diffraction. The West-Coast group is negotiating how best to make the SSRL facilities available for the COMPRES community after the SPEAR3 upgrade is completed.

6. Contribute to development of dedicated high-pressure facility.

A high-pressure laboratory has been established to support the diamond-cell experiments at ALS. It is equipped with microscopes and tools for aligning cells, loading samples and preparing gaskets. A ruby-fluorescence spectrometer has been installed, as has a two-sided Nd:YLF laser-heating system with spectroradiometry. The heating and pressure-calibration systems are operational, and have been designed so that they can be coupled on-line at the beamline dedicated to high-pressure experiments.

7. Development of user community.

The West Coast COMPRES Committee has been extending the network of new and prospective users throughout the University of California system, including at UC Berkeley, UC Davis, UCLA, UCSC, Lawrence Livermore National Laboratory and Los Alamos National Laboratory, as well as at Caltech, University of Washington, and University of Nevada at Las Vegas, among other institutions across the U. S. (e.g., Columbia University and University of Colorado at Boulder). There has been considerable interest expressed by the international community, including by colleagues in China, India, Israel, Japan and Republic of Korea.

8. Exploratory use of the ALS for HP single crystal studies.

Prompted by the recommendation made at the Dec., 2002 laser-heating workshop, Beamlines 7.3.3 and 11.3.1 have been used to carry out exploratory studies of single-crystal xray diffraction in diamond cells. Dr. H. K. Mao (Carnegie Inst. Washington) and Prof. J. R. Smyth (University of Colorado, Boulder) have participated in test runs and in initial design studies. The results of this work are highly promising, and provide strong incentive for pursuing high-pressure single-crystal x-ray diffraction at the ALS.

B. Summary of expenditure for year 2003-2004.

Our new appointments were made at a higher level than budgeted, because our appointees have far more experience than had been anticipated. This hiring decision was fully supported by the COMPRES community. The increase in personnel costs would have led to over expenditure expcept that we managed to save about $18,000 out of the first-year budget. Those savings will allow us to start up the new dedicated beamline well before the end of the 2003-2004 period, and essentially on budget. 5

We note, however, that by the end of 2003-2004 the cumulative level of funding is almost $80,000 behind the proposed budget. In combination with increased expenditures for personnel, the discrepancy between the approved program of activities and the level of funding being provided becomes critical in the third year of the program, 2004-2005.

C. Objectives for 2004-2005

The objectives for year 2004-2005 in priority order are:

1. Complete the development of high-pressure facilities at the ALS.

2. Build user base.

3. Expand high-pressure single-crystal usage.

4. Develop potential for using SSRL for high-pressure (HP) work.

5. Explore the potential of using the pulsed nature of synchrotron radiation for HP work.

Items 1, 2 and 4 are exactly what was approved in the original proposal. Items 3 and 5 represent exploratory efforts that do not require much investment of funds or personnel, but are crucial for developing new research themes in time for the renewal of COMPRES' 5-year program. In particular, Item 3 was added in response to the recommendation from the COMPRES community made at our Dec. 11, 2002 workshop: a recommendation that has been strengthened by exploratory experiments that have been carried out throughout 2003.

Item 1 includes the development of the dedicated high-pressure x-ray beamline, which will first become operational at the end of 2003, as well as of the high-pressure capabilities of the infrared beamline and the general capabilities of the ALS high-pressure laboratory. Quantitative laser- and resistance-heating continues to be a major objective of the ALS effort. Key hardware that needs to be funded and put in place in the high-pressure beamline includes the K-B mirrors and the in-hutch system for the laser-heating and spectroradiometry. We cannot overemphasize that, although the dedicated high-pressure beamline will be operational starting in the second year, 2003-2004, it will be far from its design capabilities in terms of flux and spatial resolution without completion of the second endstation.

Item 2 is crucially dependent on the expanding user base that is now emerging having access to an operational and user-friendly facility. We are finding considerable interest in the ALS high-pressure facility, both from around the U. S. and internationally. For example, we currently have requests from 6-10 of the most active high-pressure groups around the world (European Union, India, Israel, Japan, People's Republic of China and the U. S.) to get access to the ALS' emerging capability. Given this level of interest that has been expressed without solicitation, it is clear that successful support – let alone expansion – of the user base depends on adequate funding being made available to establish the facility.

Item 4 is as planned: With the renewed access to SSRL general-user beamlines (in particular, 10-2), we will need to establish facilities at SLAC to support the COMPRES community. Past experience shows that, although SSRL has not had beamlines dedicated to high-pressure research, it has proven to be a very effective resource for our community's 6 experiments. A relatively small investment in local infrastructure, mainly facilities for handling samples and loading cells, and for heating samples and determining pressure, would therefore be enormously useful to the community.

Item 5 amounts to exploring the possibility of coupling dynamic experiments based on laserdriven shock-waves to studying matter at extremely high pressures, to the TPa range and higher, at elevated temperatures. The effort will capitalize on existing collaborations with LLNL, and little investment is requested from COMPRES at the present time. Still, we foresee the possibility that this initiative can play a major role in establishing future methods for characterizing planetary fluids at conditions existing deep inside giant as well as terrestrial planets.

COMPRES user groups active at ALS year 2003-2004

Jillian Banfield

Raymond Jeanloz

Abby Kavner

Elise Knittle

David H.-K. Mao

Sang-Heon Dan Shim

David Walker

Quentin Williams

ALS COMPRES Publications

W.R. Panero, L.R. Benedetti and R. Jeanloz, Transport of water into the lower mantle: Roleof sttishovite, J. of Geophysical Research, 108, [doi:10.1029/2002JB002053] (2003).

D. Walker, P.K. Verma, L.M.D. Cranswick, S.M. Clark and S. Buhre, Halite-Sylvite

Thermoelasticity, Geochemistry, Geophysics, Geosystems In Press (2003).

K.K.M. Lee and R. Jeanloz, High-pressure alloying of potassium and iron: Radioactivity in the Earth's core? Geophysical Research Letters, In Press (2003).

A. Salleo, S.T. Taylor, M.C. Martin, W.R. Panero, R. Jeanloz, T. Sands and F.Y. Genin, Laserdriven formation of high-pressure phase in amorphous silica, Nature Materials, In Press (2003).

D. Walker, P.K. Verma, L.M.D. Cranswick, S.M. Clark, R.L. Jones and S. Buhre, Halite-Sylvite Thermoconsolution, Submitted to Am. Min. (2003).

Relevant dissertations

Kanani K. M. Lee (2003) Exploring Planetary Interiors: Experiments at Extreme Conditions (UC Berkeley, Earth and Planetary Science). Committee members Simon Clark, Chair, Raymond Jeanloz, Elise Knittle, Li-chung Ming, Tom Sharpe

B.4 Neutron Studies

[N. Ross, Virginia Polytechnic Institute and State University]

Introduction

The goals of the neutron initiative of COMPRES are to:

(a) Identify and broaden the neutron scattering community of Earth Scientists in the U.S.

(b) To stimulate and promote the use of neutron scattering in the Earth Sciences.

(c) To carry out educational activities that support the above goals.

(d) Identify the needs of the community, including future requirements for instrumentation and sources, and to represent these needs to the neutron facilities (and funding agencies).

Activities (1/1-12/31/03)

Training and Development

A. Wilkinson (GeorgiaTech) and N. Ross (Virginia Tech) co-chaired the Joint Institute of Neutron Scattering Workshop: **Neutrons In Solid State Chemistry and the Earth Sciences Today and Tomorrow,** (March 12-16, 2003). The following attendees received grants from COMPRES:

- Charles Martin (graduate student, Stony Brook)
- Darren Locke (graduate student, Arizona State Univ.)
- Meghan Knapp (graduate student,Ohio State Univ.)
- Kinsom Kam (graduate student, Univ. Calif. Santa Barbara)
- Christopher Holl (graduate student, Univ. Colorado)
- Wendy Mao (graduate student, Univ. Chicago)
- Kim Tait (graduate student, Arizona State Univ.)
- Yang Ding (postdoctoral associate, Geophysical Lab)

R. Angel (Virginia Tech) and B. Toby (NIST) ran a 2-day “hands-on” short course on Rietveld refinement methods directly after the workshop.

The website location with the full Workshop agenda and links to PowerPoint presentations can be found at: http://www.sns.gov/jins/NICEST2003/draft_agenda.pdf

Support for Neutron Research

COMPRES provided funds to offset travel/subsistence costs of U.S.-based Earth Scientists performing neutron scattering experiments at facilities in the U.S. and abroad. The primary requirement for travel and subsistence support is that the researchers have obtained approval for beamtime at the neutron facility in question. Generation of neutrons is costly and therefore proposals for allocation of beamtime are carefully reviewed. Many facilities, however, do not provide travel and subsistence costs and COMPRES is therefore able to facilitate access to neutron facilities by providing these funds.

- *J. Breger (Stony Brook) experiments at ISIS, U.K. (*H-bonded interactions on hydroxides at high pressure)
- *N. Ross (Virginia Tech) experiments at ISIS, U.K. (*High-pressure behavior of H bonding in protonated octahedral frameworks*)*
- *J. Smyth, C. Holl (Univ. Colorado) experiments at ISIS, U.K. (pending) (*Location of hydrogen in hydrous ringwoodite and wadsleyite*)*
- *R. Angel (Virginia Tech) experiments at ISIS, U. K.* Study of dynamic disoreder in lead phosphate PbPO4 at high pressure.
- *A. Celestian, P. Chupas (Stony Brook) experiments LANSCE, U. S.* The local structure of "amorphous" FeS

In addition support was provided to a student to develop the website ($2250), to support invited speakers and students for the special session on "Applications of Neutron Scattering in Earth Sciences" at the 2002 Fall AGU Meeting ($5541), participants of the 2003 NICEST Workshop ($8027) and travel to the 2003 COMPRES annual meeting ($560).

Publications

- Angel RJ, Bismayer U, Marshall WG (2004) Local and long range order in ferroelastic lead phosphate at high pressure. Acta Crystallographica B60, 1-9, 2004.
- Mao WL, Zhao Y, Hemley RJ, Mao H-k (2003) Hydrogen storage in molecular compounds (2003) in prep. [W. Mao was supported by COMPRES to present these results at the 2002 Fall Meeting of the American Geophysical Union (Mao W, Zhao Y, Hemley RJ, Mao H-k (2002) Hydrogen storage in clathrate hydrate. EOS Trans. Am. Geophys. Union 83, 47:F620]

Links with SHUG (SNS and HIFR Users Group)

N. Ross was elected to the executive committee of SHUG in 2002 and will serve through 2004. SHUG communicates the needs of the neutron scattering community to the management of the High Flux Reactor (HIFR) and the Spallation Neutron Source (SNS), both located in Oak Ridge, TN.

