

**Year 5 Annual Progress Report of the Consortium for
Materials Properties Research in Earth Sciences
(COMPRES)**

April 2007

Acronyms Used in COMPRES annual REPORT to the NSF—APRIL 2007

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| ADX | Angular Dispersive X-ray analysis |
| ALS | Advanced Light Source, a synchrotron facility at LBNL |
| ANL | Argonne National Laboratory, IL |
| APS | Advanced Photon Source, a synchrotron facility at ANL |
| BNL | Brookhaven National Laboratory, NY |
| CALIPSO | CALifornia High Pressure Science Observatory, a high pressure beamline at the |
| CAT | Collaborative Access Team, a group that manages a sector (two beamlines) at the APS |
| CDAC | Carnegie/DOE Alliance Center |
| CHESS | Cornell High Energy Synchrotron Source, a synchrotron facility at Cornell University, NY |
| COMPRES | Consortium for Materials Properties Research in the Earth Sciences |
| CU or CUP | Contributing User (Program), the facility access system at the NSLS |
| CVD | Chemical Vapor Deposition |
| DAC | Diamond-Anvil Cell |
| DMR | Division of Materials Research at NSF |
| DOE | Department of Energy |
| EAR | Division of Earth Sciences at NSF |
| EDX | Energy Dispersive X-ray analysis |
| ERL | Energy Recovery Linac |
| GSECARS | GeoSoilEnviroCARS, a CAT at the APS dedicated to earth science research |
| GU or GUP | General User (Program), a facility access system for the general scientific community at the APS |
| HPCAPS | High Pressure Consortium at the Advance Photon Source, an organization for discussion |
| HPCAT | High Pressure CAT at the APS |
| IF | Instrumentation and Facilities Program in EAR at NSF |
| LANL | Los Alamos National Laboratory, NM |
| LANSCE | Los Alamos Neutron Science Center, now known as the Lujan Center |
| LBNL | Lawrence Berkeley National Laboratory, CA |
| LCLS | Linac Coherent Light Source, a planned x-ray free-electron laser at SLAC |
| LLNL | Lawrence Livermore National Laboratory, CA |
| LVP | Large-Volume Press (equivalent to MAC) |
| MAC or MAP | Multi-Anvil Cell or Multi-Anvil Press (equivalent to LVP) |
| NSF | National Science Foundation |
| NSLS | National Synchrotron Light Source, a synchrotron facility at BNL |
| ORNL | Oak Ridge National Laboratory, TN |
| PE Cell | Paris-Edinburgh Cell |
| PRT | Participating Research Team, a group managing a beamline at the NSLS |
| SLAC | Stanford Linear Accelerator Center |
| SNAP | Spallation Neutrons At Pressure, a planned beamline at the SNS |
| SNS | Spallation Neutron Source, a neutron facility under construction at ORNL |

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Part A: Project Description

A. 1. Introduction and Background

COMPRES facilitates the operation of high-pressure beamlines for Earth Sciences at national synchrotron and neutron facilities, supports the development of new technologies for high-pressure research, and advocates for science and educational programs to the various funding agencies.

The goal of COMPRES is to enable Earth Science researchers to conduct the next generation of high-pressure science on world-class equipment and facilities. COMPRES does not fund research projects, rather it works to ensure that projects can be conducted. Individual research projects or collaborative research projects, such as the Grand Challenge, are formally independent from the COMPRES core grant; however, they are intimately related intellectually as they give prime examples of the scientific problems that can be addressed using the facilities operated and the infrastructure developed by COMPRES.

COMPRES works to enhance the access to appropriate resources and infrastructure that exceed those available to the individual researcher. With a broad user base, this organization is facilitating the next generation of science.

Research in mineral physics is essential for interpreting observational data from many other disciplines in the Earth Sciences, from geodynamics to seismology to geochemistry to petrology to geomagnetism to planetary science, and extending also to materials science and climate studies. The field of high-pressure mineral physics is highly interdisciplinary. Mineral physicists do not always study minerals nor use only physics; they study the science of materials which comprise the Earth and other planets and employ the concepts and techniques from chemistry, physics, materials science, and biology.

Such interdisciplinarity also has major international dimensions, with attendant synergistic and competitive aspects. A dramatic example of this has occurred during the past two years. In 2004, a new post-perovskite phase of MgSiO_3 was discovered by *in situ* high-pressure experiments in Japan (Murakami, Hirose, Kawamura, Sata and Ohishi, *Science*, 304, 855, 2004). The importance of this discovery for the deep earth was immediately recognized by the mineral physics communities on 3 continents, leading to rapid experimental confirmation and theoretical (first-principles) exploration, including European and US contributions.

These developments have had immediate and profound impact on multidisciplinary studies of the deep mantle of the Earth [see feature article by Lay et al in the 4 January 2005 issue of EOS].

The ability to the US mineral physics community to respond immediately to the post-perovskite discovery was in large part a reflection of the success of COMPRES in greatly expanding the size, breadth and technical capabilities of the US high-pressure community; the US now competes on equal footing with the Japanese and Europeans who had a considerable jump on this country at the time of the birth of COMPRES. Indeed, the successes flowing from the rapid growth of COMPRES are already feeding back into the international community, as documented in A.3 below under international linkages.

The field of high-pressure earth and planetary sciences has changed dramatically over the past decade. Increasingly sophisticated tools are being used to investigate the properties of matter under the extreme pressure and temperature conditions of the Earth and other planetary interiors. As a prime example, the capabilities of modern synchrotron and neutron sources have presented enormous opportunities for new types of experimentation at high pressure. In parallel with these advances in large, centralized facilities, new types of high-pressure devices, of both the diamond-anvil and multi-anvil types, have been developed to take advantage of them. Similar progress has been achieved in the computational power for calculations of mineral properties, and new facilities to perform neutron scattering studies at high pressure are emerging. As a result, it is now possible to do experiments and perform simulations that were not dreamed of 10 years ago.

Many of these exciting advances and prospects for the future have been described in the report "Current and Future Research Directions in High-Pressure Mineral Physics". This report is an outgrowth of the discussions and results of a workshop on "A Vision for High Pressure Earth and Planetary Sciences Research: The Planets from the Surface to the Center" which was held on March 22-23, 2003 in Miami, Florida. The NSF Division of Earth Sciences commissioned and supported this workshop, which was organized by Jay Bass and Donald Weidner of COMPRES (Consortium for Materials Properties Research in Earth Sciences) and attended by fifty-six scientists from throughout the world. The Miami Workshop was held to identify the most promising areas for future discovery, and areas that are ripe for future technological breakthroughs. The report was edited by Jay Bass based on input from the participants at the Workshop, and is intended to be a statement by the high-pressure earth science community on the status of this field and some of its most exciting and challenging research directions for the near future. As such, it serves as the strategic science plan for COMPRES. A poster illustrating the central themes of the Bass report is shown below. Copies of both of these items are included in the Supplementary Documents for this proposal.

At the onset of the 21st century, mineral physicists find themselves with many challenging research problems and many exciting opportunities for research at high pressures and temperatures, made possible in large measure by the COMPRES-facilitated access to synchrotron and neutron facilities at the national laboratories of the Department of Energy. To exploit such technologies to pursue research in the Earth Sciences required a change in the culture of high-pressure experimental research. Until recently, the "cottage industry" model served as the primary mode of operation: a scientist worked with a few students and/or postdocs, together in a laboratory at their home institution.

COMPRES has changed all that in a very brief period of time for high-pressure geophysics because it has opened up these facilities in a way that makes them available to a broad cross section of the community in an affordable way. The emerging new paradigm demands a different strategy including advanced preparation of samples and experiments, weeks in “the field” (at the national facility), sleepless nights, and CDs full of data. Following the experimental runs at the beamline, the fatigued team returns home for weeks of data analysis. This new mode requires re-education to enable all scientists, from student to senior faculty, to effectively participate in this new culture. The community-based approach to organizing these scientific efforts adopted by the embryonic COMPRES community in 2001 has proven to be so successful that in just 4 brief years after the initial funding of COMPRES, leaders of the DOE synchrotrons consult with COMPRES about how best to implement the new DOE rules for operating their beamlines.

A.2 Research Themes and Scientific and Technological Advances

As indicated above, the strategic science plan for COMPRES and high-pressure mineral physics is well represented by the Bass Report of September 2004, a copy of which is Supplementary Documents.

This field has witnessed numerous discoveries and breakthroughs during the past decade. Along with breakthroughs comes not only the ability to understand more-complex phenomena, but also the ability to confront exciting challenges. In the previous section, we gave the example of how discovery of the post-perovskite phase was a mineral physics breakthrough that also invigorated other aspects of deep-earth geophysics. In the future, undoubtedly, many of these challenges will come from new observational studies of the Earth’s deep interior by other geophysical and geochemical disciplines that will similarly have important mineral physics dimensions.

This important interrelationship between mineral physics and the other disciplines is one of the main emphases in the September 2004 Report on “Cooperative Studies of the Earth’s Deep Interior [CSEDI]: Developments, Discoveries, Future”, which was the outcome of a Workshop in La Jolla, California on February 22-23, 2004.

As implied in Figure 1, the research areas pursued by scientists in the COMPRES community provide the physical mechanisms that allow explanation of Earth-based observations from ~~other~~ geophysical and geochemical studies in terms of the chemical and structural state of matter and dynamical processes active at depth. In the original 2001 COMPRES proposal to the NSF, three such linkages were cited:

Structure of the Earth’s transition zone
Water in the Earth’s interior
The Core-mantle boundary

In Part C of this proposal, we present 111 one-page summaries (One-Pagers) which provide highlights of the scientific and technological achievements of students, staff and faculty researchers using COMPRES-supported facilities, data produced from COMPRES programs, and developmental projects supported by COMPRES in its first era from 2002-2006. As is evident from even a cursory perusal of these One-Pagers, substantial progress has been achieved in the past 4 years in addressing the three linkages cited above.

From the One-Pagers in Part C, it is also clear that new research themes have emerged during the first era of COMPRES. We highlight these here, with reference to representative One-Pagers in Part C.

a. Research Themes

The last four years have witnessed great growth in established areas of high-pressure mineral physics as well as defined new frontiers. Stress is the primary agent for dynamics within the Earth, be it for earthquakes or for plate motions. Yet our ability to quantify stress at high pressure has not been met until recently. X-ray (or neutron) diffraction has provided the solution to this problem. A stressed crystal is elastically distorted, with consequent changes in its symmetry and small changes in its lattice spacings. The sum of the changes for many crystals of a polycrystalline material, measured by X-ray diffraction, can be used to define the elastic strain tensor for the material and, through its elastic constants, the stress can be calculated. The implementation of this type of measure has now become routine for both the diamond cell and the multi-anvil device. X-ray transparent pathways were required for this development, and have been developed primarily by COMPRES researchers during the past five years. The next phase is to improve the precision of these measurements from about 100 MPa to 10 MPa and their accuracy to be comparable to direct stress measurements at low pressure. These new measurement tools have stimulated new high pressure apparatus. The D-DIA and the RDA have emerged to provide constant strain rate and large strain environments at high pressure and temperature. New insights and constraints are now budding. The US has pioneered this area. Nearly 20% of the one-page summaries submitted by the COMPRES community deal with the measurement of stress. Many international labs are now building or planning their stress-based programs. The future promises to provide facilities that reach lower mantle conditions (P,T) and provide for rheological experiments at steady state for extended times and large strains, with the precision of stress and strain measurements that are currently possible only with low pressure devices.

Elastic wave velocities are the principal observational exploration tool available to Earth sciences. The resulting red and blue tomography maps, radial velocity profiles, etc., all require laboratory data to provide a basis for interpretation in terms of temperature and composition. Great technical growth in this capability has been a hallmark of the past five years. Brillouin spectroscopy of samples in a diamond anvil cell, with simultaneous x-ray diffraction analysis is now feasible. Ultrasonic acoustic measurements are now possible to both high pressure and high temperature in multi-anvil devices. P, T

environments equivalent to those found in Earth down to the top of the lower mantle can be created for ultrasonic measurements. Plastic deformation of the sample can be simultaneously monitored. Theory has now matured to provide key predictions of properties that cannot be reached in the laboratory. Tested in regions where experiments are possible, computational mineral physics promises to become a key tool for the entire Earth. Nearly 25% of the ‘one pagers’ relate to elastic properties. The experimental tools, first developed in the US, are now being exported to many laboratories around the world.

With the tools developed during the past five years, to a large degree by COMPRES, new possibilities arise to augment our understanding of liquids. Radial distribution functions and infrared spectroscopic studies give us information on the structure of liquids. X-ray absorption analysis can yield the density of the liquid. Ultrasonic or Brillouin measurements can yield information about the stress – strain relations in liquids; falling sphere observations shed light on the viscosity of liquids. Approximately 10% of the ‘one pagers’ are pertinent to the study of liquids and amorphous materials. These new tools are poised for exciting new science that will help us understand liquids in the Earth.

Crystal structure is fundamental to our understanding of the properties of solids. From rheology to phase equilibria, the structure of the material plays a controlling role. Diffraction from both x-rays and neutrons, as well as spectroscopy, give insights into the nature of the crystal (or amorphous) structure. 30% of the ‘one pagers’ deal with the definition of the crystal structure. While tools in this area are fairly mature, their application at very high pressure and the need to exploit them continues as the importance of the details of crystal structure continues to be manifested. In the first four years of COMPRES, we have been able to query the effects of the spin state of iron as a function of pressure, temperature, and chemical composition. This atomistic property has the potential of rewriting the physical properties of the deep Earth. Crystal structure studies also form the basis of exploring phase transformations. While we presume that most of the depth-induced phase transformations have been identified, exploration of the details of such transitions remains important to understand the state of the Earth’s interior.

b. Scientific and Technological Highlights of COMPRES II

During the COMPRES I era [2002-2007], substantial advances in both technology and scientific productivity have been achieved at both the community facilities operated with COMPRES support and from the infrastructure development projects nurtured and funded by COMPRES. This remarkable progress would not have been possible without the leadership of the key project directors from the high-pressure, mineral physics community and without the funding from COMPRES and associated science-based grants such as the COMPRES Grand Challenges [e.g., A.10, B.1.d and B.2.c].

In the series of bullets below, we highlight some of these advances and refer the reader to the relevant sections of Part B of this proposal for details and references.

- A team led by S. Speziale demonstrated the importance of the high-low spin transition of iron in magnesiowustite to the properties of the Earth's lower mantle. J-f. Lin and colleagues further discovered that the spin-state transition is associated with a rather drastic change in the material's elastic properties. [B.1.a and B.2.d]
- Using the x-ray diffraction (XRD) microprobe technique at X17C at the NSLS, a team led by J. Chen discovered a CF-type and a CT-type polymorph of chromite composition in a shock-metamorphosed chromite in Suizhou meteorite. [B.1.b]
- Stishovite is known to transform to orthorhombic CaCl₂-type structure at 50±3 GPa which is driven by an instability of an elastic shear modulus. Shieh et al. used lattice strain measurements under nonhydrostatic compression in a diamond anvil cell to examine dense SiO₂ pressure up to 60 GPa, which provided direct experimental evidence for softening of the elastic shear modulus. [B.1.b]
- A. Goncharev and colleagues measured optical spectra of single crystals of (Mg,FeO) to pressures exceeding 60 GPa. They observed enhanced absorption in the mid- and near infrared spectral range, effectively blocking much of the light compared with low pressure conditions, which appears to require high thermal conductivities in order to mitigate huge temperature gradients calculated for the case of constant thermal conductivity in the Earth. [B.1.c]
- A team led by J-f. Lin measure sound velocities of hexagonal close-packed iron (hcp-Fe) were measured at pressures up to 73 GPa and at temperatures up to 1700 K with nuclear inelastic x-ray scattering at the beamline APS in a laser-heated diamond anvil cell. The compressional-wave velocities (V_p) and shear-wave velocities (V_s) of hcp-Fe decreased significantly with increasing temperature under moderately high pressures and could not be fitted with Birch's law, suggesting that there are more light elements in Earth's core[B.2.d]
- Construction of a completely new dedicated high-pressure facility on the superbend beamline [12.2.2] at the ALS, including an integrated laser-heating system. [B.1.a]
- Generation of ultrahigh (>200 GPa) pressures using single-crystal diamonds synthesized by chemical vapor deposition (with EAR funding via a complimentary Grand Challenge grant). This CVD project was part of a collaboration between the Carnegie Institution of Washington and the Los Alamos National Laboratory, which produced diamonds from which are the hardest and toughest known crystals to date [Yan, Mao, Li, Qian, Zhao and Hemley, *Phys. Stat. Solidi (a)*, 204, r25, 2004]. [B.1.b]
- Equipment upgrades on the dedicated ultraviolet beamline (U2A) at the NSLS have expanded the experimental capabilities for infrared studies at high pressures and led to a 10-fold increase in user proposals over a 4 year period. [B.1.c]
- Investments of more than \$1.6 M in funding from the NSLS, COMPRES/CHiPR, and Stony Brook University enabled the construction of a new hutch on the superconducting

wiggler beamline (X17) at the NSLS, which led to a doubling of the number of experimental runs which could be performed annually on both the DAC and MAC facilities. [B.1.b and B.1.d]

- Deformation of rocks and minerals: New experimental technologies for rheological experiments at high pressures and temperatures. [B.1.d]

A new high pressure deformation apparatus D-DIA has been married to the synchrotron x-ray source at the multi-anvil beamline (X17B2) at the NSLS by D. Weidner and his colleagues. The D-DIA can generate pressures of 8 GPa in an apparatus of cubic-anvil geometry. An identical instrument has been installed at GSECARS under Y. Wang and colleagues and a third D-DIA is in the final stages of construction at UC Riverside, where H. Green is developing the assemblies and techniques to study deep earthquake-generating shearing instabilities associated with phase transformations and dehydration reactions with *in situ* experiments [see One Pagers by Jung et al and Zhang et al in Part C].

A rotational Drickamer-type apparatus [RDA] for high-pressure, temperature, large strain rheological experiments has been developed by S. Karato and his team at Yale University and operated on the X17B2 beamline at the NSLS.

Both of these technological developments benefited from funding from COMPRES and the associated Grand Challenge for Rheological Studies also provided by EAR.

A deformation T-cup apparatus is being developed for installation on the X17B2 beamline at the NSLS to enable deformation studies to be performed with monochromatic X-radiation, in an Infrastructure Development project jointly supported by COMPRES, the DOD-DURIP program and Stony Brook University. [B.2.1].

- Ultrasonic interferometry measurements of elastic wave velocities in conjunction with synchrotron X-radiation have been pioneered by B. Li and colleagues in a DIA-cubic anvil apparatus on the X17B2 beamline to simultaneous pressures of 10+ GPa and temperatures above 1000K. These techniques have now been adopted at other synchrotron facilities [GSECARS at APS and at Spring-8 in Japan] and extended to higher pressures in MAC apparatus. [B.1.d]
- Standardized cells for multi-anvil experiments at both home laboratories and national facilities have been developed and tested at the Arizona State University and are now available for purchase at a subsidized rate. Pressure calibration in these cells is being conducted at synchrotron beamlines such as those operated by GSECARS at the APS [see also Multi-Anvil Workshop reported A.6 and B.2.b].
- A team led by H-k Mao used large gem crystals moissanite (silicon carbide) as anvils

in a panoramic cell with $>1\text{mm}^3$ sample volumes and demonstrated the feasibility of high-pressure, single-crystal neutron diffraction experiments. [B.1.e]

- A new Brillouin spectrometer has been developed by J. Bass and his team at the University of Illinois at Urbana-Champaign (UIUC) and installed on sector 13-BM-D at the APS in collaboration with the staff of GSECARS. See details in the November 2005 issue of the COMPRES newsletter. This infrastructure development project was supported by funding from COMPRES, the Elasticity Grand Challenge via EAR, UIUC and GSECARS. These new facilities will enable measurement of acoustic wave velocities to be performed on mineral specimens to pressures of 50+ GPa and, soon, at simultaneously high temperatures; this unique facility is now being copied at the Spring-8 synchrotron in Japan. [B.2.c]

- The facilities at the 3-ID beamline at the APS for nuclear resonant inelastic X-ray scattering and synchrotron Mössbauer spectroscopy have been interfaced with high-pressure and laser-heated diamond anvil cells, with partial personnel support from COMPRES. [B.2.D].

- The phenomenon of Johnson noise thermometry offers the possibility of absolute calibration of thermocouples, a serious experimental impediment for high-pressure research since its inception. A prototype system, funded by COMPRES, has been completed at the University of Colorado. This new equipment has now been transferred to the 13-BM beamline at the APS for testing in a multi-anvil high pressure cell under the support of COMPRES and GSECARS. [B.2.f]

- A gas-loading system for diamond-anvil cells has been designed and is being constructed at the APS with the support of COMPRES and GSECARS. This system will be disseminated to other synchrotron facilities and also be accessible to investigators from home laboratories to load gasses into their DACs for experiments in their home laboratories. [B.2.i]

- A new generation of multi-anvil deformation apparatus is being developed at the APS to enable rheological experiments on large-volume specimens to be performed to megabar pressures. This development project is jointly supported by COMPRES and GSECARS. [B.2.j]

- By adapting the nanofabrication technology developed by F. Hellman at UC Berkeley, the team of A. Navrotsky at UC Davis has demonstrated the feasibility of obtaining reliable calorimetry data on specimens from the milligram scale (from MAC experiments) to the microgram scale (from DAC experiments). [B.2.k].

- A team led by P. Dera and M. Nicol was awarded a Major Research Instrumentation grant by the NSF for “Development of Six New Approaches for Micro-focus Single-Crystal X-ray Diffraction for Materials Structure Research at Synchrotrons”. This proposal was an outgrowth of a COMPRES-sponsored workshop convened by Dera and C. Prewitt at the APS in November 2004. Following development of this new

technology, equipment will be installed on the synchrotron beamlines supported by COMPRES at the ALS and the NSLS, as well as on the GSECARS beamlines at the APS.

- The National Synchrotron Light Source at Brookhaven National Laboratory has approved four Contributing User Agreements with COMPRES to operate high-pressure facilities at the superconducting wiggler X-ray beamline (X17) and the ultraviolet beamline (U2A), with teams from the Carnegie Institution of Washington, Stony Brook University and the University of Chicago.

A.3 COMPRES—Overview and Role of Consortium

COMPRES, the Consortium for Materials Properties Research in Earth Sciences, was formed in part as a response to these new scientific and technological opportunities and developments, and this new style of conducting high-pressure science. Starting with a Town Meeting at the Fall 2000 AGU Meeting in San Francisco organized by the AGU Mineral and Rock Physics Committee, the planning process began in earnest with a workshop at the Scripps Institution of Oceanography in La Jolla in February 2001 and culminated in a successful proposal to the NSF Division of Earth Sciences in August 2001. In May 2002, a Cooperative Agreement was promulgated which projected funding for COMPRES for a five-year period to April 2007.

COMPRES is a community-based consortium that supports research in the materials properties of earth and planetary interiors with particular emphasis on high-pressure science and technology and related fields. The Consortium currently has 50 members that are educational or governmental institutions in the U. S. with research and educational programs in the science of Earth materials. There are also 28 foreign affiliate institutions.

COMPRES is charged with the oversight and guidance of important high-pressure laboratories at several national facilities, such as synchrotrons and neutron sources. It facilitates the operation of beam lines, the development of new technologies for high-pressure research, and advocates for science and educational programs to the various U. S. funding agencies, including NSF, DOE, DOD and NASA. The community-wide organization of mineral and rock physics introduced by COMPRES is directly analogous to centralization of efforts in other geophysical sciences, such as the coordination of seismic data distribution and instrument deployment orchestrated by IRIS, the Incorporated Research Institutions of Seismology.

The two major COMPRES programs are overseen by two Standing Committees for Community Facilities and for Infrastructure Development Projects. These Standing Committees are elected by the representatives of the U. S. member institutions of COMPRES.

COMPRES supports the operations of high-pressure beamlines at synchrotrons to provide access and support to faculty, students and staff scientists in the earth science community. These operations include: (1) Diamond-anvil facilities at the National Synchrotron Light Source [NSLS] of the Brookhaven National Laboratory; (2) Multi-anvil facilities at the NSLS; and (3) Diamond-anvil facilities at the Advanced Light Source [ALS] of the Lawrence Berkeley National Laboratory. COMPRES also supports a neutron studies initiative to cultivate scientific interest in exploiting the new opportunities to come available soon at the Spallation Neutron Source [SNS] of the Oak Ridge National Laboratory and coordinates its activities with those of the GeoSoilEnviroCARS [GSECARS] program at the Advanced Photon Source of the Argonne National Laboratory.

In addition to the operation of community facilities, COMPRES supports infrastructure projects to promote the development of new technologies for high-pressure research, for use in both laboratories in our home institutions and at the national laboratories. Current examples of such infrastructure development projects include: (1) Absolute pressure and temperature calibration; (2) Multi-anvil cell assembly development; (3) Brillouin spectrometer for the APS; (4) Nuclear resonant inelastic X-ray scattering at high pressure and temperature; (5) New CO₂ laser-heated diamond-anvil cell; (6) COMPRES environment for automated data analysis, a software development project; (7) Technical support for dual beam focused ion milling facility for TEM foil preparation; (8) Gas-loading system for diamond-anvil cells at the APS; (9) Development of next generation multi-anvil module for megabar research; (10) Calorimetry-on-a-chip; and (11) Monochromatic X-ray side station at the multi-anvil beamline of the NSLS.

While COMPRES derives its primary financial support from the Instrumentation and Facilities Program in the Division of Earth Sciences of the NSF, it leverages the enormous investment of the DOE in constructing and supporting the operation of its national laboratories, notably those at Brookhaven, Argonne, Lawrence Berkeley, and Oak Ridge National Laboratories. In addition, members of the COMPRES community have been very successful in obtaining other funding from the NSF, DOE and DOD to enhance the opportunities for research in high-pressure mineral physics, as detailed in Section A.10 below.

Under separate funding from the NSF Division of Earth Sciences, scientists in the COMPRES community are pursuing three Grand Challenge collaborative research programs: Growth of large synthetic diamonds by chemical vapor deposition; Rheology of earth materials; Elasticity of earth materials—all at high pressures and temperatures. While these Grand Challenge programs are formally independent from the COMPRES core grant, they are intimately related intellectually as they give prime examples of the scientific problems that can be addressed using the facilities operated and the technological developments funded by COMPRES. [see details of progress and future plans in section A.10 below].

Communication within the mineral physics community includes monthly letters from the President, quarterly newsletters, an active website [<http://www.compres.us>], and an Annual Meeting. The 2006 COMPRES Annual Meeting at the Snowbird Alpine Village in Utah attracted 101 active participants, including many young scientists; this photo clearly demonstrates the vitality and diversity of the community of mineral physicists. This meeting included focus sessions on the minerals and volatiles in the mantle, the core and planetary evolution, all of which included keynote talks by leaders in allied geoscience disciplines (seismology, geodynamics, geochemistry, planetary science) followed by group discussion, as well as reports from the Community Facilities and Infrastructure Development projects, breakout session on special topics and poster presentations highlighting the most exciting recent scientific achievements.

In the October 4, 2005 issue of EOS, an article on “The Future of High-Pressure Mineral Physics” was published by the PI on this proposal on behalf of the COMPRES community; a copy is included in Supplementary Documents.

COMPRES relationship to national facilities of the DOE

The community-based approach to organizing these scientific efforts adopted by the embryonic COMPRES community in 2001 has proven to be so successful that in just 4 brief years after the initial funding of COMPRES, leaders of the DOE synchrotrons consult with COMPRES about how best to implement the new DOE rules for operating their beamlines.

- NSLS:

COMPRES provides funding for operations and equipment upgrades at high-pressure multi-anvil and diamond-anvil X-ray facilities and infrared DAC facilities at the NSLS, under Contributing User Agreements negotiated with the NSLS on behalf of the COMPRES community.

Leading members of the COMPRES community are consulted and engaged in planning and design of the new synchrotron facilities at the Brookhaven National Laboratory, NSLS II [which was highlighted as one of the key new innovations in the American Competitiveness Initiative, February 2006].

- ALS:

COMPRES provides funding for operations and equipment upgrades at high-pressure diamond-anvil X-ray facilities at ALS, as a partner in an Approved Program with the University of California and the Lawrence Livermore National Laboratory.

- GSECARS at the APS:

High pressure mineral physics research at synchrotron X-ray facilities in the U. S. is managed by two organizations supported largely by NSF and DOE:

GeoSoilEnviroCARS (GSECARS) at the University of Chicago and COMPRES.

GSECARS is a national user facility for frontier research in the earth sciences using the high-brilliance, high energy synchrotron radiation at the third generation Advanced Photon Source (APS), Argonne National Laboratory.

Together, COMPRES and GSECARS provide strategically vital support to the operations of high-pressure beamlines at synchrotrons, including funding of beamline scientists at the facilities and access and assistance for students, postdocs, etc. in the earth science community. All the beam time at GSECARS and at the COMPRES-supported components at the NSLS and the ALS is open to the general community through proposals to the General User Programs [GUP] at each facility. There are at least two distinct communities served by operations of high-pressure facilities at the national laboratories: (1) General group of users in geosciences [students, postdocs, staff]; (2) Developers of new techniques or those who adapt new technologies developed in other disciplines. The operators of the high-pressure facilities at the national laboratories have an obligation to serve each of these distinct and important communities.

GSECARS and COMPRES collaborate closely through coordination of community development activities and the design, construction and operation of advanced instrumentation through COMPRES-supported infrastructure projects. For example, three major technological tools supported by the COMPRES Infrastructure Development program are being installed at GSECARS or associated space at the APS: (1) a Brillouin spectroscopy system (installed at GSECARS and undergoing commissioning); (2) a CO₂ laser heating system (under development at GSECARS); and (3) a gas-loading facility for diamond-anvil cells (in design phase). X-ray optics and software developed at GSECARS are being used at the COMPRES-operated x-ray beamlines at the NSLS. The current chair of the COMPRES Facilities Committee (Mark Rivers) is co-Director of GSECARS, while Robert Liebermann, the President of COMPRES, is a GeoCARS representative on the CARS Board of Governors.