Links with SNAP

The proposal SNAP, Spallation Neutrons And Pressure (PI's: J. Parise, R. Hemley, H-k Mao and C. Tulk) was approved for funding by DOE in July 2003. The funding for SNAP includes construction of a high-pressure beamline at the SNS and funds for high-pressure cell development. N. Ross organized a meeting on 11/2/03 at the Geological Society of America Meeting to discuss how to coordinate COMPRES neutron activities and SNAP. The meeting was attended by J. Parise, R. Hemley, H-k Mao, J. Smyth, R. Angel, P. Dera and R.C. Liebermann. It was clear from the discussion that at least one Postdoctoral Research Associate (none are funded by SNAP) is needed to help develop hardware, software, and instrument solutions for high-pressure neutron diffraction experiments, including:

- Assessment and testing of high pressure cells (and high P-T cells), with and without a focused beam and with different detectors;

- Determining the advantages and disadvantages of each cell for different types of experiments;
- Communication of these results to the community (once it is shown what can be done, it will help build the user community).

It was therefore decided that the best course of action would be to request funds from COMPRES for a Postdoctoral Research Associate. Details are given in the Budget Request for Year 3 of COMPRES.

C INFRASTRUCTURE DEVELOPMENT

C.1 Multi-anvil Cell Assembly Development

[K. Leinenweber, J. Tyburczy-Arizona State University]

Summary

The multi-anvil cell assembly development program was created as a collaborative nationwide effort to develop better pressure cells for multi-anvil experiments, and to pool knowledge and resources such as calibration data. The program has proceeded with an effort to produce assemblies with novel designs, and with several investigators becoming involved in collaborative development and testing of the first series of these assemblies. Highlights have included the development of new, less expensive octahedral pressure media by the technique of injection-molding, production of gasket wedges, pre-cut paper backing and aluminum "nesting plates" for more symmetrical and easier assembling of high-pressure runs, wire edm-cut metal furnaces, and ceramic parts for the inside of the assembly made by automated turning techniques. These developments were described in the COMPRES newsletter Vol. 2, # 2 (May 2003). The goals for the third year are to bring a larger number of laboratories into the project, scale up the production of proven cell assemblies, develop new ones including "hybrid" assemblies with low atomic number materials for both in-situ and conventional work, and to continue exploring new materials for use in high-pressure work.

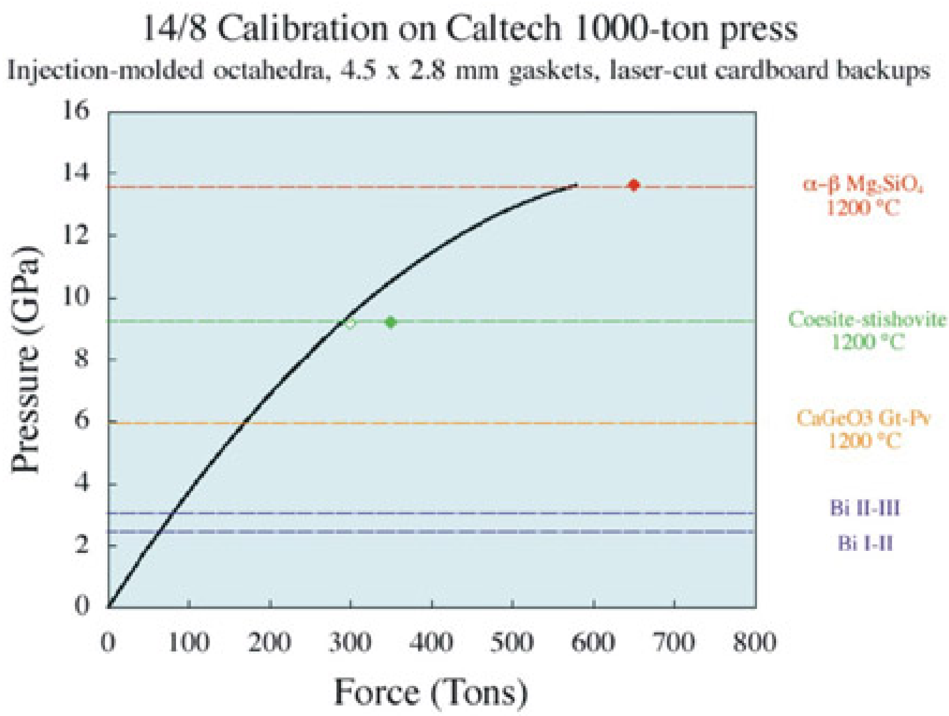
2002-2003 Progress

The multi-anvil cell assembly development effort of COMPRES has had a number of demonstrable successes in its second year. Most of these were achieved by defining areas where significant obstacles exist - the "bottlenecks" in the use of multianvil technology - and finding ways to solve them.

The first bottleneck was an economic one: the high price of the octahedral pressure media for multi-anvil cells, which in addition to being a financial burden on many labs, would have made it difficult for us to use these octahedra as part of the "COMPRES assemblies." The typical price is $25 per piece, plus the labor cost of drilling holes in them. We approached this problem by using the technique of injection-molding, from the Ceramics industry. At about $2500 per mold, finding good ceramic formulas and producing octahedra is definitely a problem that benefits from a Consortium-scale approach. However, it is very cost-effective if a good ceramic is found: with a price per piece after the mold is made of about $6.00, a breakeven point is realized after only about 130 octahedra of a given size and composition are made.

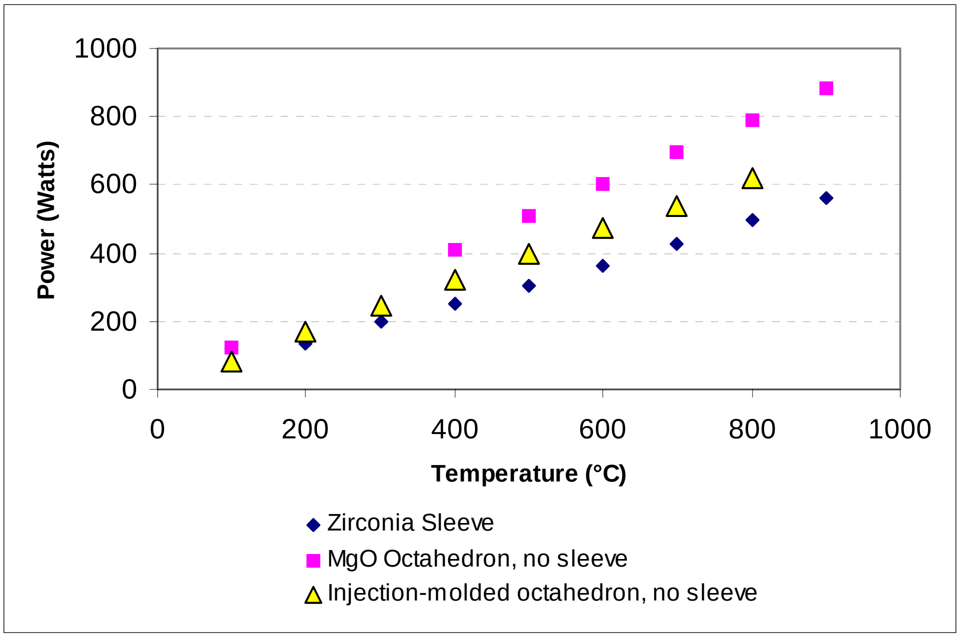
We tested ceramic formulas by having a ceramic injection-molding company[1] make blocks of particular formulas of ceramic that have 20-30 percent porosity, typical for our pressure media, and machining the ceramics into octahedra ourselves for the initial tests. If the ceramics showed reasonable success in duplicating or exceeding others that are currently used, and had other desirable properties, we would then order molds to begin the process of developing the assemblies for the long term. Based on this sequence of events, we currently have 3 molds, a 14 mm, 10 mm and 8 mm octahedral mold for

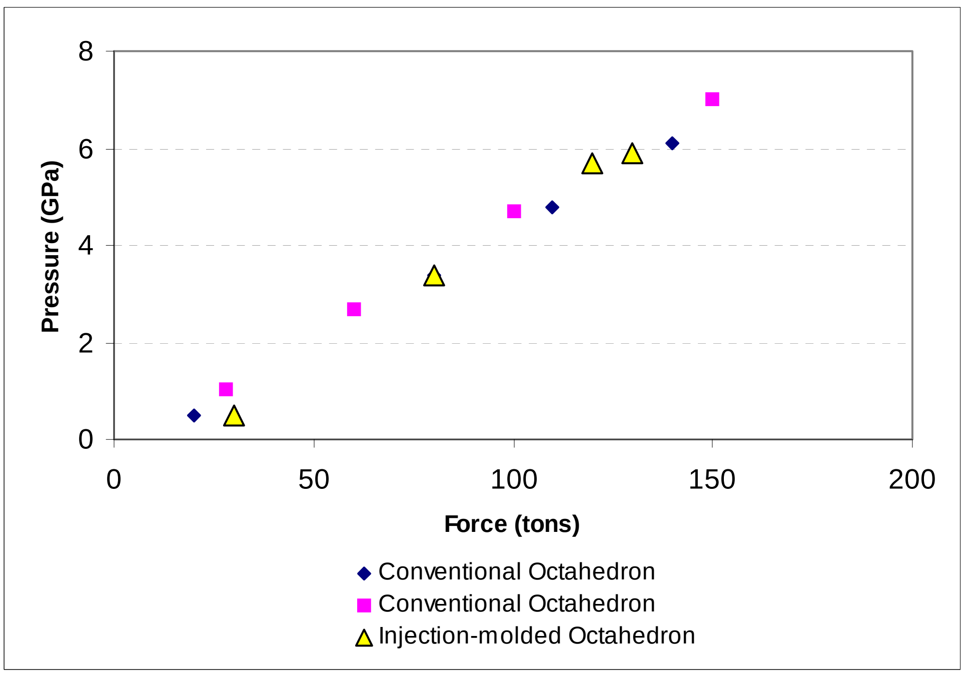
“Formula 1,” an MgO-spinel mixture. Testing has been undertaken here at ASU, and by Jennifer Kung at Stony Brook, Jed Mosenfelder at Caltech, and Thomas Sharp, on Sabbatical at ANU in Canberra. These tests have shown that the pressure generation of the 14/8 and 8/3 assemblies for the new ceramic is very similar to that of the conventional ceramic. Also, tests by Kung of the thermal efficiency of furnaces without a thermal insulating barrier showed that the MgO-spinel mixture is a much better thermal insulator than MgO, as expected from the high intrinsic thermal conductivity of spinel. This formula is now undergoing long-term testing to test overall success rates.



Calibration data on the 14/8 assembly with injection-molded octahedron from Jed Mosenfelder, at Caltech. This octahedron has been used successfully to synthesize Mg_2SiO_4 wadsleyite, demonstrating that the octahedra have the desired capabilites for this assembly size.

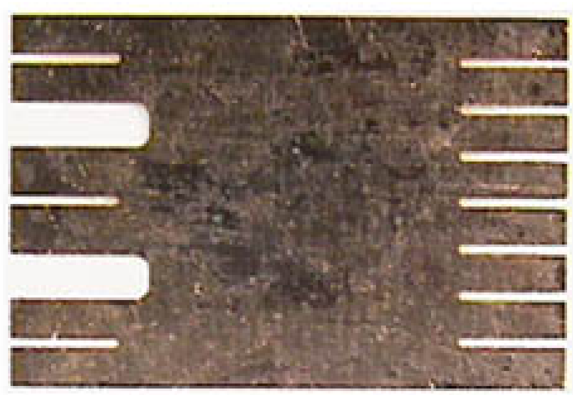
Another bottleneck was the production of gasket pieces, which we are making out of pyrophyllite, at a rate of 12 per assembly. This is often a problem in individual laboratories, and we would benefit many laboratories around the country if we were to develop a low-cost and effective means of producing these parts. We have been working on machining gaskets on an automated mill, and have been able to produce a large number of pieces for the 14/8, 10/5 and 8/3 assemblies. The dimensions have to be chosen carefully; for the 14/8 we chose dimensions slightly modified from the Bayreuth dimensions as suggested by Jed Mosenfelder at Caltech (“Caltech style”); for the 10/5 we are starting with the Bayreuth dimensions modified in the same way, and for the 8/3 we are using Fei’s dimensions. Much of this selection is based on reported blowout rates for the various assemblies, and of course we are trying to minimize the blowout rates. One decision we made early is to make the gasket thickness consistent with the octahedron/truncation dimension; thus, odd-sized gaskets that do not fit the geometry properly are not produced for the COMPRES assemblies. This, combined with the use of properly-sized nesting plates (see below) eliminates "octahedron rattle," or an octahedron that is "loose" in its nest of cubes, a condition that could be expected to cause unpredictable results during compression.





Jennifer Kung's beamline data comparing the injection-molded octahedra with conventional ones. Top: power curves for "bare" octahedra and a zirconia sleeve, showing that the injection-molded MgO/spinel octahedron is a much better thermal insulator than the "bare" MgO one, and is nearly 2/3 of the way to being as good as a zirconia sleeve. Bottom: the pressure curves for the various octahedra are nearly indistinguishable.

The teaching of techniques can also be a bottleneck; if assemblies are very hard to make, or require excessive skill or use of tricks, then the difficulty of transferring the technology between labs slows the spread of multianvil technology. This, along with reproducibility and time savings, is the main reason for certain additions to the normal multi-anvil repertoire that we are introducing for distribution through the community. One is the use of nesting plates, 5 aluminum plates that are used to pre-align the carbide cubes. Another is laser-cut backing paper; this, although modeled after the paper backing used in many labs in Japan, Bayreuth, and others, reverses the logic of assembling. Traditionally, the pyrophyllite gaskets are placed first, by eye, then the paper is glued on (3 sheets per cube) and trimmed to shape with a razor blade. In the current method, the paper is cut and scribed by laser[2] to a precise shape, so the paper can be glued on first (one foldable piece per cube), and the gaskets are positioned by the paper. It takes less training and is highly reproducible. Other improvements such as the accurate wire EDM cutting of rhenium furnaces for the 8/3 assembly[3], and the precise slicing of alumina thermocouple pieces and plugs[4], also reduce the teaching load and enhance reproducibility from run to run.




Rhenium furnace rectangle (left) about 8 mm across, cut by the wire EDM technique, with narrow slits and two thermocouple slots. The rhenium is wrapped and placed into an 8/3 assembly (right – shown with a conventional octahedron from Mino Yogyo).

Another potentially significant development was suggested by Jim Van Orman, who had tried the two available sizes of type C thermocouple wire (.005” and .010”) in the 8/3 assembly, and who suggested that an intermediate size would improve the thermocouple success rate over the .005” size, and the ease of assembly over the .010” size. We were able to locate the original manufacturer of this wire[5], and obtain intermediate sizes (although not supplied with a calibration sheet). Three laboratories are now trying a .007” wire size with very promising results, the success rate has so far been very high with this wire size compared to the .005” size.

These examples should serve to illustrate the spirit of this endeavor, and the aim of enhancing collaboration, communication, and ready comparison of results between laboratories. We are aggressively allowing this by further means; first, by open sharing of our sources, such as the companies and processes that we are using to develop these assemblies. We are making an effort to share all our information, to the point where some laboratories might even choose to pursue the same avenues on their own (Caltech, for example, has already designed its own paper backing for other sizes of assemblies using our laser-cutter); but we see this as enhancing the overall field by increasing the available array of techniques. Secondly, we are providing samples and assemblies, with drawings or ordering information as needed, with the hopes that labs will use the full "COMPRES" assemblies, but we cannot control how other laboratories use these developments, and in some cases our collaborators will use "partial" versions of our assemblies. This is part of the typical behavior of a healthy and active community. We are not trying to force rigid designs on people, but rather are providing a framework for enhancing creativity and communication. Ultimately, though, we hope that our assemblies will be made, characterized, and calibrated well enough that many laboratories will adopt them in their full form. Finally, it should be mentioned that new improvements in success rates, thermal gradient reductions, etc. will always be desirable, so we also do not view our assemblies as final, but only as stepwise developments in multi-anvil high-pressure research techniques.

Plan for 2004-2005

In the third year of this project, we plan to expand the distribution and use of the existing COMPRES assemblies, and to continue an aggressive program of improvements to existing techniques.

The planned improvements will begin with the programming of various additional ceramic and metal parts into the new HAAS mini-lathe, for production. This has already been done for some parts, but the provision of several full assemblies for labs throughout the community will go into full swing in this phase of the project. This includes pre-cutting the ceramic blocks for putting into a chuck (this will be done in some cases at an outside company[4]), then programming a cutting procedure on the lathe to make the part. It is similar to the manual procedure, but the program allows the parts to be made in number sufficient to supply many laboratories, once the programming and setup is done. During this phase, we will also introduce the new step of cutting thermocouple grooves into the pieces while they are still on the lathe, using the live tooling attachments. These are normally cut by hand by individual researchers. This is another step that will reduce the teaching load for the use of the assemblies, and improve reproducibility between runs and between laboratories. Also, if we can gather enough data on thermocouple success relative to the thermocouple path, an "ideal" thermocouple path can be programmed into the machine, and we believe that thermocouple success rates can be improved. We intend as an additional step to look for ways to reduce or eliminate the use of ceramic cement in the placement of thermocouples in assemblies.

The pursuit of new ceramics will be continued, again benefiting from the scale of a Consortium approach. We are trying to create "hybrid" assemblies for use both at beamlines and in conventional laboratories, with the thought that the vast amounts of information gained on assembly behavior at beamlines will then be directly available in conventional laboratories (without the use of special windows and other alterations to assemblies). This will require the elimination of high-atomic number materials, in particular zirconia thermal insulation, lanthanum chromite furnaces/thermal insulation, and precious metal furnaces. Light element materials would allow use with x-rays, but if they degrade the performance otherwise, then they will not be beneficial for conventional use. We are currently testing some trial ceramics already obtained through the COMPRES project to replace high-Z ceramics. For example, we have obtained samples of porous mullite[1]and pure $MgAl_2O_4$ spinel insulation sleeves made by slip-casting[6], which could replace zirconia thermal insulation. We have also found a commercially available machinable furnace material that is a composite of BN and TiB_2[7], which is a candidate to replace $LaCrO_3$ and precious metals for use at very high temperatures (above the temperatures that graphite can be used) with *in-situ* diffraction. We will test all of these, and will continue throughout the third year to consult with ceramics companies in search of new ceramics that can be used for high-pressure research.

Another promising avenue is extruded ceramics[8], which are made in tube form to high tolerances. These can be made in a wide range of compositions, including Mg_2SiO_4 and other interesting compositions, and we will investigate these in the third year, also targeting spinel because the tolerances and mechanical strength might be better than that of the slip-cast material we have seen so far. Indeed, some ceramics may even improve performance over the conventional ones. For example, we have observed that the cubic fraction of the stabilized zirconia in some common assemblies undergoes a transformation to the high-pressure orthorhombic phase, with a significant volume change, in many runs above 10 GPa. In general, phase transitions are known to cause pressure to drop in multianvil experiments. If we use spinel as an insulation material, it will not have such transformations and could potentially reach higher pressure, a hypothesis that will be tested in this project.

We will also continue looking at new injection-molded pressure media (octahedra), beginning with porous mullite ceramic[1] because it has promise as a thermal insulator. We want to have several new ceramic options open to the high-pressure community, since each ceramic has its own properties of strength, thermal conductivity, ductility, and other properties that may make them useful under differing conditions.

Starting in the latter half of the second year, we will be able to supply the community with full assemblies (beyond the 8/3 assembly that we have supplied in full already) in larger quantities, and this will increase the scale of the project. First, we will fulfill the requests of some laboratories to become involved, that have not yet been fulfilled because the particular assemblies of interest to them were not yet fully ready (other sizes besides the 8/3 have only been supplied in part so far, so that the laboratories that desire entire assemblies of these sizes “off the shelf” have not started their involvement in the project yet). Then, we will actively seek out other collaborators that are not currently involved in the project. The involvement of those working at beamlines in the “hybrid assembly” part of the project will be sought as soon as low-Z assemblies are found that perform up to standards. This work has already been ongoing with Jennifer Kung, but we wish to expand the usage of these materials at beamlines. We also need to carefully examine the effect of our modifications, such as backing paper, etc., on the pressure generation, and the beamlines are the best place to do this. These tests will need to be introduced into preexisting scientific projects on the beam lines, since it is difficult to obtain beam time for calibration work alone. Because of the importance of the beam time, all the materials are being tested in-house first, to make sure they can attain the desired pressures and temperatures, are well-insulated thermally, and generally well-behaved. The x-ray transparency as a function of energy can also be predicted accurately using an x-ray absorption calculation, to make sure the cells are appropriate for the projects in question.

Companies doing the work mentioned in the text:

[1]Ceramco, Center Conway, New Hampshire.

[2]Master Designs, Tempe, Arizona.