GSECARS has agreed to be included in the COMPRES evaluation of high pressure mineral physics facilities. For this purpose, the elected COMPRES Facilities Committee visited GSECARS in October 2005 and an advisory report was submitted in December 2005 to both GSECARS and the COMPRES Executive Committee.

A joint statement of the relationship between COMPRES and GSECARS was prepared by the Principal Investigators of the two organizations in January 2006 and endorsed by the Program Director of the Instrumentation and Facilities Program in EAR at the NSF.

High Pressure Summit Meeting

In September 2005, COMPRES convened a meeting among a number of organizations funded by the NSF and/or the DOE which are engaged in developing and operating facilities at national laboratories for high-pressure mineral physics research, including the following:

COMPRES: COnsortium for Materials Properties Research in Earth Sciences

GSECARS: GeoSoilEnviron Consortium for Advanced Radiation Sources

HPCAT: High Pressure Collaborative Access Team

SNAP: Spallation Neutrons and Pressure at the Spallation Neutron Source [SNS]

ALS: High-Pressure Partners at the Advanced Light Source

LLNL: Lawrence Livermore National Laboratory

LANSCE: Los Alamos Neutron Science Center

CHESS: Cornell High Energy Synchrotron Source

LCLS: Linac Coherent Light Source at the Stanford Linear Accelerator Center [SLAC]

CDAC: Carnegie/DOE Alliance Center

The objectives of the meeting were to describe:

1. The shared and broad missions for high-pressure mineral physics.
2. The structure and responsibilities of the various organizations.
3. The relative roles in the high-pressure community.

4. Current funding status and future needs.

This meeting was held on September 24-25, 2005 in Ronkonkoma, Long Island, New York. As an outcome of this meeting, the attendees prepared a report to the NSF and DOE Program Managers and presented this report in person at the NSF on November 29, 2005.

COMPRES linkages with international programs

The membership of non-US institutions as foreign affiliates of COMPRES has grown from 0 in 2002 to 28 in 2007, including representation from Australia, Canada, China, France, Germany, Korea, Netherlands, Russia, Switzerland, Taiwan, and United Kingdom.

By the very nature of the desire to access synchrotron and neutron facilities for high-pressure experimentation, COMPRES is inherently connected to internationally to colleagues and research opportunities at the specialized laboratories in France [ESRF and ILL in Grenoble], Japan [Spring-8 and the Photon Factory], the United Kingdom [ISS and Daresbury], and Germany [HASYLAB]. At the same time, the COMPRES-supported beamlines are open to researchers from all over the world.

In addition to the international workshops supported by COMPRES and cited below in A.6, colleagues from France, the United Kingdom, Germany, Israel, Japan, China and Australia have attended the COMPRES Annual Meetings (often to give keynote talks) and workshops.

Indeed, the successes flowing from the rapid growth of COMPRES are already feeding back into the international community, as noted by two examples: (a) the mineral physics community of France is currently organizing itself along the lines of COMPRES because of the success of the “grass-roots” structure of COMPRES. [P. Raterron et al: *Presse Instrument National couplée au Synchrotron-PINS*]; and (b) in Germany, scientists in Earth Sciences, Materials Science and Solid State Chemistry have obtained funding for a Priority Program of the Deutsches Forschung Gemeinschaft [B. Winkler et al: “Strukturen und Eigenschaften von Kristallen bei extreme hohen Drücken und Temperaturen”].

A.4 Member Institutions of COMPRES, New Faculty in Mineral Physics and Awards for Mineral Physicists

Member Institutions

As already indicated above, COMPRES is a community-based consortium that supports research in the materials properties of earth and planetary interiors with particular emphasis on high-pressure science and technology and related fields.

Educational or governmental institutions that are chartered in the United States with research and educational programs in high-pressure research in the science of Earth materials are eligible to be members of COMPRES. At the time of the original proposal to the NSF in August 2002, there were 18 institutions represented in the list of Principal and Co-Principal Investigators.

The Consortium currently has 50 US members. There are also 28 foreign affiliate institutions. These institutions are listed in a table in the Supplementary Documents for this proposal and indicated on the attached maps [see Figs x, xx and xxx].

New Mineral Physics Faculty at U. S. and Non U. S. Institutions

The vitality and youth of the mineral physics community in the U. S. and overseas is dramatically represented by the large increase in the number of new tenure-track appointments made recently in institutions both here and abroad. In the half decade preceding the establishment of COMPRES, 8 such appointments were made in the U. S.. In the half decade of the first era of COMPRES, 20 appointments were made in the U. S. and another 25 overseas.

Mineral Physics faculty appointments in U. S. Universities in Pre-COMPRES Era [1997-2001]

| Name | Date of appt | Institution |
|-----------------|--------------|---|
| Ross Angel | 2000 | Virginia Polytechnic Institute and State University |
| Paul Asimow | 1999 | California Institute of Technology |
| Pamela Burnley | 1998 | Georgia State University |
| Thomas Duffy | 1997 | Princeton University |
| Marc Hirschmann | 1997 | University of Minnesota |
| Nancy Ross | 2000 | Virginia Polytechnic Institute and State University |
| Thomas Sharp | 1997 | Arizona State University |
| Lars Stixrude | 1997 | University of Michigan |

Mineral Physics faculty appointments in U. S. Universities in COMPRES Era [2002-2006]

| Name | Date of appt | Institution |
|---------------------|--------------|---------------------------------------|
| Sofia Akber-Knutsen | 2005 | University of California at San Diego |
| Andrew Campbell | 2005 | University of Maryland |
| Uli Faul | 2006 | Boston University |

| | | |
|----------------------------|------|--|
| Mark Frank | 2003 | Northern Illinois University |
| Sasawata Hier-Majumdar | 2006 | University of Maryland |
| Jennifer Jackson | 2006 | California Institute of Technology |
| Steven Jacobsen | 2006 | Northwestern University |
| Bijay Karki | 2003 | Louisiana State University |
| Abby Kavner | 2002 | University of California at Los Angeles |
| Boris Kiefer | 2003 | New Mexico State University |
| Kanani Lee | 2006 | New Mexico State University |
| Jie Li | 2003 | University of Illinois at Urbana-Champaign |
| Yanzhang Ma | 2002 | Texas Tech University |
| Wendy Panero | 2005 | Ohio State University |
| Henry Scott | 2003 | Indiana University at South Bend |
| Anurag Sharma | 2004 | Rensselaer Polytechnic Institute |
| Sang-Heon Dan Shim | 2003 | Massachusetts Institute of Technology |
| Sarah Stewart-Mukhopadhyay | 2003 | Harvard University |
| Oliver Tschauner | 2003 | University of Nevada at Las Vegas |
| James van Orman | 2002 | Case Western Reserve University |

Non-US Mineral Physics appointments in COMPRES Era[2002-2006]

| Name | Date of appt | Institution |
|-------------------------|--------------|---|
| Natalie Bolfan-Casanova | 2002 | Université Clermont-Ferrand [France] |
| Isabelle Daniel | 2004 | Université Lyon [France] |
| Agnes Dewaele | 2002 | Commissariat à l'Energie Atomique [France] |
| Daniel Errandonea | 2004 | Universitat de Valencia [Spain] |
| Haemyeong Jung | 2005 | Seoul National University [Korea] |
| Tomoaki Kubo | 2005 | Kyushu University [Japan] |
| Jennifer Kung | 2005 | National Cheng Kung University [Taiwan] |
| Sung Yeon Lee | 2003 | Seoul National University [Korea] |
| Yongjae Lee | 2006 | Yonsei University [Korea] |
| Isabelle Martinez | 2003 | Université Paris [France] |
| Jan Matas | 2003 | École Normale Supérieure Lyon France] |
| Sebastien Merkel | 2006 | Université Sciences et Technologies de Lille [France] |
| Kenji Mibe | 2003 | University of Tokyo [Japan] |
| William Minarik | 2002 | McGill University [Canada] |
| Motohiko Murakami | 2005 | Tokyo Institute of Technology [Japan] |
| Artem Oganov | 2004 | ETH-Zurich [Switzerland] |
| Chrystele Sanloup | 2002 | Université Paris VI (France) |
| Frank Schilling | 2003 | Freie Universität-Berlin [Germany] |
| Sean Shieh | 2005 | University of Western Ontario [Canada] |
| Yang Song | 2005 | University of Western Ontario [Canada] |
| Gerhard Steinle-Neumann | 2004 | Bayreuth Geoinstitut [Germany] |
| T. Tsuchiya | 2006 | Ehime University [Japan] |
| Michael Walter | 2004 | University of Bristol [United Kingdom] |

| | | |
|-------------------|------|----------------------------------|
| Wim van Westrenen | 2002 | Vrije Universiteit [Netherlands] |
| Daiksuke Yamazaki | 2005 | Ehime University [Japan] |

Awards for Mineral Physicists

In the past 4 years, many mineral physicists in the COMPRES community have been selected for special recognition for their achievements by the National Academy of Sciences, the American Geophysical Union, the Mineralogical Society of America, the American Physical Society, AIRAPT, the Royal Society of London, the Royal Swedish Academy of Sciences, the Balzan Foundation, the Geochemical Society, the European Geosciences Union, and the European Association of Geochemistry. These awards not only honor the recipients but bring visibility to the community of mineral and rock physicists throughout the world.

We highlight here four young scientists who recently received special recognition. Their awards honor, in large measure, research done at COMPRES-supported facilities as part of their Ph. D. dissertation:

2004:

Jennifer Jackson (University of Illinois at Urbana-Champaign) won the Jamieson Outstanding Student Award at the Gordon Research Conference on High Pressure.
Yongjae Lee (Stony Brook University) won the Van Valkenburg Young Investigator Award at the Gordon Research Conference on High Pressure.

2006:

Wendy Mao (University of Chicago) won the Rosalind Franklin Young Investigator Award from the Advanced Photon Source of the Argonne National Laboratory.
Li Li (Stony Brook University) won the Van Valkenburg Young Investigator Award at the Gordon Research Conference on High Pressure.

See COMPRES Newsletters and Annual Reports to the NSF for further information on other awards to members of this community.

A.5 Management and Organization of COMPRES

By-Laws

By-Laws were adopted at the First Annual Meeting of COMPRES in September 2002. At the COMPRES Annual Meeting in June 2003, a By-Laws Committee was elected. The process of reviewing and revising the By-Laws of COMPRES, initiated at the 2003 Annual Meeting, was completed in September 2004. The revised By-Laws are posted on the COMPRES website. The By-Laws of COMPRES are subject to amendment or repeal and new By-Laws made by an affirmative vote of two-thirds of the responding Electorate.

Electorate of U. S. Member Institutions

Educational or governmental institutions that are chartered in the United States with research and educational programs in high-pressure research in the science of Earth materials are eligible to apply to become members of COMPRES. As of April 2007, there are 50 U. S. members; each institution has one vote in any business decisions of COMPRES.

There are also 28 foreign affiliate members; these do not have voting rights.

Standing Committees of COMPRES

At the Annual Business Meeting each year, the Electorate votes on new officers and new members of the three Standing Committees: Executive Committee, Facilities Committee and Infrastructure Development Committee. Each Standing Committee has five elected members who serve for three-year terms [and are eligible for re-election, except the Chair of the Executive Committee].

The 15 elected members of these committees conduct the business of COMPRES and oversee the activities of the organization on behalf of the Electorate. The role and duties of each Standing Committee are described below.

Executive Committee

The Executive Committee is comprised of the Chair and four members, all elected.. The responsibilities of the Executive Committee include oversight of activities, meetings, and workshops, educational and outreach programs, and coordination with the Grand Challenge programs. The elected chairs of the Standing Committees on Facilities and Infrastructure Development serve as non-voting advisors to the Executive Committee. The appointed President attends all meetings of the Executive Committee, as a non-voting member.

Current members and affiliation (term of service)

Harry Green, Chair, University of California at Riverside (2004-2007)

Jay Bass, University of Illinois at Urbana-Champaign (2006-2009)

Michael Brown, University of Washington (2005-2008)

Donald Weidner, Stony Brook University (2004-2007)

Quentin Williams, University of California at Santa Cruz (2004-2007)

Facilities Committee

The Facilities Committee oversees the community facilities program. It evaluates the effectiveness of the service delivered by the community facilities. It coordinates between facilities (such as between beamlines) so as to maximize the community's effectiveness in using these facilities. This committee will consider the community's needs and recommend changes in the levels of support of all community facilities. It will formulate policies for evaluation of user proposals for accessing COMPRES community facilities. Elected by Electorate.

Current members and affiliation (term of service)

Mark Rivers, Chair (2005-2007), University of Chicago. Member (2005-2008).

William Durham, Lawrence Livermore National Laboratory (2005-2008)

Abby Kavner, University of California at Los Angeles (2004-2007)

Charles Leshner, University of California at Davis (2006-2009)

Wendy Panero, Ohio State University (2006-2009)

Infrastructure Development Committee

The Infrastructure Development Committee reviews infrastructure development projects that are supported by COMPRES. It has the responsibility to assure that these projects serve the needs of the community. The committee will recommend whether a project should continue or not, and what changes are needed to better meet the needs of the community. It will also evaluate proposals by the community for new development projects and make recommendations concerning funding.

Members and affiliation (term of service)

Nancy Ross, Chair (2006-2008), Virginia Polytechnic Institute and State University, Member (2006-2009).

Pamela Burnley, Georgia State University (2005-2008)

Russell Hemley, Carnegie Institution of Washington (2005-2008)

Thomas Sharp, Arizona State University (2006-2009)

Sang-Heon Dan Shim, Massachusetts Institute of Technology (2004-2007)

A statement of the Policies and Procedures for the Standing Committees and the history of membership in the Standing Committees of COMPRES are included in the Supplementary Documents for this proposal.

Advisory Committee

The Advisory Committee is appointed by the Executive Committee and meets regularly with the Executive Committee, most commonly at the beginning of the Annual Meeting of the Electorate.