[3]Microtronics, Inc., Tempe, Arizona

[4]Machined Ceramics, Louisville, Kentucky

[5]Sandvik Rhenium Alloys

[6]Custom Technical Ceramics, Arvada, Colorado

[7]GE Advanced Ceramics

[8]Du-Co Ceramics Company, Saxonburg, PA

Collaborators (2002-3):

Jed Mosenfelder (Caltech; partial 14/8 assembly)

Jennifer Kung (Stony Brook; partial 14/8 assembly, “hybrid” assemblies)

Jim Van Orman (Case Western Reserve; 8/3 assembly, experiments performed at Geophysical Laboratory)

Tom Sharp (on sabattical at ANU, Canberra; 8/3 assembly)

Yousheng Xu (Yale University, Karato group)

Emmanuel Sougnard (University College London, McMillan group; 8/3 assembly, experiments performed at Arizona State University)

Have been sent assembly parts, potential collaborators:

Tony Withers (University of Minnesota; partial 14/8 assembly)

Have requested assemblies or parts, potential collaborators:

Jie Li (University of Illinois Urbana-Champaign; Complete 14/8 assembly)

Larissa Dobrzhinetskaya (UC Riverside; Complete 14/8 assembly)

Joe Smyth (UC Boulder; partial 14/8 assembly, experiments performed at Bayreuth)

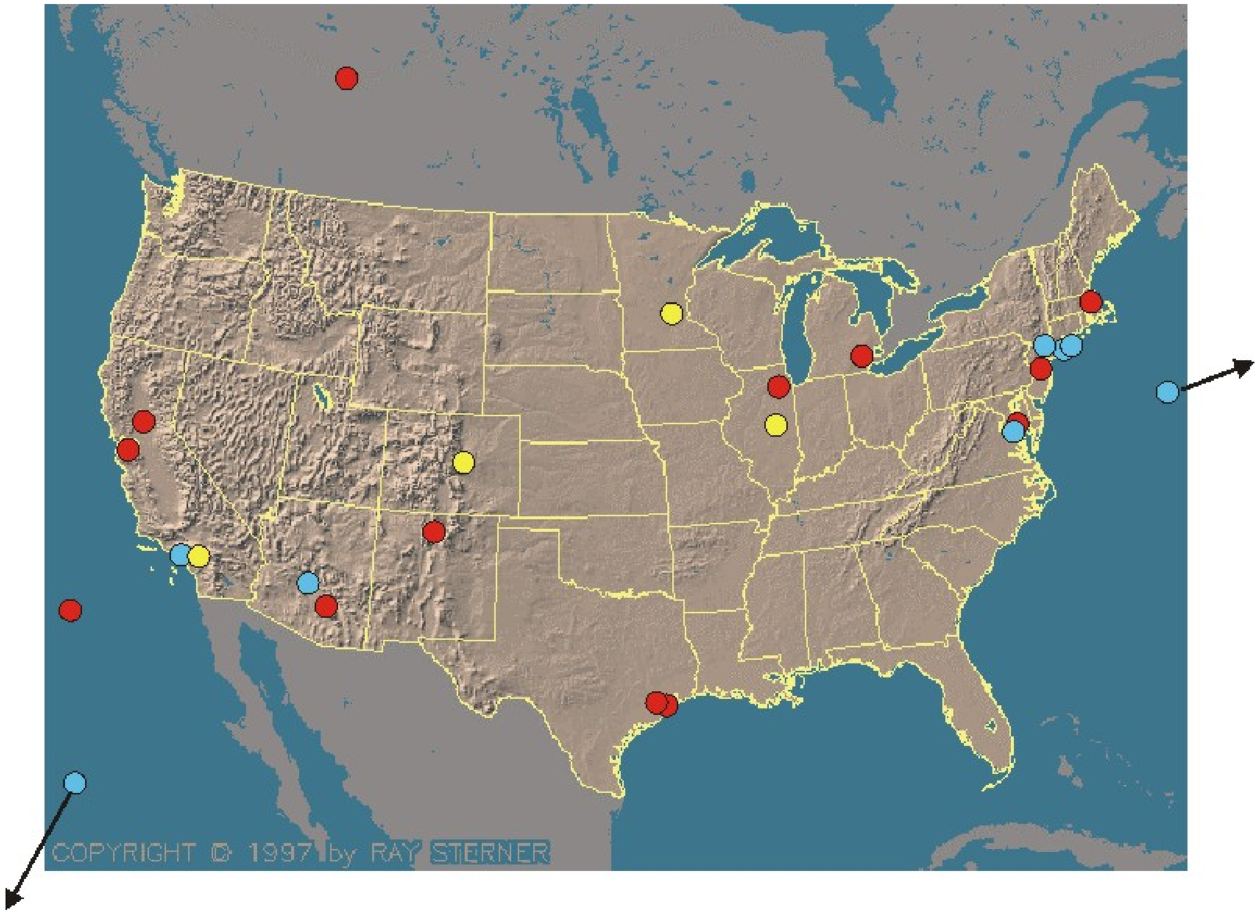


Figure: Map of laboratories that have tested cell assemblies or parts (blue), laboratories that desire to become involved in the project (yellow), and laboratories that we wish to contact in the next phase of the project (red). The authors apologize for any inaccuracies or omissions.

C.2 Development of the Laser-Heated Diamond Anvil Cell

[T. Duffy, Princeton, University, G. Shen and D. Heinz, University of Chicago]

Project Goals

(1) Design and build a new laser-heating system to be installed at the Advanced Photon Source
(2) Develop new procedures and techniques to evaluate and improve laser-heating experiments
(3) Provide a forum the community discussion of needs, goals, and opportunities in diamond anvil cell laser heating

Year 2 Achievements

1. Thermal structure of the laser-heated diamond anvil cell

(a) Finite element models

A series of finite element calculations have been initiated to model heat flow in the diamond anvil cell and test potential new experimental configurations. Much has been learned about thermal structures in the laser-heated diamond cell using analytical or semi-analytical heat flow models (e.g., Bodea and Jeanloz, 1989; Panero and Jeanloz, 2001). However, to date, the use of finite element modeling has been restricted to examining a very narrow range of parameter space (Dewaele et al., 1998). Finite element modeling has the advantage that complex sample geometries can be readily handled and that fully three dimensional and time-dependent calculations can be carried out. The goals of our work are to comprehensively study how axial and radial temperature gradients are affected by properties of the laser (e.g., mode), sample (e.g., thickness), and insulation medium (e.g. thermal conductivity). We are using time dependent calculations to examine heat flow and temperature histories that develop when using pulsed lasers and to determine quench rates. For the laser, we are varying laser mode and heating configuration (TEM_{00} and TEM_{01}, one-side vs. two-side and various permutations thereof), perheating of the sample, and looking at effects of defocusing. We are also simulating complex sample geometries such as the double hot plate and the micro-furnace assemblage. For optically thin samples, we have identified two critical factors in controlling the axial thermal gradient that have not been previously quantified in the literature. These are the ratio of the sample thickness to the anvil gap and the ratio of the thermal conductivity of the insulation medium to the sample. In order to minimize the axial thermal gradient, the insulation medium should have a thermal conductivity less than 10% of the sample while the sample thickness should be less than one-third of the anvil gap. However, high-pressure thermal conductivities are very poorly constrained. We are therefore initiating a study of the thermal conductivity of solid argon (the most commonly used insulating medium) at high P-T conditions using theoretical methods (with S. Scandolo). In addition, we are using finite element models to test possible techniques for measurement of thermal conductivities in the laser heated DAC. Our finite element simulations also suggest that the double hot-plate and micro-furnace methods are very promising for providing homogeneous temperature environments, and we plan to explore these using experiments in the near future. The modeling results are described in more detail in the appendix which includes the figures presented at the COMPRES annual meeting. This work was carried out by B. Kiefer and T. Duffy (Princeton).

(b) Experiments to determine thermal pressure in laser-heated diamond cell

One important question regarding the laser-heated diamond cell involves the role of thermal pressure and its effect on determination of phase boundaries in silicates under mantle conditions. For example, the location of the post-spinel transition in Mg_2SiO_4 has been found to occur at significantly lower pressure in the large-volume press (LVP) (e.g., Irifune et al., 1997) compared with the laser-heated diamond anvil cell (Shim et al., 2001; Chudinovskikh and Boehler, 2001). As this transition is thought to be responsible for the 660-km seismic discontinuity in the Earth's mantle, resolving this controversy is a first-order problem in mantle geophysics. Similar discrepancies between the diamond cell and LVP have been identified for other silicate phase boundaries. The origin of these differences is not understood at present. In particular, the magnitude of thermal pressure effects in the diamond anvil cell are not well constrained. For example, no systematic comparison between pressures determined by *in situ* x-ray standards (e.g. platinum) and by ruby fluorescence from grains located away from the heated spot has ever been carried out, although many workers rely on ruby fluorescence for pressure measurement in laser-heated samples. In experiments conducted in August, 2003 at the GSECARS sector of the Advanced Photon Source, we have laser-heated samples of MgO and platinum in an Ar pressure medium at pressures of 20-40 GPa and temperatures of 1500-2500 K. *In situ* and post-heating pressure measurements were carried out using the x-ray standards. The distribution of deviatoric stresses before, during, and after heating was evaluated using Singh's lattice strain theory. Pressure measurements across the sample were made using the ruby fluorescence technique. The analysis of the data is still in progress, but our preliminary indications are that this study will provide useful constraints on pressure and stress distribution in the laser-heated diamond cell. Future studies will focus on such factors as the effects of different pressure-transmitting media and the relative proportions of pressure-transmitting medium and sample, as well as extension to higher pressures. This work was carried out by T. Duffy, S. Shieh, and A. Kubo (Princeton University) and G. Shen (U. of Chicago).

2. Ultrahigh-pressure laser heating

A series of laser-heating experiments on SiO_2 samples at ultra-high pressures (~70-130 GPa) have been carried out to both address important scientific issues in the SiO_2 system as well as technical issues associated with such ultra-high pressure laser heating where issues of heating homogeneity and thermal stability become very critical. In our experiments on SiO_2, we have examined the following aspects of the sample environment: 1) role of starting material; 2) effect of insulation medium; 3) x-ray/laser positioning issues. The extensive polymorphism of SiO_2 makes it especially interesting for investigating the extent to which differences in starting materials can explain discrepant results among laser heating studies on a particular system. We obtained consistent results under extreme high pressure and temperature conditions from three different starting materials: silica glass, stishovite, and cristobalite. In the case of cristobalite starting material, an α-PbO_2-type structure was observed during room-temperature compression consistent with earlier results by L. Dubrovinsky and colleagues. However, in contrast to these earlier studies, we observed the growth of peaks of the $CaCl_2$-type phase and diminishment of the α-PbO_2-type peaks under high P-T conditions. Our results suggest the α-PbO_2 type under these conditions is a metastable phase. Discrepancies between our results and some earlier studies on SiO_2 might also be ascribed to differences in the type and quality of insulation material which greatly influence the thermal gradients developed in the laser-heated DAC. Hence, we performed runs both with and without an insulation media for stishovite starting material. However, we found no difference in the observed phases in these two runs. Finally, it has been noted previously that determination of the averaged temperature in the sample volume probed by x-rays is particularly sensitive to small misalignments between the heating spot and the x-ray beam. We have developed a new method to position the x-ray and laser spots to within a few μm through the use of an x-ray fluorescent YAG crystal. The small (~5 μm) YAG crystal is placed in the sample chamber away from the sample where it is optically visible under the microscope. The location of the x-ray beam can then be precisely identified on a video monitor by observation the fluorescence of this crystal. The center of the laser heating spot can then by accurately aligned with the YAG crystal. This method can also be used to check for drift of the relative position of the x-ray and heating spot after heating. This work was carried out by S. Shieh and T. Duffy (Princeton U.) and G. Shen (Chicago).

3. Development of a new laser heating system at GSECARS sector of the Advanced Photon Source

The new laser heating system at GSECARS must complement existing laser heating systems at the APS and must be developed to expand and optimize the opportunities for conducting high pressure-temperature experiments at the APS. The major features of the system under development are:

- Continuous (cw) CO_2 laser system. Existing DAC laser heating systems (sectors 3, 13, 16) at the APS are all based on ~1 μm radiation (Nd:YAG or Nd:YLF) and generally follow the Shen design (Shen et al., 01). A CO_2 laser heating system will thus expand the range, quality, and flexibility of experiments that can be carried out. Both pulsed and continuous (cw) TEM_{00} systems were considered. While a pulsed laser system offers some intriguing possibilities (Rekhi et al., 03), it was concluded that a (quasi-)cw system would satisfy the broadest range of user needs. On the basis of power, stability, divergence, and price considerations, the laser choice has been narrowed to two candidates: Coherent Diamond 250 and Synrad Evolution 240.
- The laser-heating system will be installed on the insertion device beamline at the GSECARS sector. CO_2 laser heating is often the optimum choice for heating of weakly diffracting silicates. Thus, the highest x-ray intensity is required. It was judged that this benefit outweighs the disadvantage of greater competition for ID beamtime. The new CO_2 system will be combined with the existing Nd:YLF system and a redesigned x-ray diffractometer in the 13-ID-D enclosure. Combining both laser heating systems in the same station optimizes use of resources and allows for future development of other experimental capabilities at the 13-ID-C station. Also, the development of the new system will be coordinated with the redesign of the x-ray diffractometer at 13-ID-D to a more stable, more compact, and more user-friendly

arrangement. This will also lead to a higher degree of flexibility and stability for the laser-heating systems (See preliminary design in Appendix II).

- The CO_2 laser and incident optics will be mounted above the diffractometer. The beam will be directed onto the sample using motorized stages from the CO_2 laser (refracting) objective and carbon mirror. Modular motorized optics will allow for insertion and removal of Nd:YLF or CO_2 optics. CO_2 heating will be one-sided and we expect to achieve a heating spot of 60-80 microns. The other side will be for temperature measurements using either a reflecting objective or shared refracting optics with Nd:YLF system. The combined system will offer a wide flexibility in heating optics with the best possible diffraction capabilities.
- A detailed design will be presented at the laser-heating workshop planned for March (see below). After refinement based on community input, we plan to complete major equipment purchases near the end of year 2. Construction and testing will carried out in summer of 2004, and we expect that installation on the beamline will follow shortly although we recognize that coordination with GSECARS maintenance, upgrade, and beamtime schedules will be necessary.

4. Community Outreach activities

A workshop entitled "The Laser-Heated Diamond Anvil Cell: Progress and Future Prospects" will be held on March 28-29, 2004 at the Advanced Photon Source and planning for this is ongoing. This will be held immediately after the American Physical Society March Meeting in Montreal Canada in order to facilitate participation from international scientists. The purpose of the workshop will be to provide a forum for the community to discuss advances in laser heating techniques and to solicit community input into the ongoing design of the new system. Other recent outreach activities include participation in the ALS Laser heating workshop (Feb. 2003) and organization of a Laser Heating mini-workshop at the COMPRES annual meeting (June 2003). Presentations in the workshop were given by: T. Duffy (Princeton), A. Kavner (UCLA), D. Heinz (Chicago), O. Tschauner (UNLV), D. Mao (CIW), J. Crowhurst (LLNL), S. Sinogeiken (Illinois), G. Shen (Chicago), and C. Zha (Cornell). Special sessions at the Fall AGU meeting emphasizing the links between high-pressure mineral physics and deep Earth geophysics were organized by T. Duffy (2002 Fall Meeting) and G. Shen (2003 Fall meeting).

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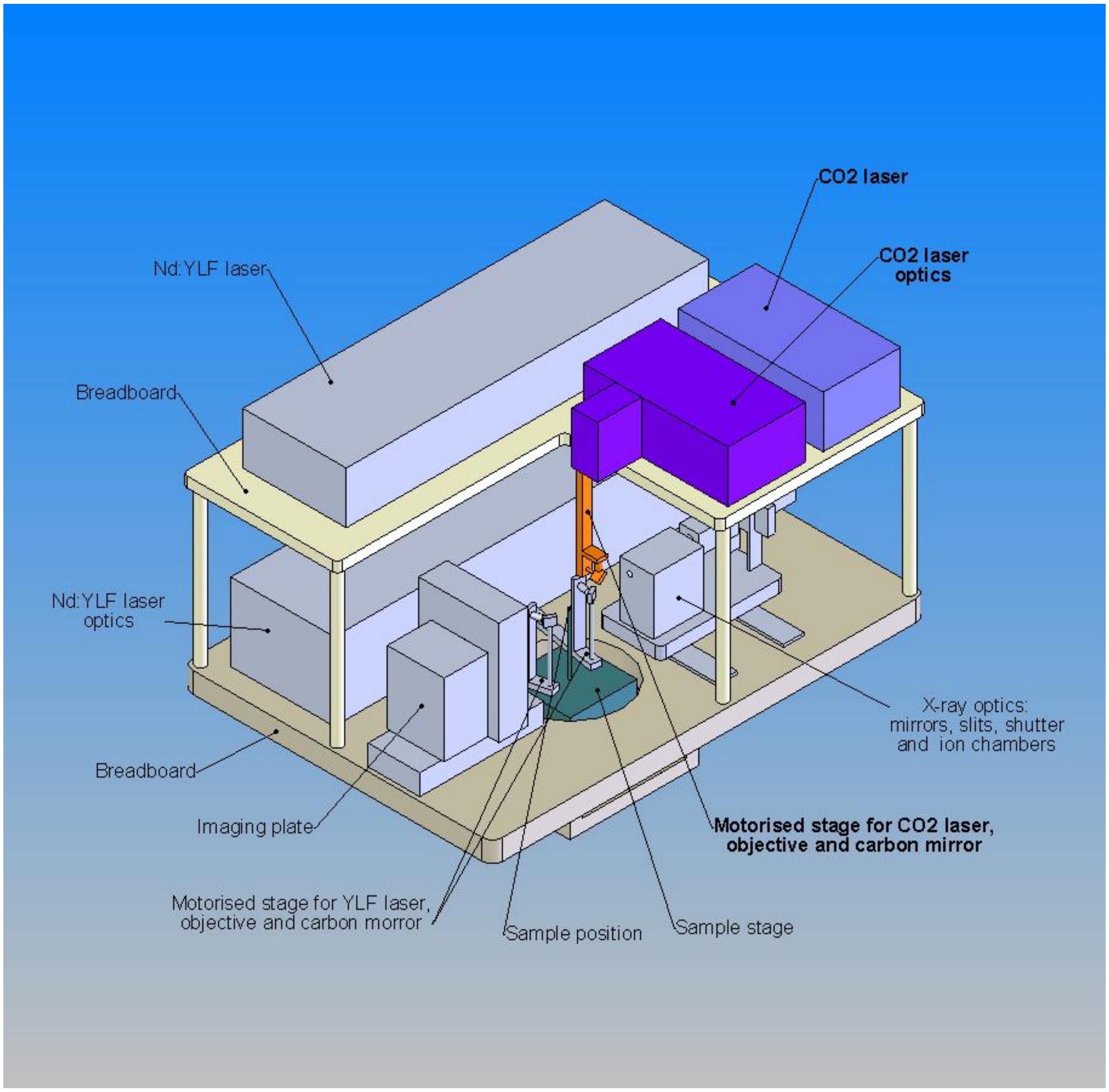
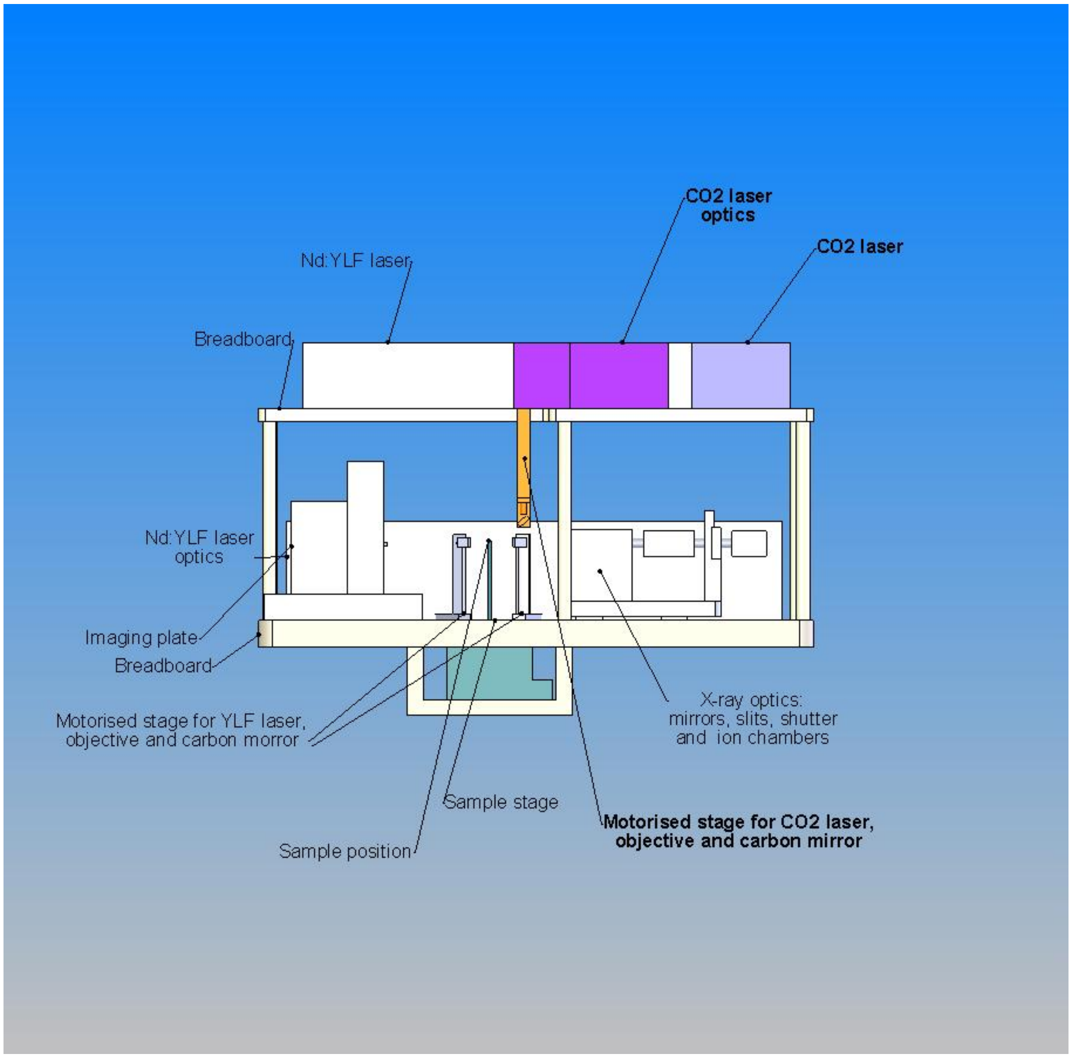
S. Rekhi et al., *Rev. Sci. Instrum., 74*, 3820, 2003.