Members of the Advisory Committee and their terms of service are:

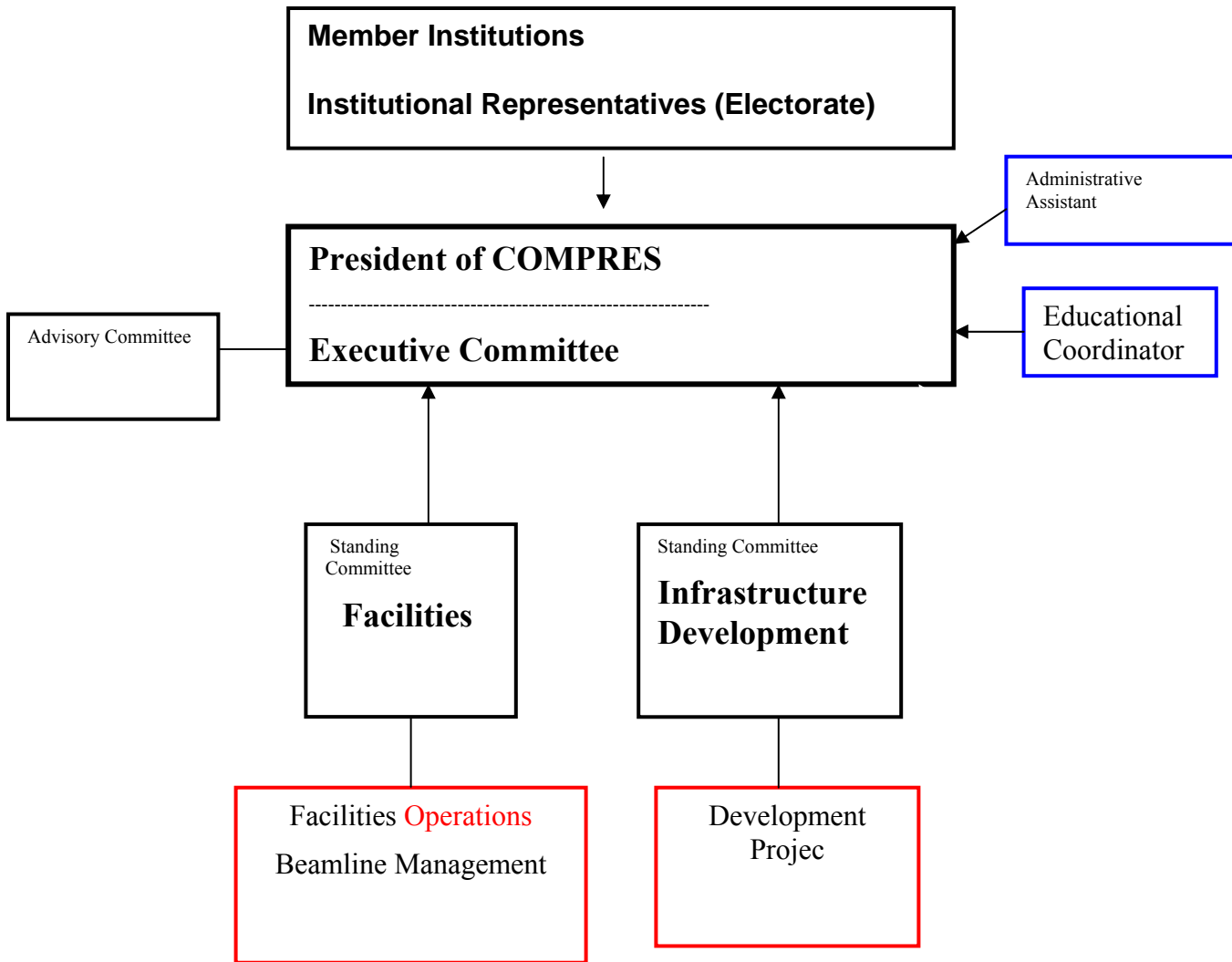
Bruce Buffett-University of Chicago [2003-2007]

Wang-ping Chen-University of Illinois at Urbana-Champaign [2006-2009]

Chi-Chang Kao-Brookhaven National Laboratory [2003-2008]

Guy Masters-University of California at San Diego [2003-2008]

Malcolm Nicol-University of Nevada at Las Vegas [2006-2009]
Richard O'Connell-Harvard University [2003-2007]
Paul Silver Carnegie-Institution of Washington [2003-2006]



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 Cooperative 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: Juhua 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 listserv and database. Samantha Lin, Administrative

Assistant: COMPRES role: Video-conferencing logistics; cooperate 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 three staff members of the Department of Geosciences at Stony Brook: Owen Evans, Director of Laboratories; Claire Ondrovic, Assistant to the Chair; and Benedict Vitale, Electronics Engineer.

Review of Management and Organization of COMPRES

The first review of the management and organization of COMPRES occurred in October 2002. During the first two years of COMPRES II [2007-2009], it will be appropriate to review the management structure again, including the location of the headquarters of the COMPRES central office. In preparation for this review, the COMPRES Executive Committee will commission a self-assessment in 2007 and obtain feedback from the Advisory Committee.

A.6 Meetings and Workshops sponsored or related to COMPRES

Fall Meetings of the American Geophysical Union

At the Fall AGU Meeting in San Francisco each December, many members of the COMPRES community have organized and convened special sessions on mineral and rock physics, often under the auspices of the Focus Group on Mineral and Rock Physics. In the Fall 2005 Meeting, for which Steve Jacobsen served as the representative for Mineral and Rock Physics on the Program Committee, there were 242 abstracts [2% of the meeting total] submitted under the MRP designation, of which 72 were first-authored by students. This created 7 different special sessions with 80 oral presentations. There were additional papers from our field submitted under the Tectonophysics and Volcanology-Geochemistry-Petrology sections.

Annual Meetings of COMPRES

Each year, COMPRES has convened an annual meeting of the entire community. The first of these meetings was held in Stony Brook, New York in September 2002 and was primarily devoted to business of established the policies and procedures of the consortium.

Since 2003, the annual meetings have been held in June at sites which have moved each year:

2003: Coast Santa Cruz Hotel, Santa Cruz, California; 31 active participants plus guests

2004: Granlibakken Resort, Lake Tahoe, California; 57 active participants plus guests

2005: Mohonk Mountain House, New Paltz, New York; 109 active participants plus guests

2006: Snowbird Alpine Village, Utah; 101 active participants plus guests

These meetings have included focus sessions on the mantle, the core and planetary evolution, all of which will include keynote talks followed by group discussion, as well as reports from the Community Facilities and Infrastructure Development projects, breakout sessions on special topics and issues, and poster presentations highlighting the most exciting recent scientific achievements. Most of the keynote talks are invited presentations by non-mineral physicists from disciplines (seismology, geodynamics, geochemistry, planetary science) that use mineral physics data to interpret their Earth observations [examples from the past two annual meetings include B. Romanowicz-UC Berkeley, A. Hoffmann-Max Planck Insitut Mainz, M. Ishii-UC San Diego, W-P. Chen-University of Illinois at Urbana-Champaign, E. Garnero-Arizona State University, A. McNamara-Arizona State University, G. Masters-UC San Diego, and D. Stevenson-Caltech]. Keynote talks have also been presented by prominent mineral physicists from overseas, including Guillaume Fiquet (France), Kei Hirose (Japan) and Isabelle Daniel (France).

For the past two years, the social events of the meeting have been underwritten by industrial sponsors. Additional details of these Annual Meetings may be found on the COMPRES website at:

<http://www.compres.stonybrook.edu/Meetings/index.html>

Workshops and Special Focus Meetings

Over the past five years, COMPRES has sponsored and provided partial financial support to many workshops and special focus meetings.

The workshops have emerged as one of the most important components of our facilities operations and infrastructure development projects. These have generally fallen into two categories with distinct purposes:

(1) To cultivate and train new users of beamlines at the national laboratories. Examples include the NRIXS, MAC and IR workshops [see list of acronyms above].

(2) To nurture new initiatives which may lead to proposals for leveraged funding. Examples include the Single Crystal workshop convened by p. Dera and c. Prewitt in 2004 and the High P Melts workshop convened by c. Agee in 2005.

In the case of special meetings of specific relevance to high-pressure mineral physics, COMPRES has provided sponsorship, help in dissemination of information, and modest amounts of funding. For special sessions or symposia within regular meetings [e.g., AGU or GSA], COMPRES has decided not to provide any financial support, and thus, for these workshops/meetings below, the level of COMPRES funding is indicated as \$0K. However, COMPRES has provided assistance in advertising these sessions and dissemination information on behalf of the conveners.

2002

International Seminar on High Pressure Mineral Physics

Verbania, Italy. August 26-31, 2002. COMPRES funding: \$20K

2003

Workshop on A New Generation of Quantitative Laser-Heating Experiments

Advanced Light Source. February 22, 2003. COMPRES funding: \$3K

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

Virginia Polytechnic Institute and State University. March 12-15, 2003.

COMPRES funding: \$15K. Co-sponsored with Joint Institute for Neutron Sciences.

Workshop on High-Pressure Earth & Planetary Sciences in the Future

Miami. March 22-23, 2003. COMPRES funding: \$30K

Workshop on Mantle Composition, Structure, and Phase Transitions

Frejus, France. April 2-6, 2003. COMPRES funding: \$12K

Special Symposium in Honor of Charles T. Prewitt

Seattle, WA. November 5-8, 2003. COMPRES funding: \$0K

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

Advanced Light Source. December 4-7, 2003. COMPRES funding: \$3K

Special Symposium in Honor of Don L. Anderson

Fall AGU. December 7-12, 2003. COMPRES funding: \$0K

2004

CSEDI Science Plan Workshop

La Jolla, CA. February 21-24, 2004. COMPRES funding: \$0K.

Future Directions for the Laser-Heated Diamond Anvil Cell at the APS

Advanced Photon Source. March 20, 2004. COMPRES funding: \$7K

COMPRES Workshop on Focused Ion Beam (FIB) applications in Earth Sciences.

Riverside, CA. March 28-29, 2004. COMPRES funding: \$13K

Rheology Grand Challenge Workshop on "Ultra-High Pressure Rheology"

Yale University. May 1-2, 2004. COMPRES funding: \$0K

Elasticity Grand Challenge Workshop

University of Illinois at Urbana-Champaign. May 8-9, 2004. COMPRES funding: \$0K

SCEC/COMPRES Workshop on the Science, Status, and Future Needs of Experimental Rock Deformation

Mount Holyoke College. August 13-14, 2004. COMPRES funding: \$5K

Structure Determination by Single Crystal X-ray Diffraction (SXD) at Megabar Pressures

Advanced Photon Source. November 12-13, 2004. COMPRES funding: \$20K

Special Symposium in Honor of Jean-Paul Poirier

Fall AGU. December 13-17, 2004. COMPRES funding: \$0K

2005

Workshop on Nuclear Resonant Scattering on Earth Materials using Synchrotron Radiation

Advanced Photon Source. February 11-13, 2005. COMPRES funding: \$8K

Workshop on Multi-Anvil Techniques

Advanced Photon Source. March 1-3, 2005. COMPRES funding: \$20K

Workshop on Calorimetry-on-a-Chip

New Paltz, NY. June 17, 2005. COMPRES funding: \$4K

Workshop on High Pressure Melts

Albuquerque, NM. July 20-22, 2005. COMPRES funding: \$20K

3rd Workshop on Earth's Mantle Composition, Structure, and Phase Transitions

Saint Malo, France. August 30-September 3, 2005. COMPRES funding: \$10K

Workshop on Neutrons at High Pressure

Los Alamos National Laboratory. September 13, 2005. COMPRES funding: \$2K

Elasticity Grand Challenge Workshop

Stony Brook University. September 16-18, 2005. COMPRES funding: \$0K

High Pressure Summit Meeting

Ronkonkoma, NY. September 24-25, 2005. COMPRES funding: \$2K

Special Topical Session on High Pressure Mineral Physics

Salt Lake City, UT. October 17-19, 2005. COMPRES funding: \$0K

Workshop on Rheology and Elasticity Studies at Ultra High Pressures and Temperatures

Advanced Photon Source. October 21-23, 2005. COMPRES funding: \$20K

Workshop on Evaluation of Synchrotron Mossbauër Data

Advanced Photon Source October 29-30, 2005. COMPRES funding: \$8K

Synchrotron Infrared Spectroscopy for High Pressure Geoscience and Planetary Science

National Synchrotron Light Source. November 3-5, 2005. COMPRES funding: \$20K

Workshop on New Directions in High-Pressure Science: Probing Extreme Conditions with Ultrashort X-ray Sources

Advanced Light Source. December 3, 2005. COMPRES funding: \$5K

2006

Workshop on Future Directions for X-ray High-Pressure at the NSLS

National Synchrotron Light Source. 25-26 February 2006. COMPRES funding: \$20K

Working Group on Gas-Loading DAC system at APS

Advanced Photon Source. 28 April 2006. COMPRES funding: \$7K

Synergy of 21st Century High-Pressure Science and Technology

Advanced Photon Source. 29 April-1 May 2006. COMPRES funding: \$20K

A.7 Education and Outreach

Current and past activities

During the past five years, COMPRES has worked with other organizations to promote inquiry-based education and outreach as nationwide collaborations between scientists, educators, materials developers, government agencies and other stakeholders. Glenn Richard and William Holt at Stony Brook, and Michael Hamburger at Indiana University are currently PIs on an NSF grant entitled “Collaborative Research: Map Tools for EarthScope Science and Education”. This project is aimed at the development of a suite of mapping tools and curriculum materials to enable the research and educational communities to work with EarthScope and other geological, geodynamic and geophysical data. In 2002 and 2003, Glenn Richard was the Chair of the Planning Committee for Skills Workshops at the DLESE Annual Meeting.

COMPRES maintains a searchable image library which is available on the web from its home page [see link at: <http://www.compres.stonybrook.edu:8080/COMPRESImageLibrary/index.html>]. This is designed to make images available to the academic community for education and research. This Library contains graphic images drawn from COMPRES meetings and workshops, with notes for referencing and appropriate attribution. We encourage members of the COMPRES and wider community to take advantage of this resource and to contribute to its growth.

During the summer of 2006, Glenn Richard was a co-mentor of an REU student with Bill Holt. The student worked on developing strain rate models for the western United States. The output of the model will be represented in the form of raster maps of the western United States and numerical geospatial data that students can use in a GIS or other mapping tool to study the model.

We plan to continue similar educational and outreach programs in the next five years, always taking advantage of leverage and linkages to other such programs in the Earth sciences.

Proposed COMPRES REU Summer Program

COMPRES is planning to submit a proposal to the NSF Research Experience for Undergraduates [REU] program in 2006 or 2007. This proposal will be for an independent COMPRES-wide REU site, which would be complimentary to the sites currently operated by the Geophysical Laboratory of the Carnegie Institution of Washington and the Mineral Physics Institute of Stony Brook University. Mentors from COMPRES member institutions who wish to participate in the program would inform the COMPRES office of their proposed project before the students are selected.

The COMPRES-wide program would serve undergraduates entering their junior or senior years by providing them opportunities to do research at COMPRES member institutions. They will be selected from colleges and universities throughout the United States based

on information, such as a transcript, essay, and letters of reference, which they will provide as part of their applications. Preference will be given to students whose home institutions do not offer the degree of research opportunity that is typically available at COMPRES member institutions who will serve as hosts. Students from underserved groups will be actively sought for participation in the program. Each summer, a total of ten student interns will be selected for this program.

The 10 week program will be modeled after the very successful REU program currently being run by the Mineral Physics Institute at Stony Brook. The first week would consist of an orientation at COMPRES headquarters, in conjunction with the MPI program. This component of the program will develop cohesiveness among the students in the group, who will continue to communicate with each other through regularly scheduled teleconferencing and other technology while the students are distributed across the United States during the bulk of the program. The orientation will offer academic and social events, including a discussion of scientific ethics, training in laboratory safety, and various recreational trips. These could include a field trip to Fire Island National Seashore, a visit to the National Synchrotron Light Source and other facilities at Brookhaven National Laboratory, and a get-to-know-one-another breakfast and evening barbecue.

For the next 8 weeks, each intern will work with a mentor at a COMPRES member institution. For the final 10th week of the program, the students will return to COMPRES headquarters to complete and deliver their final presentations for each other and Stony Brook Geosciences students and faculty. They will also be offered the opportunity to present their results at a professional meeting, with all expenses paid, preferably the Fall American Geophysical Union (AGU) meeting in San Francisco.

For the summer, each intern will receive benefits competitive with other programs, including a stipend, housing, travel, and a small research budget (~\$1000):

Student applications will be sent to COMPRES headquarters and selection will be made by a committee there in early March. From mid to late March, COMPRES will perform a preliminary match between students and mentors. During early April, COMPRES will work with the mentors at the host institutions to revise these matches, if necessary, to achieve optimal results.

Student Interns at Beamlines of National Facilities

In 2004-2005, with supplemental funding provided by the IF Program of EAR, COMPRES supported the internships of two students working at beamlines of national facilities.

Arianna Gleason

Home Institution: University of Arizona

National Facility: Advanced Light Source of the Lawrence Berkeley National Laboratory.

Supervisors: Simon Clark and Martin Kunz
Topic: Phase Transitions in Talc

Christopher Young
Home Institution: University of California at Davis
National Facility: National Synchrotron Light Source of the Brookhaven National
Laboratory
Supervisors: Jihua Chen and Michael Vaughan.
Topic: Density of Molten FeS

Both of these interns commenced their one-year appointments in late summer 2004. Over the ensuing year, both pursued independent research projects as well as offering operational support to users at the high-pressure beamlines.

This pilot experiment proved extremely rewarding for both the interns and their supervisors. Arianna Gleason is currently enrolled in graduate school in Earth Sciences at UC Berkeley, and Christopher Young is considering pursuing graduate study in Earth or materials science.

A.8 Information Technology and Communications

Communication within the mineral physics community includes an active website and other electronic services, quarterly newsletters, monthly messages from the President, and an exhibition booth at the Fall Meeting of the AGU.

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>; all of the files related to the COMPRES website are physically located on the <http://www.compres.stonybrook.edu> server in the COMPRES central offices at Stony Brook University and are maintained by Glenn Richard, Ann Lattimore, and Michael Vaughan. This 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.
- Publications of COMPRES and links to lists for associated organizations [e.g., GSECARS]:.

[Annual Reports to NSF for Years #1-4.](#)

[COMPRES Booth PowerPoint presentation at Fall AGU Meetings](#)

Minutes of the Executive Committee

Monthly Messages from COMPRES President

“Current and Future Research Directions in High-Pressure Mineral Physics-The Bass Report [August 2004]

- The quarterly COMPRES Newsletters
- Education and Outreach.
- The COMPRES Image Library, described in the Education and Outreach section of this report

[link at: <http://www.compres.stonybrook.edu:8080/COMPRESImageLibrary/index.html>]

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. These lists are used to distribute job postings, special meeting announcements, monthly messages from the President.
- **People database:** Contact information for people involved in COMPRES. In 2006, this was made available online through a browser-based form
- **Online Forms for meeting registration:** This offers online registration for meetings and workshops.
- **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 has published a quarterly newsletter with information and announcements of interest to the COMPRES community, in the broadest sense.

The 2005 issues featured reports on the Virtual Laboratory for Earth and Planetary Materials (VLab at the University of Minnesota), COMPRES-sponsored Workshops, the 2005 COMPRES Annual Meeting, Beamline interns, recent PhDs in mineral physics, membership updates, and the Brillouin spectroscopy infrastructure development project (led by the University of Illinois at Urbana-Champaign for installation at the Advanced Photon Source).

The March 2006 issue contains a feature article on the new monochromatic side-station at the multi-anvil beamline at the NSLS, plans and procedures for the renewal proposal to the NSF, report on a high-pressure X-ray workshop at the NSLS, an update from the neutron corner, and an article on recent Ph. D. graduate from the University of California at Riverside.

These newsletters are edited by Jihua Chen and may be found on the COMPRES web site at www.compres.us/Newsletter/

Monthly Messages

In addition to a column in the quarterly COMPRES newsletter, the President of COMPRES [Robert Liebermann] has sent a Monthly Message to the COMPRES community using the listserv 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 2005 AGU Meeting

At the Fall Meeting of the American Geophysical Union in San Francisco in December 2005, COMPRES had a special booth in the Exhibition Area each year since 2003. This exhibition booth is jointly sponsored by GSECARS and COMPRES, and has attracted lots of visitors. Jihua Chen and Ann Lattimore created the materials for the booth based on input provided by the Community Facilities and Infrastructure Development projects. Glenn Richard and Michael Vaughan helped in staffing the booth, in cooperation with Nancy Lazarz and Mark Rivers of GSECARS.

A PowerPoint presentation created for the 2005 COMPRES Booth by Jihua Chen can be found at

www.compres.us/Meetings/2005-12-12-AGU-Powerpoint/COMPRESbooth05.ppt

A. 9. Publications

In the first 5 years of COMPRES [2002-2007], 313 papers were published based on work at the COMPRES-supported beamlines or as part of the infrastructure development projects. These are included with the progress reports on these programs in Part B of this proposal.

A complete list of these publications is included in the Supplemental Documents for this proposal. Of these publications, 86 or 27% appeared in premier journals with high impact factors: Science, Nature, Physical Review Letters, Physical Review B, Journal of American Chemical Society, American Mineralogist, Journal of Geophysical Research, Geophysical Research Letters, Earth and Planetary Science Letters, and *Geochemica and Cosmochimica Acta*.

A. 10 Program Plan and Request for No-Cost Extension

Current Cooperative Agreement

The current Cooperative Agreement [EAR 01-35554] was originally scheduled to expire on 30 April 2007.

In February 2007, COMPRES submitted a request for a no-cost extension of this expiry date to 30 April 2008 to provide additional time for the projects undertaken via this Cooperative Agreement between the NSF and COMPRES to be completed in an efficient and wise manner.

This Cooperative Agreement is administered by the Research Foundation of the State University of New York via the Mineral Physics Institute of Stony Brook University. It includes subawards to other institutions for operating facilities at national laboratories and for infrastructure development projects at university sites.

For each of the following projects supported by COMPRES, we indicate the specific rationale for requesting this extension of time.

Community Facilities Operations:

X-ray Diamond-Anvil High-Pressure Facilities at the ALS
Operated by the University of California Berkeley.
Acquire and install minor equipment upgrades.

X-ray Diamond-Anvil High-Pressure Facilities at the NSLS
Operated by the University of Chicago.
Install equipment upgrades and complete software development.

Infrared Diamond-Anvil High-Pressure Facilities at the NSLS
Operated by the Carnegie Institution of Washington
Complete software development.

X-ray Multi-Anvil High-Pressure Facilities at the NSLS
Operated by Stony Brook University.
Complete minor equipment upgrades and development of software.

Neutron Studies at National Facilities
Operated by Virginia Polytechnic Institute and State University
Complete development of software for reduction of neutron scattering data.

Infrastructure Development Projects:

Multi-Anvil Cell Development
Arizona State University

Complete production of cell parts for users and deliver to other institutions.

Brillouin Spectroscopy at Advanced Photon Source

University of Illinois at Urbana-Champaign

Acquire minor equipment for automation and complete software development.

Nuclear Resonant Inelastic X-ray Scattering at High P & T

University of Illinois at Urbana-Champaign

Complete software development for user-friendly interface.

Development of CO₂ Laser-Heated Diamond-Anvil Cell

Princeton University and University of Chicago

Take delivery of equipment and install and commission on beamline at the Advanced Photon Source.

Absolute Temperature Calibration using Johnson Noise Thermometry

University of Colorado and University of Chicago

Complete testing of sensors in multi-anvil apparatus and software development.

Development of CEAD

Operated by Virginia Polytechnic Institute and State University

Complete development of software for reduction of X-ray diffraction data.

Dual beam Focused Ion Milling Facility for TEM

Operated by the University of California Riverside

Acquire new TEM equipment and commence training of users.

Gas-loading System for Diamond-Anvil Cells

Operated by University of Chicago.

Take delivery of equipment and assemble and install at the GSECARS facilities at the Advanced Photon Source.

Development of Next Generation Multi-anvil Module for Megabar Research

Operated by University of Chicago.

Take delivery of equipment and assemble and install at the GSECARS facilities at the Advanced Photon Source.

Calorimetry-on-a-Chip

Operated by University of California Davis

Complete pilot studies and analyze data.

Monochromatic X-ray Side Station at the X17B2 Beamline of the NSLS

Operated by Stony Brook University.

Take delivery of new hydraulic press and install on the side station at the beamline.

The request for a no-cost extension was approved on 7 March 2007.

Leveraging of NSF-EAR funding to COMPRES in 2002-2007

While COMPRES derives its primary financial support from the Instrumentation and Facilities Program in the Division of Earth Sciences of the NSF, it leverages the enormous investment of the DOE in constructing and supporting the operation of its national laboratories, notably those at Brookhaven, Argonne, Lawrence Berkeley, and Oak Ridge National Laboratories. In addition, members of the COMPRES community have been very successful in obtaining other funding from the NSF, DOE and DOD to enhance the opportunities for research in high-pressure mineral physics. Examples in the past 4 years include:

SNAP: Spallation Neutrons at Pressure: DOE grant to J. Parise, H-k Mao, R. Hemley and C. Tulk for construction of high-pressure beamline at the SNS of ORNL.

DOD-DURIP grant to J. Chen and colleagues for monochromatic X-ray side station at X17B2 beamline of the NSLS.

NSF-Major Research Instrumentation [MRI] program grant to P. Eng and colleagues for construction of a side-station on the bending magnet beamline operated by GSECARS at the APS.

NSF-ITR program grant to R. Wentzcovitch and colleagues for establishment of a Virtual Laboratory for Earth and Planetary Materials [VLab]

NSF-MRI grant to M. Nicol, P. Dera and colleagues for development of new approaches for micro-focus single-crystal X-ray diffraction for materials structure research at synchrotrons.

Grand Challenges

Under separate funding from the NSF Division of Earth Sciences, scientists in the COMPRES community were awarded grants for three Grand Challenge collaborative research programs:

Experimental Study of Plastic Deformation under Deep Earth Conditions
Team coordinated by S. Karato of Yale University, and including partners from University of California at Riverside, Stony Brook University, Georgia State University, and the University of Chicago.
Rheology of Earth materials at high pressures and temperatures.

Elasticity Grand Challenge of COMPRES Initiative
Team coordinated by J. Bass of the University of Illinois at Urbana-Champaign, and including partners from Delaware State University, University of Minnesota, University of Michigan, Carnegie Institution of Washington and Stony Brook University.
Elasticity of Earth materials at high pressures and temperatures.

Development of Next Generation Megabar High-Pressure Cells

Team led by R. Hemley at the Carnegie Institution of Washington.
Growth of large synthetic diamonds by chemical vapor deposition

While these Grand Challenge programs are formally independent from the COMPRES core grant, they are intimately related intellectually as they give prime examples of the scientific problems that are being addressed using the facilities operated and the technological developments funded by COMPRES.

Leveraging of NSF-EAR funding to COMPRES in 2007-2012

As in the era of COMPRES I [2002-2007], we anticipate that members of the COMPRES community will be aggressive and successful in formulating proposals and obtaining grants from other funding sources within the NSF, DOE, DOD and NASA agencies.

For the “Grand Challenge” style of collaborative research projects, we are aware that three such proposals were submitted to NSF in 2006:

(1) High Pressure Melts

Team coordinated by C. Agee of the University of New Mexico

(2) Rheological Properties of Earth Materials

Team coordinated by S. Karato of Yale University

(3) Elastic Properties of Earth Materials

Team coordinated by J. Bass of the University of Illinois at Urbana-Champaign

B.1 Community Facilities Operations

B.1.a X-ray Diamond-anvil High-Pressure Facilities at the Advanced Light Source

Operated by University of California Berkeley [R. Jeanloz and S. Clark]

Curent funding for 2002-2007: \$1100K [including \$115K for equipment upgrades]

1. Current status of high-pressure facilities and operations

High-pressure research in the Earth Sciences has a long tradition on the West Coast of the United States. This community made large contributions to the development of some of the key technologies, such as laser-heated diamond-anvil cell and shockwave methods, used in our field. The West Coast community initially focused its local synchrotron use at the Stanford Linear Accelerator Center. Development of high-pressure facilities at the Advanced Light Source (ALS), together with loss of high-pressure facilities at SSRL due to the SPEARIII upgrade, led to a switch of activity from Stanford to the ALS.

Prior to 2002 there were few high-pressure experiments conducted at the ALS. This was due to the lack of any high-pressure infrastructure. Those measurements that were made were really demonstration experiments set up in an empty hutch. Support from COMPRES has contributed toward the necessary infrastructure and provided the impetus to enable the community led development of high-pressure facilities at the ALS designed specifically to meet the needs of the COMPRES community. During the four year period since COMPRES funding started in May 2002 we have established a high-pressure laboratory containing all of the necessary equipment to allow high-pressure diamond anvil cell research at the ALS, converted two beamlines (11.3 and 1.4) that already existed to allow high pressure research and built a completely new dedicated high-pressure beamline (12.2.2) which includes an integrated laser heating system (See Appendix I). Since the beginning of this year these facilities have been fully operational serving the needs of the COMPRES community.

Facilities for high-pressure research on the West Coast would not be in the advanced state that they are today if it had not been for the support of COMPRES.

In the early stages of these developments COMPRES funds allocated to the west coast community were used primarily for capital equipment purchases while now virtually all of the funds are used to support two beamline scientists. In addition to the COMPRES contribution capital equipment funds have also been provided by the University of California (~\$1.4M), the Lawrence Livermore National Laboratory (~\$0.3M) and the

Lawrence Berkeley National Laboratory (~\$1.2M). General running costs for the high-pressure laboratory and beamlines are provided by the ALS (~\$80k/a). The ALS also provides beamline scientists on all three beamlines as well as engineers and technicians who are available to assist COMPRES users in designing and constructing novel pieces of scientific equipment as well as devising and executing their experiments. Also Prof. Jeanloz has worked as the PI for the west coast component of COMPRES with no salary for the whole of this period at about the 5% level and Simon Clark has provided project management services at no cost to COMPRES at about the 20% level. Recently Raymond Jeanloz has transferred extramural funds to construct Brillouin and Raman systems (\$0.25M) and has transferred approximately \$0.25M worth of laboratory equipment. Lawrence Livermore National Laboratory is donating resistively heated and cryogenically cooled diamond anvil cells (\$0.1M).

Take up of beamtime at the ALS is principally through two mechanisms: Approved Program and General User. COMPRES users have been particularly successful with both routes. During the current allocation period (January – July 2006) 59 days of beam time on beamline 12.2.2 and 25 days of beamtime on beamline 11.3 have been allocated to COMPRES users. That is over 80% of the available beamtime on beamline 12.2.2.