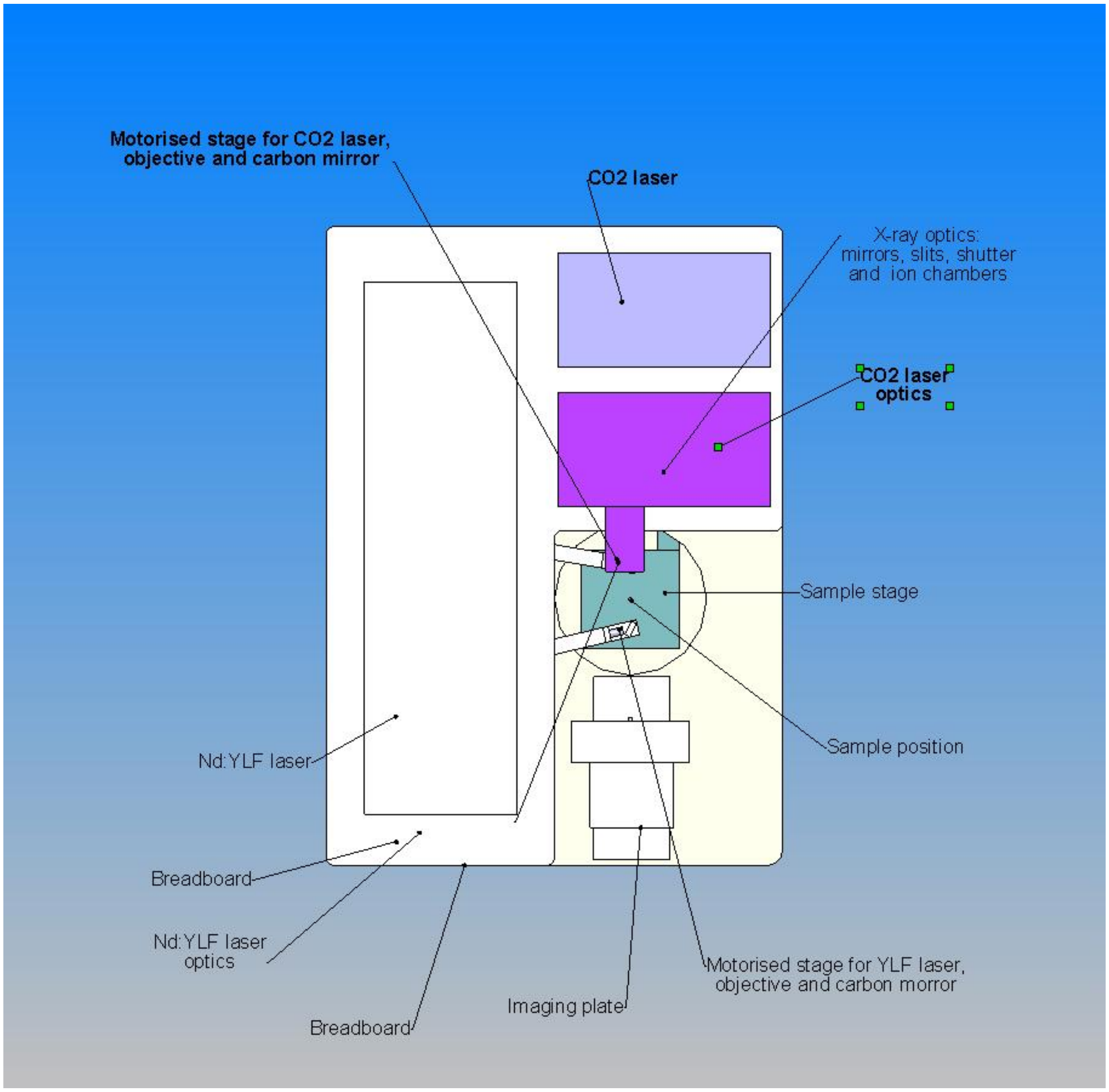
G. Shen et al., *Rev. Sci. Instrum., 72*, 1273, 2001.

S. Shim et al., *Nature, 411*, 571, 2001

Preliminary design of laser heating/diffraction system




CO2 laser optics
CO2 laser
Nd:YLF laser
Breadboard
Nd:YLF laser optics
Imaging plate
Breadboard
Motorised stage for YLF laser, objective and carbon morror
X-ray optics: mirrors, slits, shutter and ion chambers
Sample stage
Motorised stage for CO2 laser, objective and carbon mirror
Sample position


Motorised stage for CO2 laser,
objective and carbon mirror
CO2 laser
X-ray optics:
mirrors, slits, shutter
and ion chambers
CO2 laser
optics
Sample stage
Sample position
Nd:YLF laser
Breadboard
Nd:YLF laser
optics
Breadboard
Imaging plate
Motorised stage for YLF laser,
objective and carbon morror

Publications

Publications (supported wholly or in part by this project):

Duffy, T. S., Advances in Understanding the Earth's Deep Interior From High-Pressure Experiments at Community-Based Synchrotron Facilities, *Eos Trans. AGU, 83,* Fall Meet. Suppl., Abstract U12A-07, 2002.

The development of second- and third-generation synchrotron x-ray facilities has led to enormous growth and development in the field of high-pressure research that has dramatically improved our understanding of deep planetary interiors. In this talk, I will primarily focus on results from research conducted at the National Synchrotron Light Source and the GSECARS sector of the Advanced Photon Source. These synchrotron facilities have enabled entirely new classes of experiments that take advantage of the unique characteristics of the synchrotron beam including extremely high brilliance and low divergence, together with advances in beamline components. As an example of just one developing research area, the coupling of the brightest synchrotron x-ray diffraction beamlines with the most advanced systems for laser heating allow researchers to explore both simple and complex mineral systems under pressure-temperature conditions reaching up to those of the Earth's core-mantle boundary and beyond. For example, we have recently carried out a comprehensive study of the behavior of both SiO_2 and $MgSiO_3$ up to 120 GPa and 2500 K to understand the phase transitions occurring in these fundamental systems in the deep mantle. At conditions corresponding to the 660-km discontinuity and the top of the lower mantle, we have carried out the first in situ measurements of the spinel- perovskite + periclase transition in the laser-heated diamond cell. Examples of other advances in high-pressure experimentation arising from the development of these facilities and the dynamic user community that has sprung up around them will be discussed. Examples of such advances include: mineral elasticity from coupled ultrasonic/synchrotron experiments, high-resolution crystal structure determination, stress/stain measurements of rheology and elasticity, viscosity and liquid structure determination, melting curves, and x-ray spectroscopies for determination of sound velocities, thermodynamic properties, and electronic structure. The future looks very bright for the continued rapid advancement of experimental capabilities using community-based synchrotron facilities, and some important future directions will be highlighted.

Shieh, S. R., T. S. Duffy, G. Shen, The Enigma of High-Pressure Silica Polymorphs: Is There a Post-$CaCl_2$ Phase the Lower Mantle? , *Eos Trans. AGU, 83* Fall Meet. Suppl., Abstract S52C-11, 2002.

The high-pressure polymorphs of silica have attracted much attention due to their geological, materials science, and crystal chemical importance. It has been suggested that phase transitions in SiO_2 may be related to seismic structure at ~1000 km depth, the mid-mantle discontinuity, and anomalous structure near the core-mantle boundary. Stishovite is known to transform to the $CaCl_2$ form near 50 GPa on the basis of Raman spectroscopy, x-ray diffraction, and theoretical calculations. Many studies have investigated possible post-$CaCl_2$ phases at lower mantle conditions but current results are contradictory. Because the post-$CaCl_2$ phases are believed to be energetically competitive, different results among experimental studies could depend on differential stress, pressure and temperature gradients, starting material, and heating history. In this study we used a variety of different starting materials (stishovite, cristobalite, silica glass) and also varied the heating environment. Sample materials were mixed with Pt and loaded into a Re gasket hole. In most experiments, Ar was used as an insulator and pressure transmitting medium, but a few experiments were performed with no insulation to investigate the effects of P and T gradients. Experiments were carried out using the double-sided Nd:YLF laser heating system at 13-ID-D of the GSCECARS sector of the Advanced Photon Source. Alignment of the x-ray and heating spots was facilitated by the placement of YAG crystals in the sample chamber. Angle-dispersive diffraction techniques were employed together with a CCD detector. Our in situ studies on SiO_2 show that the $CaCl_2$ phase remains stable to about 120 GPa using both stishovite and glass starting materials. No differences were found between experiments employing an Ar insulation medium and those that did not. For cristobalite starting material, the α-PbO_2 phase was observed at pressures above 54 GPa and room temperature. However upon heating at 80 GPa, the α-PbO_2phase transformed into the $CaCl_2$ phase. A separate set of experiments on GeO_2 carried out at Spring-8 showed that the $CaCl_2$-α-PbO_2 phase transition occurred near 65 GPa in this material.

Shen, G. et al., High Pressure Research With the Diamond Anvil Cell at GSECARS
EOS Trans. AGU, Fall Meeting, 2002.

The high pressure diamond anvil cell (DAC) program at GSECARS started with a combination of commissioning and experiments from December 1996. Since early 1999, it has been running proposal-based experiments full time. These experiments address key geochemical and geophysical problems in the Earth's deep interior. Research topics include: -equations of state, crystal structures, and phase relations of mantle and core materials, high PT studies of melts, glasses, and other non-crystalline materials,-properties of light elements relevant to the outer planets, rheology of minerals at high pressure, pressure effects on magnetic and electronic properties of deep Earth materials, ultra high pressure-temperature experiments, kinetics of phase transitions and chemical reactions at high PT. Meanwhile, major technical advances have been made, providing a unique facility at the third generation synchrotron source. Some examples include: providing small and intense x-ray beam suitable for miniature sample in the diamond anvil cell (beam size <10 μm at FWHM and <30 μm at FWHM), a double-sided laser heating system combined with x-ray area detectors for high-resolution diffraction applications at high PT and a feedback system incorporated to dramatically improve long-term temperature stability, the implementation of amorphous boron gaskets, making it possible to use x-ray area detectors for strain measurements and resulting in high quality texture and rheology investigations, using x-radiography in the DAC for accurate melt volume determinations, a high-resolution Rowland circle spectrometer for high resolution spectroscopic experiments, a pair of 1-meter long focusing mirrors focusing almost the entire undulator x-ray beam to a size of 30x70 μm FWHM, making it possible for collecting weak signals in x-ray inelastic scattering experiments. These developments will be presented together with some highlights of scientific results. Acknowledgments: We thank the DAC design team members: Russell Hemley (Leader), William Bassett, Thomas Duffy, Dion Heinz, Ho-kwang Mao, Li-Chung Ming, Surendra Saxena for their continuous support. The DAC program is supported by NSF-Earth Science Instrumentation and Facilities, DOE-Geosciences, and the W. M. Keck Foundation.

Kiefer, B., and T. S. Duffy, Finite-Element Modeling of the Thermal Structure in Laser-Heated Diamond Anvil Cell Experiments, *EOS Trans. AGU*, Fall Meeting, 2003.