COMPRES funds are clearly achieving substantial leverage at the ALS in terms of both beamtime and infrastructure.

Given that there are only 108 work days during the January-July period the current beam time usage results in our two beam line scientists spending over 80% of their working days actually supporting users on the beamlines. This is addition to other necessary background work such as setting up experiments for specific user needs, routine maintenance and quality assurance, development of small end station upgrades, working with COMPRES users to prepare for experiments and helping with data analysis, preparing documentation for research publications as well as facilities reporting, preparing reports for COMPRES, participation in COMPRES meetings and at other conferences. This high work load is having an impact on their personal research productivity and their ability to develop new experimental facilities and methodologies. This is a matter of concern because the world-class quality of support provided by the beamline scientists can only be sustained if these issues are resolved.

Additional staff resource is essential if we are to retain our current beamline scientists, continue providing a high level of user support and fully develop our existing hardware.

In order to allow the highest possible ease of user access and ensure the greatest user diversity we provide the entire infrastructure necessary to allow laser heated diamond anvil cell experiments. This includes: gasket preparation and sample loading equipment, diamond anvil cells, data acquisition systems and data analysis software.

Although our facilities have only been fully operational since January 2006 we have made every effort to incorporate user experiments into our building and commissioning phases. Detailed beamline schedules are contained in Appendix 2. This work has

already resulted in a number of publications (See Appendix 3). Demand for beamline 12.2.2 is already high with an over subscription rate of more than 3:1 for dedicated COMPRES beamtime.

Scientific highlights include work demonstrating the importance of the high-low spin transition in magnesiowustite to the properties of the lower mantle (S. Speziale, A.A. Milner, V.E. Lee, S.M. Clark, M.P. Pasternak, and R. Jeanloz, **Spin Transition in Earth's Mantle**, *Proceedings of the National Academy of Sciences*, **102(50)**, 17918-17922 (2005)), investigation of the role of hydrogen bonding in the compression of talc (S.A. Parry, A.R. Pawley and S.M. Clark, **An infrared spectroscopic study of 10-Å phase to 10 GPa, and comparison to talc** *Am. Min.* In Press (2006)) and detection of subtle electronically-driven phase transitions in CdHg alloy (S. Speziale, S.M. Clark, R. Jeanloz, S. Meenakshi, V. Vijayakumar, A.K. Verma, R.S. Rao and B.K. Godwal, **High pressure investigation of Cd₈₀Hg₂₀ alloy** Submitted to *Phys. Rev. Lett.* (2006)).

We have also made significant contributions to the COMPRES community through general student training, mentoring an intern and organizing and hosting workshops and meetings (see list below).

Our aim as an institution over the next five years is to establish a firm link between seismology and mineral physics and help the community significantly advance its understanding of the lower mantle. This requires our users to be able to measure the full range of elastic properties of mineral assemblages. Currently we are adequately equipped for phase-equilibrium studies, unit-cell volume measurements and simple structure determination and refinement from powder data under conditions relevant to the lower mantle. We also are equipped for radial diffraction, viscosity measurements using falling spheres and density measurement by direct imaging. The recent allocation of NSF funds to provide high-pressure single crystal diffraction and Brillouin spectroscopy will complete the set of necessary techniques. Details of the single-crystal proposal are given below. Our aim is to make all of these facilities available to COMPRES users but given the existing over-commitment of our beamline scientists it is likely that we will have to put these developments on hold until additional resources are available.

We will not be able to make the single crystal or spectroscopy systems available to the general user community given our current level of staffing.

High-pressure publications by COMPRES users of the ALS.

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

A. Salleo, S.T. Taylor, M.C. Martin, W.R. Panero, R. Jeanloz, T. Sands and F.Y. Genin, **Laser-driven formation of high-pressure phase in amorphous silica**, *Nature Materials*, **2(12)** 796-800 (2003).

Liu, H., Caldwell, W. A., Benedetti, L. R., Panero, W., and Jeanloz, R., **Static compression of α -Fe₂O₃: Linear incompressibility of lattice parameters and high pressure transformations**, *Phys. Chem. Min.*, **30**, 582-588 (2003).

K.K.M. Lee and R. Jeanloz, **High-pressure alloying of potassium and iron: Radioactivity in the Earth's core?**, *Geophys. Res. Lett.*, **30**, SDE 6-1 – 6-4 [doi: 10.1029/2003GL018515] (2003).

K.K.M. Lee, B. O'Neill, and R. Jeanloz, **Limits to resolution in composition and density in ultrahigh-pressure experiments on natural mantle-rock samples**, *Phys. Earth Planet. Int.*, **143-144**, 241-53 (2004).

K.K.M. Lee, G. Steinle-Neumann, and R. Jeanloz, **Ab-initio high-pressure alloying of iron and potassium: Implications for the Earth's core?**, *Geophys. Res. Lett.*, **31**, L11603, 1-4 (2004).

S-H. Shim, T.S. Duffy, R. Jeanloz, and G. Shen, **Stability and structure of MgSiO₃ perovskite to the core-mantle boundary**, *Geophys. Res. Lett.*, **31**, L10603– 1-5 (2004).

K.K.M. Lee, B. O'Neill, W.R. Panero, S-H. Shim, L.R. Benedetti, and R. Jeanloz, **Equations of state of the high-pressure phases of a natural peridotite and implications for the Earth's lower mantle**, *Earth Planet. Sci. Lett.*, **223**, 381-393 (2004).

S-H. Shim, T.S. Duffy, R. Jeanloz, C-S. Yoo and V. Iota, **Raman spectroscopy and x-ray diffraction of phase transitions in Cr₂O₃ to 61 GPa**, *Phys. Rev. B*, **69**, 144107– 1-12 (2004).

D. Zaziski, S. Prilliman, E.C. Scher, M. Casula, J. Wicjham, S.M. Clark and A.P. Alivisatos, **Critical size for fracture during solid-solid phase transformations**, *NANO Letters* **4(5)** 943-946 (2004).

D. Walker, P.K. Verma, L.M.D. Cranswick, S.M. Clark, R.L. Jones and S. Buhre, **Halite-Sylvite Thermoelasticity**, *Am. Min.* **89** 204-210 (2004).

S.M. Clark and R. Jeanloz, **A new paradigm to extend diffraction measurements beyond the megabar regime** *J. Synch. Rad.* **12(5)** 632-636 (2005).

D. Walker, P.K. Verma, L.M.D. Cranswick, S.M. Clark, R.L. Jones and S. Buhre, **Halite-Sylvite Thermoconsolution**, *Am. Min.* **90** 229-239 (2005).

R.W. Cumberland, M. Weinberger, J.J. Gilman, S.M. Clark, S.H. Tolbert and R.B. Kaner, **Osmium Diboride, An Ultra-incompressible, Hard Material**, *J. Am. Chem. Soc.* **127(20)** 7264-7265 (2005).

M. Kunz, A.A. MacDowell, W.A. Caldwell, D. Cambie, R.S. Celestre, E.E. Domning, R.M. Duarte, A.E. Gleason, J.M. Glossinger, N. Kelez, D.W. Plate, T. Yu, J.M. Zaug, H.A. Padmore, R. Jeanloz, A.P. Alivisatos, and S.M. Clark. **A beamline for high pressure studies at the Advanced Light Source with a superconducting bending magnet as the source**, *J. Synch. Rad.* **12(5)** 650-658 (2005).

S.M. Clark, S.G. Prilliman, C.K. Erdonmez and A.P. Alivisatos, **Size dependence of the pressure-induced γ to α structural transition in iron oxide nanocrystals**, *NanoTechnology*, **16** 2813-2818 (2005).

S. Speziale, A.A. Milner, V.E. Lee, S.M. Clark, M.P. Pasternak, and R. Jeanloz, **Spin Transition in Earth's Mantle**, *Proceedings of the National Academy of Sciences*, **102(50)**, 17918-17922 (2005).

D Walker, **Core-mantle chemical issues** *Canadian Mineralogist* **43**, 1553-1564 (2005).

C.E. Tommaseo, S. Merkel, S. Speziale, J. Devine and H-R. Wenk, **Texture development and deformation mechanisms in magnesiowustite at high pressure**, *Physics and chemistry of minerals*, **33**, 84-97 (2006).

S. Speziale, I. Lonardelli, L. Miyagi, J. Pehl, C. Tommaseo, and H-R. Wenk. **Deformation experiments in the diamond-anvil cell: Texture in Copper to 30 GPa**, *J. Phys. Cond. Matt.* In Press (2006).

S.A. Parry, A.R.Pawley and S.M.Clark, **An infrared spectroscopic study of 10-Å phase to 10 GPa, and comparison to talc** *Am. Min.* In Press (2006).

S. Speziale, R. Jeanloz, A.A. Milner, M.P. Pasternak and J.M. Zaug, **Vibrational spectroscopy of Fe(OH)₂ at high pressure: Behavior of the O–H bond**, *Phys. Rev. B*, Submitted (2006).

H-R Wenk, I. Lonardelli, S. Merkel, L. Miyagi, J. Pehl, S. Speziale and C.E. Tommaseo, **Diamond anvil deformation experiments in radial diffraction geometry**, Submitted to *J. Phys. Cond. Matter*, (2006).

S. Speziale, V.E. Lee, S.M. Clark, M.P. Pasternak and R. Jeanloz, **Mechanical effects of Fe spin transition in (MgFe)O and implication for the seismological properties of the Earth's lower mantle** Submitted to *Journal of Geophysical Research* (2006).

M. Walter, R. Tronnes, L. Armstrong, O. Lord, W.A. Caldwell and Simon Clark, **Subsolidus phase relations and perovskite compressibility in the system MgO-AlO_{1.5}-SiO₂ with implications for Earth's lower mantle** Submitted to *Earth and Planetary Science Letters* (2006).

B. Gilbert, H. Zhang, B. Chen, M. Kunz, F. Huang, and J. F. Banfield, **The Compressibility of Zinc Sulfide Nanoparticles**, Submitted to *Phys. Rev. B*. (2006).

S. Speziale, S.M. Clark, R. Jeanloz, S. Meenakshi, V. Vijayakumar, A.K. Verma, R.S. Rao and B.K. Godwal, **High pressure investigation of Cd₈₀Hg₂₀ alloy** Submitted to *Phys. Rev. Lett.* (2006).

A.E.Gleason, S. Parry, M. Kunz, W.A. Caldwell, A.R. Pawley and S.M. Clark, **Pressure Temperature Stability Studies of Talc and 10-Å Phase using x-ray diffraction**, *Am. Min.* In Prep. (2006).

K.K.M. Lee, O. Tschauner and P. D. Asimow, **Phase Assemblage and stability of pyroxenite at Lower-Mantle conditions**, In Prep. (2006).

List of relevant workshops and meetings organized and hosted at the ALS.

1. **COMPRES laser heated diamond anvil cell workshop.** Organisers: Simon Clark and Michael Walter. 22 February 2003.
2. **IUCr/COMPRES sponsored high-pressure practicum.** Organiser: Simon Clark. 4 December 2003.
3. **IUCr/COMPRES sponsored workshop: High Pressure Structure and Reactivity: The Science of Change.** Organisers Simon Clark, Martin Kunz and Sarah Tolbert 5-7 December 2003.
4. **California High-Pressure Science Seminar Series.** Organisers: Simon Clark and Raymond Jeanloz. 27-28 March, 2004.
5. **LCLS shockwave meeting.** Organiser: Simon Clark. 28 January 2005.

6. **Using ultra short pulses to probe the shocked state**, Organisers: Simon Clark, Yogi Gupta, Jerry Hastings, Rus Hemley and Raymond Jeanloz. 3 December 2005.

B.1.b X-ray Diamond-Anvil High-Pressure Facilities at the National Synchrotron Light Source

Operated by:

Carnegie Institution of Washington [H-k.Mao, 2002-2005]

University of Chicago [M. L. Rivers, 2005-2007]

New Operation Team starting May 2007:

Stony Brook University [J. Chen], Princeton University [T. S.

Duffy], University of Chicago [M. L. Rivers], and Carnegie

Institution of Washington [A. Goncharov]

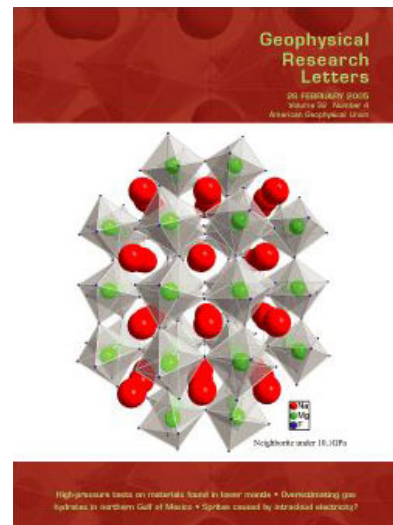
Current funding for 2002-2007: \$1074K [including \$87K for equipment upgrades]

I. Summary of the Progress Achieved in the Four Year Period of COMPRES from May 1, 2002 to April 30, 2006

A. Selected Scientific Highlights 2002-2006 (88 Peer-Reviewed Publications)

Perovskite structure evolution and post-perovskite phase transition in NaMgF₃:

Geophysical interests in the perovskite family date from the 1960s when geologist A.E. Ringwood proposed that the Earth's lower mantle is dominated by iron-bearing MgSiO₃ perovskite. Knowledge about perovskite structure at high-pressure/high-temperature conditions is valuable for understanding the lower mantle. A series of experiments were carried out using various pressure-transmitting medium in a DAC to study the perovskite structure evolution and the post-perovskite phase transition in NaMgF₃ under high pressures. The centrosymmetrically distorted orthorhombic perovskite with space group Pbnm is described by two independent octahedral tilting angles Θ and Φ , where Θ is an anti-phase tilt and Φ is an in-phase tilt. The octahedral tilting of the NaMgF₃ perovskite was quantitatively derived from the cell parameters (macro-approach) as well as from the positional parameters of atoms (micro-approach). The overall trends of the octahedral tilting angles, i.e. increasing with increasing pressure, are similar for both the macro and micro approaches. The volumetric compression was dominated by the shortening of the octahedral Mg-F bond at the beginning of compression below 6 GPa. In the 6-12 GPa pressure range, the contribution from the octahedral tilting matches that of the bond length compression. This is followed by an increasing contribution from the octahedral



tilting above 12 GPa. The octahedral tilting, increasing with pressure, finally destroys the perovskite structure. A phase transition to post-perovskite structure (Cmcm) was observed at about 19.4 GPa (Liu *et al. Geophys. Res. Lett.* 2005).

Discovery of Two New Post-Spinel Minerals in a Shock-Metamorphosed Chromite Grain in Suizhou Meteorite: Using the x-ray diffraction (XRD) microprobe technique at X17C, Chen *et al.* discovered a CF-type [Chen *et al. Geochim. Cosmochim. Acta* 2003] and a CT-type polymorph [Chen *et al. Proc. Natl. Acad. Sci.* 2003] 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: A. Kavner [Earth Planet. Sci. Lett. 2003] performed radial X-ray diffraction experiments with OH-bearing (hydrous) ringwoodite compressed uniaxially in a diamond anvil cell. The material 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.* 2001]. The result suggests 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.