Laser-heated diamond anvil cell experiments (LHDAC) form an integral part of the exploration of the physical properties of planetary materials at pressures and temperatures characteristic of planetary interiors. While it is possible to measure the radial temperature distribution in LHDAC experiments it is far more difficult to study the axial temperature gradient in these experiments. However, experimental observations are commonly made along the DAC axis and the observed signal represents a volume average of the thermal structure along the DAC axis. It is therefore important to analyze the effect of different sample assemblages and different insulating materials on the axial temperature gradient. In order to address these issues we have performed finite element simulations for different geometries and insulating media. All calculations were performed in (2d) cylindrical geometry with dynamic grid refinement. Our steady-state calculations confirm previous studies in that the diamond anvils remain essentially at room temperature, regardless of the insulating medium. Furthermore we identify the thermal conductivity contrast of sample and insulating medium as a key parameter to determine the thermal structure in LHDAC experiments. Based on these insights we have been able to develop a simple analytical model that allows us to predict the axial temperature distribution to within 10%. The analysis of different experimental geometries shows that the microfurnace assemblage reduces the axial gradient most efficiently, followed by double sided laser heating geometries. These results also indicate that finite-element modeling is a viable tool to assess and design new LHDAC experiments.

C.3 Brillouin Spectroscopy at the Advanced Photon Source

[J. Bass-University of Illinois and G. Shen-University of Chicago]

Accomplishments:

The objective of this project is to construct a Brillouin spectrometer at that Advanced Photon Source (APS) for measurement of sound velocities (by Brillouin) and density (by x-rays) simultaneously. There has been a great deal of progress on this project within the last year. The Fabry-Perot interferometer, which is the heart of the Brillouin system, has been delivered and been set up. To facilitate transport of the instrument to the APS, it has been mounted on a TMC© breadboard, which in turn is fixed to a larger optical table purchased for this project. This will also give us flexibility in system configuration at the APS beam-line. The larger optical table has all optics external to the Fabry-Perot on it. At the present, we have completed the set up a preliminary Brillouin spectrometer around the tandem Fabry perot interferometer that will be installed at APS. This simple setup (see figure below) was used to test the interferometer and test various ways in which we can configure the optics to be compatible with beamline service. The Brillouin system is now operational with this very basic optical setup.

It has been decided that the Brillouin spectrometer will be installed on the bending magnet beamline 13BM-D at the APS. This beamline is not as heavily subscribed as the double-sided laser heating beamline, thus making more time available for the set-up and testing of the instrument. Also, after the system if fully operational, there will be more time for Brillouin experiments. We have worked out a general plan for the location and general design of the Brillouin system. It will be installed permanently in the 13BM-D hutch, on an elevated 2nd level (above the level of the x-ray beam). So as not to interfere with other experiments, we need to make some of the optics moveable or retractable. Various options for how to achieve this are currently being discussed between Bass, Shen, Mark Rivers, and the design engineers at sector 13. The basic design concepts for accommodating the spectrometer at APS are decided, and this year the numerous details and specifications will be worked out.

Tasks for the coming year:

Now that the APS-bound interferometer is set up, many additional tasks remain to be done. These include:

- Finalize the optical design and order/install additional optical components. The final configuration of the system will be considerably more sophisticated than its present form. We will install polarizers, motorized optical mounts and translators, beam steering devices, and other optical components. We must test various alternative optical systems and lens combinations, systems for remote viewing of sample images, etc.
- Install a solid state laser (Coherent Verdi)
- Continue to work with APS on design of the elevated table, support system, retractable optics, etc., (see below) for the synchrotron installation.
- Once the construction of something close to the final system is finished at UIUC, we will move the system to the APS. We've decided that an L-shaped table will

be installed on an upper 2nd level in 13BM. We will reinstall the Brillouin system in the hutch. We will work on remote control of optical components so that the system can be aligned with the xray beam and ultimately a laser heater. We will install retractable supports for some of the optics steering the light into the Brillouin system.

- We must develop software to interface the spectrometer with our existing data reduction programs. Unlike previous versions of the Fabry-Perot, the current instrument is run with a PC-based multi-channel scaler (MCS) that controls all functions and accumulates individual spectra. This program needs to feed raw results into our data reduction programs, which are set up to handle large single-crystal data sets. Efficient transfer of information from the MCS to our data reduction programs in a compatible format needs to be achieved.

C.4 Absolute Pressure and Temperature Calibration

[I. Getting, University of Colorado]

This component of the COMPRES Infrastructure Development program seeks to establish and implement within the high pressure community accurate temperature and pressure scales based on NIST traceable measurements and sound metrological practice.

Temperature

Temperature measurements have proven very difficult in high pressure environments. Decades of consideration have failed to yield realistic calibration for thermocouples as used at high pressure. Temperature can be measured accurately in a high pressure environment by Johnson noise thermometry, however. Johnson noise is the very small voltage noise generated across any resistor at temperature above absolute zero. The mean square noise voltage across a resistor *R* is $\langle E^2 \rangle = 4kBRT$, where *k* is Boltzmann's constant, *T* is the absolute temperature, and *B* is the electrical bandwidth in which the noise voltage is observed. This Gaussian-distributed, random fluctuating voltage has zero mean, but a definite quadratic-average value. All of the effects of pressure, strain, and any chemical reactions on the resistor sensor are cast into the resistance term. The resistance is measured separately for each reading thereby accounting for all such effects. Very subtle electrical measurements are required to determine the value of $\langle E^2 \rangle$. John Hall, an invaluable metrologist colleague at the University of Colorado, and I have constructed all the circuits for an automated Johnson noise thermometer to be used at high pressure. In the last few months these components have been assembled and tested successfully. A new computer sufficient to the task of sampling and reducing this data has been acquired. Computer code and instrument networking have been completed permitting reliable and reproducible measurements.

The recently developed software allows us to calculate and display the power spectral density of the noise signal. This new tool has permitted us to eliminate spurious electrical pick-up in the circuitry preserving the Johnson noise signal and to improve the stability of circuit components.

The value of $\langle E^2 \rangle$ depends only on several constants and the product *RT*. We are now making fully automated readings of all the relevant electrical parameters simulating high temperatures by changing resistance. One million samples of this random noise requires 10 seconds sampling time and results in a temperature resolution of better than 2 K at 1000 K. The time to sweep through a complete set of all the required measurements is 15 seconds. Higher resolution is achieved by additional sampling.

It would be very difficult to make an absolute Johnson noise thermometer in and of itself due to slight non-linearity and ambiguous definition of band width in the electronic amplifiers involved. This thermometer will be calibrated against NIST traceable thermocouples at one atmosphere to establish its intrinsic calibration. An high quality, NIST traceable digital voltmeter and NIST traceable thermocouples for this purpose are on hand. The final result will be NIST traceable determinations of absolute temperature at high pressure. Measurements of the effect of hydrostatic pressure will be made on several thermocouple types in the gas piston cylinder apparatus. This does not directly solve the problem of temperature measurements in the various solid medium devices, however, as thermocouples are additionally affected by strain and chemical reactions in those instruments. The beauty of the Johnson noise thermometer is that it can be applied in any large volume high pressure device to the maximum pressure and temperature attainable. All that is required is a resistor and four electrical leads and appropriate electrical insulation between the critical components. Using thermoelements for the leads yields a direct calibration of thermocouples. The major effort in the next two years will be concentrated on this Johnson noise thermometry.

Pressure

Pressure refers to uniform, isotropic, compressive stress. The gas piston cylinder apparatus at Colorado offers unique capability in producing very nearly hydrostatic pressure of know magnitude. It uses fluid/solid argon as its pressure medium and has a sliding piston seal with extremely low friction. Absolute, NIST traceable pressure uncertainty ranges from 0.2 % at 2 GPa to an anticipated value of 0.5 % at 6 GPa.

In response to a significant reduction in funding and personnel NIST traceable absolute pressure calibration efforts have been suspended in favor of temperature calibration. Relative pressure discrepancies are under study by comparative equation of state measurements elsewhere under this infrastructure initiative. Absolute pressure determinations remain critical, however. Redundant equation of determinations of absolute pressure would be extremely valuable, but are not part of this infrastructure program at this time to the best of my knowledge.

Plans

My post-retirement employment at the University of Colorado is limited to five months per year. I anticipate working from December through April each of the first 3 years of COMPRES. My original work plan called for 18 months collaboration with a post-doc. The loss of this component of the project means a large reduction in total effort during the 2nd and 3rd years. During the five month of the current, second year of COMPRES, I plan to complete one atmosphere development, testing, and calibration of the Johnson noise thermometer and to calibrate thermocouples under hydrostatic stress in the gas piston cylinder apparatus.

During the third year Johnson noise thermometry should be migrated to solid media, large-volume high pressure devices. The best, in fact the only reasonable, way to begin this process will be to bring multi-anvil tooling sets to Colorado. Issues of electrical noise pollution from the ambient will all ready have been well solved in the course of running the Johnson noise thermometer in the gas piston cylinder apparatus. For instance, a specialized power supply may be required for cells in which the Johnson noise thermometer is used as the electrical noise from typical SCR power supplies may be intolerable. I anticipate the further assistance of my colleague Jan Hall as we explore these issues during the second year.

There are tooling sets presently on hand in various labs for both cubic and octahedral multi-anvils which can be run in my 7 MN (~800 ton) capacity press. Thermocouple calibration depends on both apparatus type and the specific cell utilized, but is independent of the press and lab in which the tests are made. A variety of cells could be calibrated in Colorado with the results applied elsewhere. If the COMPRES effort to develop standardized cells has been effective by that time, the Johnson noise thermometer should surely be run in those cells. This would constitute a very efficient means of migrating accurate temperature measurement to many other labs simultaneously as the thermocouple calibration procedure need not be repeated in the labs using those cells.

The cubic D-DIA tooling set at Livermore National Laboratory and DIA tooling sets at SUNY and APS are self-contained and can be run in my press to full capacity. The results from any one of these X-ray transparent tooling sets would be applicable to the others using the same cell. The unique D-DIA device would enable us to calibrate thermocouples not only at quasi-hydrostatic pressure, but also in the deviatoric stress fields associated with controlled sample deformation.

The “T-cup” apparatus at SUNY and the “T-10” apparatus at APS are X-ray transparent, miniature octahedral multi-anvils. They also can be run to their full capacity at Colorado.

The “Walker Module” is a “full size” octahedral device with one inch carbide cubes. This apparatus can be run at Colorado to a large fraction of it maximum load capacity. This type of apparatus is in wide use. Thermocouple calibration transferal, especially with standard COMPRES cells, would be very efficient in this device.

This strategy will require others to commit their time, tooling sets, cells parts, personnel, and skills to the migration effort. They will have to come to Colorado for requisite periods of time with their equipment. They can reasonably hope to leave with the best temperature measurements ever made at high pressure. No workshops are anticipated in the first two years of this lab-intensive effort. This will be made up for in the third year with collaborative thermocouple calibration sessions at Colorado involving a wide range of devices and cells.