Space weathering on airless planetary bodies: Clues from the lunar mineral hapkeite: The crystal structure of mineral hapkeite has been studied by in situ synchrotron energy-dispersive, single-crystal x-ray diffraction technique. [Anand *et al.*, PNAS, 2004]. It is confirmed that the crystal structure of hapkeite is cubic with a space group Pm3m (221) and lattice parameter of 2.831 (4) Å, similar to the structure of synthetic Fe₂Si. This mineral and other Fe-Si phases are probably more common in the lunar regolith than previously thought and are directly related to the formation of vapor-deposited, nanophase elemental iron in the lunar soils. The formation of this nanophase elemental Fe⁰ (np-Fe⁰) is related to space weathering. Physical and chemical reactions occurring as a result of the high velocity impacts of meteorites and micrometeorites and of cosmic rays and solar-wind particles are major causes of space weathering on airless planetary bodies, such as the Moon, Mercury, and asteroids. These weathering processes are responsible for the formation of their regolith and soil.

Strength and Elasticity of SiO₂ across the Stishovite - CaCl₂-type Structural Phase Boundary: Stishovite is known to transform to orthorhombic CaCl₂-type structure at 50±3 GPa. The study of elastic instabilities is important for understanding phase

transformations, and the stishovite–CaCl₂-type transition, which is driven by an instability of an elastic shear modulus, has attracted much attention. Shieh et al. [*Phys. Rev. Lett.*, 2002] used lattice strain measurements under nonhydrostatic compression in a diamond anvil cell to examine dense SiO₂ pressure up to 60 GPa. The ratio of differential stress to shear modulus t/G is 0.019(3) to 0.037(5) at pressure from 15 to 60 GPa. The ratio for octahedrally coordinated stishovite is lower by a factor of about 2 than observed in four-coordinated silicates. Using a theoretical model for the shear modulus, the differential stress of stishovite is found to be 4.5(1.5) GPa below 40 GPa and decrease sharply as the stishovite to CaCl₂-type phase transition boundary is approached. The differential stress then recovers rapidly to values of 5±2 GPa at 52-55 GPa in the CaCl₂-type phase. The inversion of measured lattice strains provides direct experimental evidence for softening of C_{11} - C_{12} .

High-Pressure Phase Transition and Hydrogen Disordering in Gibbsite: Gibbsite Al(OH)₃ is studied using XRD at room temperature up to 53 GPa by H. Liu *et al.* [*Phys. Chem. Minerals*, 2004]. 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 were $a = 8.690 \text{ \AA}$, $b = 5.044 \text{ \AA}$, $c = 9.500 \text{ \AA}$, and its unit cell volume was 416.4 \AA^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 $75 \pm 2 \text{ GPa}$ 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.

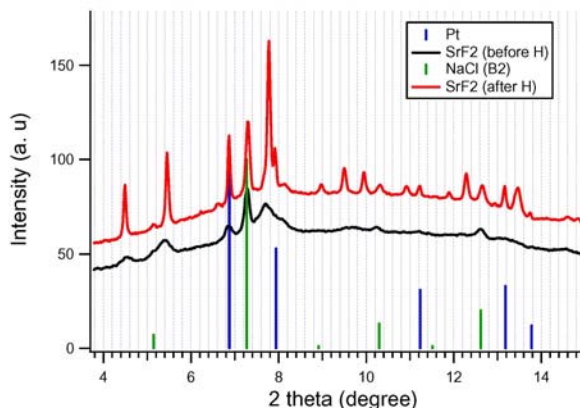
Constraining the equation of state of fluid H₂O to 80 GPa using the melting curve, bulk modulus, and thermal expansivity of Ice VII: The equation of state properties of Ice VII and supercritical H₂O at temperatures of 300 - 902 K and pressures of 6 - 60.5 GPa have been studied using a diamond anvil cell with an external resistance heater [M. Frank et al, *Geochimica et Cosmochimica Acta*, 2004]. X-ray diffraction data of ice VII fitted to the third-order Birch-Murnaghan equation of state yield the isothermal bulk modulus $K_{T0} = 21.1 \pm 1.3 \text{ GPa}$, its pressure derivative $K'_{T0} = 4.4 \pm 0.1$ and the volume $V_0 = 12.4 \pm 0.1 \text{ cm}^3/\text{mol}$ at zero pressure, respectively. Additionally, the melting curve of Ice VII was determined to greater than 40 GPa by using the disappearance of the diffraction pattern of Ice VII to monitor melting in the system. These results were used further to constrain the PVT properties of fluid H₂O at elevated pressures and temperatures by taking the pressure derivative of the Gibbs free energy difference between Ice VII and

fluid H₂O along the Ice VII melting curve. Comparison of these results suggests that the previously stated equations of state of fluid H₂O overestimate the molar volume of fluid H₂O at pressures greater than 20 GPa.

Phase Transition in SrF₂ by laser heating at X-17B3: There has been considerable interest in understanding phase transition sequences in divalent metal fluorides AF₂ (A = Pb, Ca, Sr, Ba, etc.) as a function of pressure. These materials have applications as scintillators, luminescent materials, and ionic conductors. A cubic (fluorite-type) to orthorhombic (cotunnite-type) phase transformation at high pressures has been observed in these materials. Further phase transitions and metallization are expected at higher pressures. This study [Jiang et al., in prep.] examined SrF₂ using the laser-heated diamond anvil cell at beamline X17B3

at the NSLS. Pure SrF₂ was mixed with platinum and insulated from the diamonds using NaCl layers. The sample was directly compressed to 60 GPa and then heated. Before heating, broad diffraction lines are observed which are distinct from the low-pressure fluorite-type phase indicating that a room-temperature transition had occurred. After 3 minutes of heating many new diffraction lines appear, and the pattern appears related to that prior to heating. The new diffraction peaks can be fit to a hexagonal unit cell. The observed peaks are similar to those of the post-cotunnite phase observed in BaF₂ at lower pressures (12 GPa)

(Leger et al., 1995). Upon further compression and heating to 92 GPa, the diffraction pattern is largely unchanged. Future work will focus on profile refinements of the powder patterns and further experiments to constrain the phase boundary.



Diffraction patterns obtained at X17B3 from SrF₂ at 60 GPa prior to heating (black) and after heating (red). The blue and green vertical bars show expected diffraction peak positions for Pt and CsCl-type phase of NaCl.

Lack of the critical pressure for weakening of size-induced stiffness in 3C-SiC nanocrystals under hydrostatic compression: **The compressibility of 30 nm 3C-SiC nanocrystals was studied under hydrostatic conditions while helium was used as pressure transmitting medium, as well as under nonhydrostatic conditions without pressure medium. [H.Liu et al Applied Phys. Letters, 2004] No threshold pressure phenomenon was observed for the compressibility of the nano-crystals during compression in hydrostatic conditions, while the critical pressure around 10.5 GPa was observed during nonhydrostatic compression. These indicate that the threshold pressure phenomena, recently reported that the nanocrystals initially exhibited much higher bulk modulus below the threshold pressure during compression [Appl. Phys. Lett. 83, 3174 (2003); J. Phys. Chem. 107, 14151 (2003)], were mainly caused by the non-hydrostatic effect instead of a specific feature of nanocrystals upon**

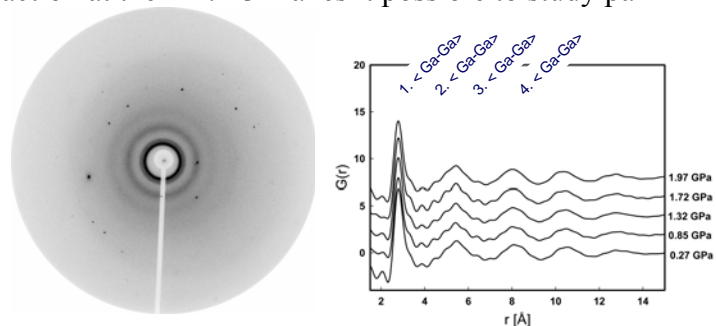
compression. The bulk modulus of 3C–SiC nanocrystals is estimated as 220.6±0.6 GPa based on the hydrostatic compression data.

Compressibility of Osmium and Other Dense Transition Metals: Compressibility (reciprocal of the bulk modulus) is an important physical property of a material. Strongly bonded materials have short interatomic distances and correspondingly strong repulsive interatomic forces, leading to high bulk moduli. The bulk modulus has been correlated empirically with the interstitial electron density, cohesive energy, and mechanical hardness. Cynn et al. [*Phys. Rev. Lett.*, 2002] studied compressibilities of 5d transition metals Ru, Ir, and Os to 60 GPa by energy dispersive and angle dispersive x-ray diffraction. Using third order Birch-Murnaghan equation to fit experimental data yields the bulk modulus of Os, Ir and Ru as 462±12 GPa, 383±14 GPa and 348±18 GPa, respectively. They note that the bulk modulus of Os is higher than the diamond bulk modulus of 443 GPa which is the highest known. Their experimental results are also compared with the results obtained by first principles electronic structure calculations of equation of state for C, Os, Ir, Re, Ru and W. The transition metals compressibility decreases in the order W-Ru-Re-Ir-Os. This result provides impetus for a continued search for superhard materials, including transition metal carbides, nitrides, and oxides.

Bioceramic hydroxyapatite at high pressure: Hydroxyapatite (HA), $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, is an important bioceramic and it is the main mineral constituent of the bone tissue in humans. With advances of deposition techniques, various nanostructured ceramics including HA have become increasingly available for biomedical implant applications. The mechanical properties of HA coatings like hardness and elastic modulus are sensitive to the preferred orientation presented in the samples. A bioceramic hydroxyapatite, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ polycrystalline sample was studied under high pressure in a diamond anvil cell to investigate its structural, electrical, and mechanical properties under compression. [Velisavljevic et al. *Appl. Phys. Lett.* 2003]. No phase transformation was observed in the pressure range of 0.1 MPa up to 32 GP. But its c/a ratio with pressure showed an anisotropic compression effect. Initially the c/a ratio is increasing up to 8 GPa suggesting that the c axis is less compressible than the a axis. Above 8 GPa, c/a reaches a steady value of 0.741, an isotropic compression persists up to the maximum pressure. These results imply that the least compressible c axis might align itself with the stress axis minimizing the elastic strain energy if a uniaxial stress applied. The present studies demonstrate that a fully dense and translucent hydroxyapatite sample is attained above 10 GPa at 300 K.

Structure and compression mechanism of high pressure liquid: Recent development of high energy monochromatic diffraction at the X17B3 makes it possible to study pair distribution function (PDF) of liquid at high pressures.

Experiments have been carried out on liquid Ga using 80 keV x-ray (Chen et al.). PDF of liquid Ga at different pressure are derived from the quality data. Results



X-ray diffraction pattern of liquid Ga and derived PDF as a function of pressure.

indicate that the 1st and 2nd nearest neighbor bond lengths are insensitive to pressure whereas the 3rd and 4th nearest neighbor bond lengths are more sensitive to pressure. A clustered local structure in the liquid is inferred for the compression mechanism. These experiments require high x-ray energy to cover large range of Q, and the X17B3 is currently the only dedicated high pressure beamline where such experiments are conducted in the nation.

B. Beamline Capabilities and Technical Developments

The diamond anvil cell X-ray (DAC-XR) facilities at the National Synchrotron Light Source (NSLS) are located on a superconducting wiggler beamline and consist of two stations (X-17C and X-17B3) together with a sample preparation laboratory. The DAC-XR facility is one of the longest-running high-pressure beamlines in the world, and has been a workhorse for diamond anvil cell research for more than two decades. It remains highly productive and, in fact, has seen a major expansion of its capabilities during the first generation of COMPRES. Over the last four years (2002-2005), an average of about 20 journal publications per year have produced based on work carried out wholly or in part at DAC-XR. This includes many papers in high-impact journals such as *Science*, *Nature*, *PNAS*, *APL*, *PRL*, as well as top geoscience journals including *GRL*, *JGR*, and *GCA*.

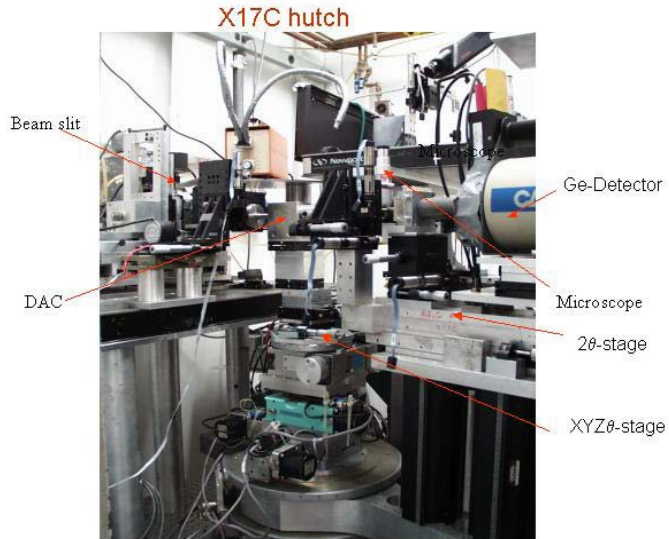
A major feature of DAC-XR is the high brightness associated with the wiggler source of this second generation synchrotron – a feature that is extremely important for diamond cell experiments in which sample volume (and hence diffraction intensity) is normally the key experimental limitation. The wiggler has a high superconductor magnetic field (5 Tesla) that produces a spectral profile similar to bending magnet (BM) beamlines at the Advanced Photon Source (APS), but has the additional advantage of multiple (5) poles and higher current that in principle yield about 5-10 times higher brightness and brilliance than the APS BM. Actual values achieved in practice depend on various beamline factors (e.g. distance from source, monochromators, focusing optics). With further improvements to beamline monochromators and focusing optics at DAC-XR, significant additional gains in x-ray brightness and brilliance can be realized over current capabilities during the next stage of COMPRES.

The current research focus at DAC-XR is mainly single-crystal and polycrystalline x-ray diffraction at ultrahigh pressures. The beamline is used for studies of phase transitions, melting, equations of state, structure refinements, yield strength, and elasticity on a range of materials including metals, oxides, silicates, nitrides, manganites and clathrates. Single-crystal studies have included examination of micro-inclusions, as well as single-crystal samples contained within the diamond anvil cell. In recent years, new techniques pioneered on the beamline include development of rotational diamond anvil cell, applications of synthetic and designer anvils, gem anvil cells, and radial x-ray diffraction techniques.

X-17C:

The X-17C beamline is a side station that runs 100% of the time. Dr. Jingzhu Hu has been the beamline scientist at X-17C since 1990. The X-17C system includes a θ -2 θ goniometer with a large detector arm, an optional χ -circle for single-crystal diffraction

studies, and an intrinsic Ge detector. A table-top Kirkpatrick-Baez mirror system provides a doubly focused beam. For many years, this station was restricted to energy dispersive X-ray diffraction (EDXD). In 2004-5, a sagittally bent double crystal Laue monochromator was developed for the X-17C hutch in collaboration with NSLS staff. The monochromator can provide 20-40 keV x-rays. Monochromatic angle dispersive x-ray diffraction (ADXD) experiments at 23, 25, and 30 keV energies are currently in operation, in addition to traditional energy dispersive mode.