Some types of high pressure devices and cells would be best calibrated in their home labs. This might include large octahedral multi-anvils with bear loads in excess of 7 MN. My lab is not equipped to handle these types of device. Eventually the Johnson noise thermometer should be carried to other labs. In anticipation of my withdrawl from high pressure research after the first three years of COMPRES it is imperative that COMPRES identify, train, and support others to carry on the accurate measurement of temperature with the Johnson noise thermometer that I have built.

C.5 Pressure Calibration at High Temperatures

[Y. Fei- Carnegie Institution of Washington]

Progress

The main goal of the project is to examine differences in the existing pressure scales at high temperature and establish a self-consistent practical pressure scale. We conducted multi-anvil experiments at simultaneous high pressures and temperatures using multiple internal pressure standards including Au, Pt, MgO, W, Mo, Pd, and Ag. Extensive synchrotron X-ray diffraction data for Au, Pt, and MgO were collected at pressures up to28 GPa and temperatures between 300 K and 2173 K. We compare pressures calculated from different pressure scales and demonstrate large discrepancies in pressure determination using different pressure standards or different thermal equations of state for the same standard. The comparison allows us to quantitatively determine the differences in pressure using different pressure scales in the high P-T experiments. Using the MgO scale of Speziale et al. (2001) as a reference pressure scale, new Au and Pt scales are presented that are consistent with the MgO scale. We further examined the validity of the assumption of constant *q* value (volume dependence of the Grüneisen parameter in the Mie-Grüneisen relation) for the calculations of thermal pressures, and show that an expression of *q* as a function of temperature and pressure may be necessary to best fit the simultaneous high P-T data. These results will be published in a special PEPI issue on high-pressure mineral physics volume edited by D. Rubie, T. Duffy and E. Ohtani (Fei et al., 2003).We further examined the MgO, Au, and Pt pressure scales using an externally-heated diamond-anvil cell up to 60 GPa and 1150 K. The experiments were performed for gold + MgO mixtures in a neon medium and for Pt + NaCl mixture at the GSECARS sector of APS. Above 900 K and below 27 GPa, both Jamieson's and Shim's gold scales are consistent with the MgO scale within 0.5 GPa, whereas Anderson's gold scale under estimates pressure significantly (1 GPa) compared with the MgO scale. A greater magnitude of discrepancy is found above 30 GPa: 300 K isotherms for gold are discrepant by 1 GPa with the isotherm for MgO. In addition, thermal pressure calculated from the gold scales is systematically smaller than that from the MgO scale by 3 GPa. Possible reasons for the discrepancies at high temperature include (1) electronic and anharmonic contributions, (2) different equations of state used, and (3) differential stress in the samples. The results will be presented in the AGU Fall meeting, 2003 (Shim et al., 2003).

We will continue to perform in-situ X-ray diffraction measurements of the commonly used pressure standards and provide a critical evaluation of these pressure scales. In addition, we plan to establish a spectroscopically-based pressure scale at high temperature. Isotopic pure $_{13}$C diamond and cubic BN will be used as pressure calibrants. The Raman shifts of $_{13}$C diamond and cubic BN as a function of pressure (up to 100 GPa)at high temperatures (up to 1173 K) will be established by simultaneous Raman spectroscopic measurements and X-ray diffraction.

Community involvement and broad impact

We will actively collaborate with people who are interested in the pressure scale problem and can provide unique expertise to enhance our program. We previously distributed a program for calculating pressures at high temperature using the existing equations of state for the commonly used pressure standards. Many users who conduct high-PT experiments in the synchrotron facility have used the program for pressure determination. The updated version that incorporates our latest results will be distributed to replace the early version. It would be appropriate to organize a workshop on pressure calibration in the near future to assess our understanding of the uncertainties in pressure determination at high temperature. Although we have made significant progress in understanding the discrepancy among the existing pressure scales, establishing an absolute pressure scale at high temperature requires redundant equation-of-state measurements under simultaneous high pressures and temperatures. We propose to organize a pressure-calibration workshop that aims to establish an absolute pressure scale at high temperature. Significant inputs from the "elasticity" group are needed to maximize the impact of the workshop.

Publications

Fei, Y., J. Li, K. Hirose, W. Minarik, J. Van Orman, C. Sanloup, T. Komabayashi, and K.Funakoshi, A critical evaluation of pressure scales at high temperatures by in-situ X-ray diffraction measurements, in *the High Pressure Mineral Physics volume*, special PEPI issue (eds. D. Rubie, T. Duffy and E. Ohtani), in press, 2003.

S.-H. Shim, M. Frank, Y. Fei, and R. Jeanloz, Comparison of gold and MgO pressure scales at 22-56 GPa and 300-1150 K and its implications for mantle models, *EOS*,AGU Fall meeting, 2003.

C.6 New Projects and Workshops

In September 2003, the Infrastructure Development Committee issued a call to the COMPRES community for proposed new initiatitives for technological projects that would contribute to the COMPRES mission.

In response to this call, eight new initiatives were proposed for Year #3. Following review of these initiatives by the Infrastructure Development and Facilities Committees, the Executive Committee decided to initiate one new project and to sponsor Workshops to explore in more detail three of the other initiatives.

The one new project proposed for Year #3 is

Nuclear Resonant Scattering at High Pressure and Temperature: A New Capability for the COMPRES Community

[W. Sturhahn, Advanced Photon Source; J. Bass, University of Illinois at Urbana-Champaign; G. Shen, University of Chicago]

Summary

Nuclear Resonant Scattering (NRS) techniques are relatively new applications of synchrotron radiation for determining the properties of condensed matter. The infrastructure development outlined here is aimed at creating state-of-the-art NRS techniques for characterizing the properties of materials under the high-P-T conditions of planetary interiors. The development and advance of two related techniques are of interest: Synchrotron Mössbauer Spectroscopy (SMS) and Nuclear Resonant Inelastic X-ray Scattering (NRIXS). The applications include (but are not limited to) determining the valence states of iron, the phonon density of states, sound velocities, detection of melting, and detection of high-spin low-spin transitions, all for iron-bearing compounds of geophysical interest. It has recently been shown that SMS and NRIXS can be performed using small samples at high pressures in a diamond anvil cell.

We propose to develop a new experimental capability at sector 3-ID of the Advanced Photon Source and to make it accessible to the COMPRES community. We will develop laser heating of samples in a diamond anvil cell (DAC) for NRS experiments. The motivation is to characterize materials by NRS while they are at simultaneous high P-T conditions that are similar to those in the deep Earth. The feasibility of NRIXS and SMS at high pressure has already been established. Iron-containing compounds were successfully studied at pressures above 1 Mbar. However, some of the crucial issues in Earth and planetary science can only be addressed if high- P experiments are performed at simultaneous high-temperature conditions. Such a capability would be unique worldwide. Thus far, a laser heating system has been purchased and partially tested.

The specific tasks to be performed under this proposal are

- Complete design, construction, and commissioning of a laser heating system suitable for NRS experiments at sector 3-ID of the Advanced Photon Source.
- Development of refined high-pressure equipment and techniques for NRS.
- Development of user-friendly instrumentation and controls to facilitate easy access of the COMPRES community to this new capability.
- Education and outreach to the Earth-sciences community to encourage the use of NRS, to develop productive collaborations, and to address common experimental issues confronting users.

For the successful and timely completion of these tasks, we request a full-time post-doctoral scientist who will be dedicated to developing the high-P-T infrastructure, serving its users, and building a user base among the COMPRES community. We also request funds for workshops and necessary travel expenses. The completion of the tasks outlined in this proposal, will provide a truly unique capability for the study of vibrational dynamics and valence states of Fe under high pressure and high temperature conditions. No other facility in the world exists for such experiments. This enhancement of sector 3-ID capabilities presents an ideal synergy with developments at the beamlines of GSECARS and HPCAT, which focus on diffraction and XAFS-type methods.

This collaborative project will be funded via the University of Illinois at Urbana-Champaign (see details in budget discussion below).

Workshops:

COMPRES has proposed funding for three Workshops in 2004:

Dual Beam Focused Ion Milling System for TEM Foil Preparation + 3D Chemical Analysis and Imaging for COMPRES Users

Organizer: H. Green, University of California at Riverside.

As this Workshop will be held in late March 2004, the funding for it will come from the Year #2 budget of COMPRES.

Stress characterization of composites in the diamond anvil cell

Organizer: A. Kavner, University of California at Los Angeles.

Synchrotron megabar-pressure single-crystal XRD (MPSXD) program

Organizer: P. Dera, Carnegie Institution of Washington

D BUDGET REQUEST FOR YEAR #3

Budget Request for Year #3 [May 1, 2004 through April 30, 2005]. Details may been seen in following NSF 1030 Forms and the associated budget justification pages.

The COMPRES budget request for $2,200,000 for Year #3 [May 1, 2004 to April 30, 2005] is comprised of three major components (units of $K, which include fringe benefits and indirect costs):

Community Facilities

West Coast Synchrotron Facilities (R. Jeanloz, University of California at Berkeley)

$193K Operational budget

$115K Equipment acquisition at Advanced Light Source

Diamond-anvil cell facilities at the National Synchrotron Light Source [H-k. Mao, R. Hemley, Carnegie Institution of Washington]

$328K Operational budget

Multianvil Press Facility at the NSLS [D. Weidner, M. Vaughan-Stony Brook University]

$318K Operational budget

Neutron Studies [N. Ross, Virginia Polytechnic Institute and State University]

$70K Operational budget

Infrastructure Development Projects

$158K Multi-anvil cell assembly development [K. Leinenweber, J. TyburczyArizona State University]

$107K Development of the Laser-Heated Diamond Anvil Cell [T. Duffy, Princeton University, G. Shen and D. Heinz, University of Chicago]

$106K Absolute Pressure and Temperature Calibration [I. Getting, University of Colorado]

$70K Brillouin Spectroscopy at the Advanced Photon Source [J. Bass, University of Illinois and G. Shen, University of Chicago]

$60K Nuclear Resonant Scattering at High P & T:A New Capability for the COMPRES Community [W. Sturhahn, Advanced Photon Source, J. Bass, University of Illinois and G. Shen, University of Chicago]

Other COMPRES Activities

This component of the budget is devoted to two principal areas of activities:

$236K **Workshops, Meetings, Education & Outreach, Publications** which includes:

- $60K Annual Meeting
- $70K Workshops (see list below*)
- $14K Education & Outreach
- $64K Student Interns
- $14K Publication of brochure

$439K **Central Office** which includes:

- $363K Salaries and fringe benefits
- $35K Materials and Supplies
- $41K Travel (including that of Advisory Committee, Executive Committee and Standing Committees)

$675K Total for Other COMPRES Activities

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*COMPRES has proposed funding for several Workshops in 2004, including:

$14K Dual Beam Focused Ion Milling System for TEM Foil Preparation +3D Chemical Analysis and Imaging for COMPRES Users. Organizer: H. Green, University of California at Riverside.

`$20K Stress characterization of composites in the diamond anvil cell Organizer: A. Kavner, University of California at Los Angeles.

$20K Synchrotron megabar-pressure single-crystal XRD (MPSXD) program Organizer: P. Dera, Carnegie Institution of Washington