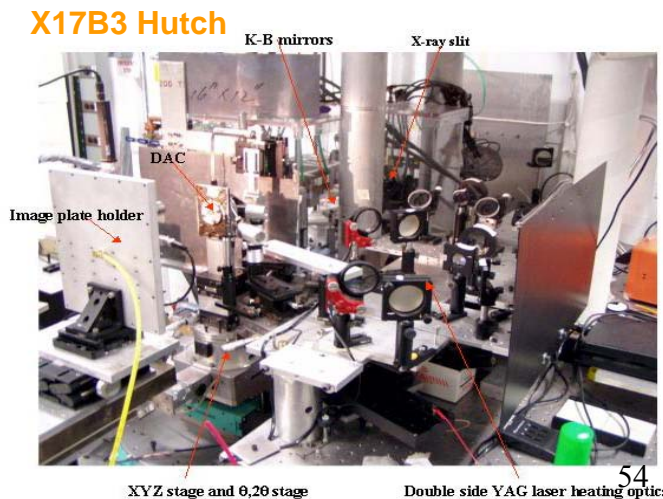


X-17B3:

X-17B3 beamline is available for energy dispersive and monochromatic experiments. It features a double-sided laser heating system with an Nd:YAG laser. The X-17B3 beamline runs 25% of the time in dedicated mode with an additional 25% available in parasitic mode when the X-17B2 (multi-anvil) station is running. Dr. Quanzhong Guo has been beamline scientist at NSLS since 1999.

A monochromator with 4 Laue crystals was designed and installed on the X-17B3 beam line. A key feature of this device is that it maintains the x-ray position when switching from EDXD to ADXD. The device has been tested and used successfully in experiments, but alignment has proven difficult and as a result the x-ray flux levels achieved have been below expectation. Our current plan is to work with NSLS staff to replace this monochromator with a Laue-Laue monochromator (provided by NSLS). We expect the new device together with improved K-B optics will result in much improved x-ray brilliance and thus better utilize the high-energy, high-brightness wiggler source.

The X-17B3 station was completed in 2003 and a two-circle goniometer previously used in X-17B1 is now set up there. A Kirkpatrick-Baez mirror system was developed which can currently produce a double-focused beam of $\sim 10 \times 20 \mu\text{m}$ in size. A double-sided laser heating system using a Nd:YAG laser has now been installed and is in operation. This system mainly uses components from the laser heating system previously used at X-17B1 in the 1990s that pioneered the now widespread coaxial double-sided heating design. The temperature measurement system was recently upgraded and tested. The system is



open to users and has been used successfully to heat materials at pressures as high as 90 GPa.

User Support Laboratory:

Sample preparation and user support capabilities include optical microscopes, mechanical and EDM micro-drilling machines, argon loading, and a ruby fluorescence system. Access to Raman and IR spectroscopy can be obtained through the U2 infrared facility.

On-going Equipment Development and Upgrades:

The DAC-XR facility has been extremely productive for a long number of years, but operated with a minimal budget during the first stage of COMPRES. The beamlines are heavily used and most of the budget has been for beamline support staff and there was relatively little money for equipment upgrades and development.

Sample preparation lab



As a result, upgrades and improvements to the facility are urgently needed in order to maintain this productivity. In 2006, with new funding (\$87k) from a supplemental budget request by COMPRES to the NSF, we have begun to implement some of the most urgent upgrades including new beamline control and data processing computers, new motor controllers, motorization of monochromators, slits, and KB mirrors, and replacement of worn out motors and stages. Other planned upgrades for this year include purchase of diamond cells and anvils, improved cryogenic loading capabilities, and improved clean-up slits. The Kirkpatrick-Baez mirror system will be overhauled with the aim to achieve $<10 \mu\text{m} \times 10 \mu\text{m}$ beamsizes at both DAC stations.

Management History

Prior to 2002, DAC-XR was developed and operated by PRT members (NRL, LLNL, ASU, and Carnegie Institution of Washington). With the establishment of COMPRES, DAC-XR was converted into a community facility that was managed by H-k. Mao of the Carnegie Institution. On September 1, 2005, the operations of NSLS X-

17C and X-17B3 diamond cell beamlines were transferred from Carnegie Institution of Washington (Mao PI) to University of Chicago (M. L. Rivers PI). Beamline scientists Drs. Jingzhu Hu and Quanzhong Guo became University of Chicago employees at that time.

At the same time, a new agreement with the NSLS for these beamlines was instituted. This agreement converted the beamlines to “Facility Beamlines” with a “Contributing User” agreement with COMPRES. The main differences from the previous PRT arrangement are:

- The amount of general user time has increased from 25% to 50%.
- The amount of COMPRES time is 50% of the available beamtime on each beamline.
- The NSLS assumes responsibility for the operation of the “beamline” (optics, safety system, etc.), while COMPRES is responsible for the operation of the experimental stations.

Peer-Reviewed Publications for DAC-X-ray at NSLS

2006

He, D. W., and T. S. Duffy, Static strength of tungsten to 69 GPa, *Physical Review B*, in press, 2006. (X17C)

Speziale, S., S. R. Shieh, and T. S. Duffy, High-pressure elasticity of calcium oxide: A comparison between Brillouin scattering and radial x-ray diffraction, *Journal of Geophysical Research*, B02203, 2006. (X17C)

2005

Duffy, T. S., Synchrotron facilities and the study of deep planetary interiors, *Reports of Progress in Physics*, **68**, 1811-1859, 2005. (X17C)

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B.1.c Infrared Diamond-Anvil High-Pressure Facilities at National Synchrotron Light Source

Operated by Carnegie Institution of Washington [R.J. Hemley and Z. Liu]

Current funding for 2002-2007: \$545K

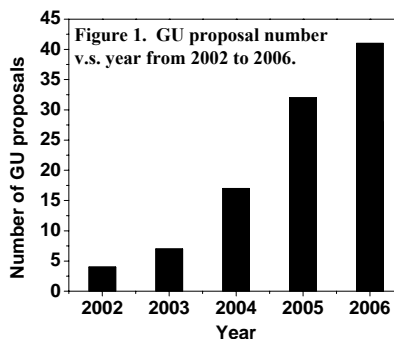
Summary

High-pressure spectroscopy provides crucial and often unique information on the properties of Earth and planetary materials from near-surface conditions to those of the deepest interiors. Vibrational infrared (IR) spectroscopy, for example, provides detailed information on bonding properties of crystals, glass, and melts, thereby yielding a microscopic description of thermochemical properties. Using of synchrotron radiation for infrared studies substantially improves our ability to probe microscopic samples (including in situ measurements under extreme conditions), due to its high brightness and broad-spectrum distribution. Beamline U2A on the VUV ring of the National Synchrotron Light Source is the first, and probably still the only, dedicated high pressure IR beamline. It has many unique capabilities compared to other high-pressure x-ray beamlines. Beginning in May 2001, COMPRES provided operational funding for the facility. In return, the IR beamline gives all COMPRES users access to this world-class facility and makes frontier high-pressure spectroscopic measurements as well as a diversity of other microspectroscopy experiments possible.

Summary of the Progress Achieved in the Five Year Period of COMPRES from May 1, 2002 to April 11, 2007

Growing user community, education, and outreach

Unlike the synchrotron x-ray technique that has been widely used for high-pressure research for a long time where there is a large user base, just a few groups took the advantage of the synchrotron IR spectroscopy technique for their high-pressure studies until 2000. Therefore, the U2A beamline not only provided access for users but also played a very important role in increasing and expanding the user community during past years. A steady increase in the number of general user proposals was seen with time;



a key factor reflected in beam time demand, shown in Fig. 1 (data provided by the NSLS user office). It should be pointed out that we offered quite a few PRT beam times to first time users in order to simplify the procedure for beam time allocation in the early years. Therefore, the actual number of experiments performed at the facility are many more than the numbers of the GU proposals.

In addition, the infrastructure of COMPRES has also been very helpful for us to attract users from the community, via the COMPRES annual meetings and the various sponsored workshops. Recently, we organized a COMPRES sponsored workshop on Synchrotron Infrared Spectroscopy for High Pressure Geoscience and Planetary Science at the NSLS, Brookhaven National Laboratory on November 3-5, 2005. The IR workshop was suggested, promoted, and supported by the members of the COMPRES Executive Committee. It was a great success in terms of the excellence of the lectures, broad attendance including many new potential users, student participation, extensive program, and the hands-on experiences for new users. More than 50 attendees took part, which was the maximum allowed by the budget and lecture room (see Fig. 2). The feedback from attendees has been very positive overall. The workshop not only increased the visibility of the IR facility in the community but also provided an opportunity for students and post-docs to do real experiments at the U2A beamline immediately after the workshop. One of these results was presented at the AGU meeting last fall (W. Montgomery et. al., *Eos Trans. AGU*, 86(52), Fall Meet. Suppl.)



Figure 2. Group photo from the IR workshop

The U2A IR facility has attracted many high-pressure groups throughout the country and around the world during the past four years. These activities play an important role fulfilling the mission of COMPRES. Some users are just starting and still others that are in the preliminary planning stages, but in all cases the infrastructure made possible by COMPRES has given leverage to work on a number of exciting new research directions. Recent U2A users/collaborators include faculty and students from the many U. S. and non-U. S. institutions are given in an Appendix. Finally, access to the IR

facility has become important to many summer interns, including undergraduate and graduate students from institutions such as SUNY, MIT, National Cheng Kung University, Colby College, and Indiana University-South Bend during the past years.

Technical breakthroughs and scientific highlights

We now describe technical advances and just a few of the scientific highlights at the beamline, focusing mainly on results of the past year. Additional information and summaries of earlier results can be obtained from previous COMPRES Annual Reports.

1. Improved beamline performance and synchrotron far-IR spectroscopy of small molecular compounds at high pressure and room/low temperature

The performance of the “old” U2A beamline at long wavelengths was found to be significantly less than ideal. In contrast, a somewhat comparable beamline (U10A) has reasonably good far-IR performance. In particular, the measured far-IR brightness at U2A is 4 to 5 times lower than that at U10A. The suspected cause is related to the principle difference between the two beamlines. U2A uses 38mm diameter optics and nearly 14 meters of beam pipe to bring the IR from near the beamline front-end to the spectrometer endstations. In contrast, U10A uses 57mm diameter optics to transport the beam a distance of about 6 meters. In order to reduce the beamline losses due to diffraction, we made a thorough to the vacuum pipe system for the beam delivery as well as the microscopes at the U2A endstation in 2002. Figs. 3 show that increasing the U2A transport mirrors from 38 mm to 76 mm leads to a significant improvement in the quality of the beam image and the far-IR performance. Many high profiled works have been benefited from the upgrade and a few selected highlights are shown as following.

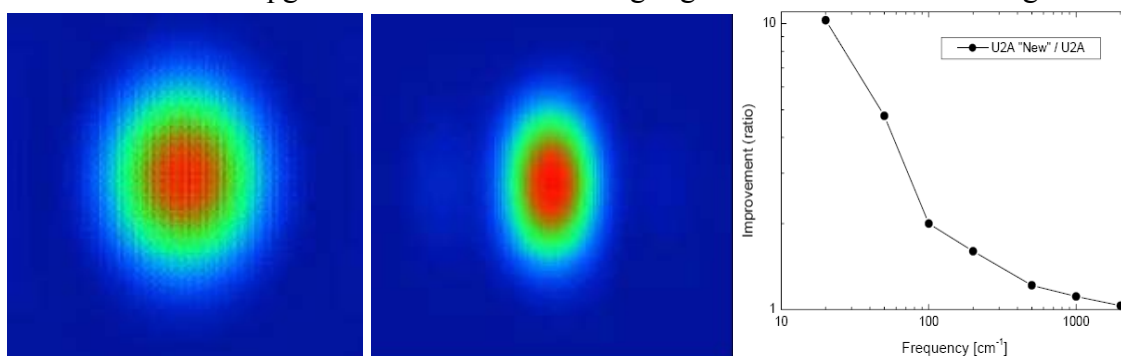


Figure 3. Diffracted beam profile for $\lambda=200\ \mu\text{m}$ ($50\ \text{cm}^{-1}$). Left: U2A with old 38 mm diameter optics. Center: U2A with 76 mm diameter optics. The red oval represents the profile of the collection optic at the end of the 12 m transport length. The scale is the same for both images. Right: Relative performance improvement for an upgraded U2A beamline (same length as present, but with 2 \times larger optics).

a. Synchrotron Far-IR spectroscopy and the Transformation in Ice VIII

New far-IR measurements were carried out by Klug *et al.* [*Phys. Rev. B* **70**, 144133 (2004)] to test proposed pressure-induced phase transformations in H₂O and D₂O 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-temperature 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-temperature high-pressure far-IR and theoretical calculations give an anomalous transformation in ice VIII that supports the experimental

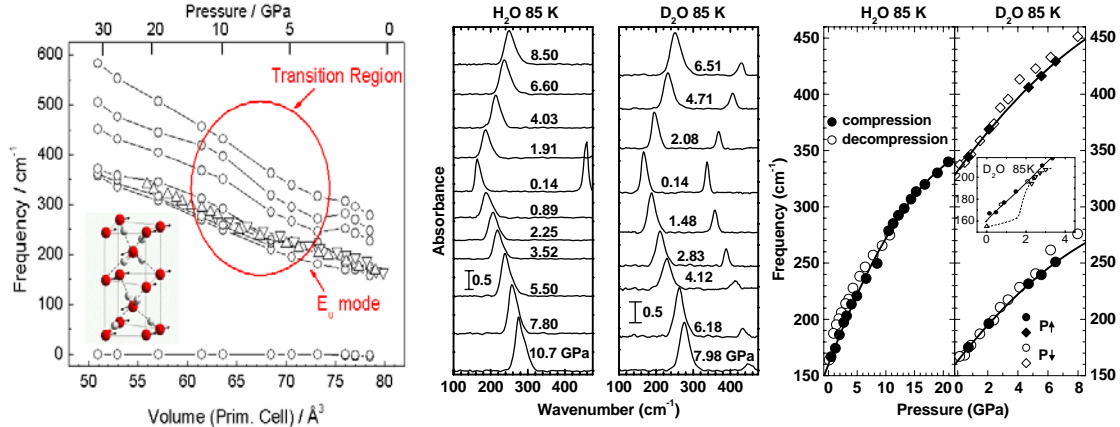


Figure 4. Right: The calculated pressure dependence of the lattice modes of ice VIII. Center: The measured low-temperature far-infrared spectra of ice VIII in a diamond anvil cell. The low frequency peak is the infrared active E_u mode. The higher frequency peak is an infrared active mode due to molecular librations. Left: The frequency versus pressure results for the E_u infrared active mode compared with previous measurements and suggested pressure dependence (Inset).

results from a high-pressure neutron diffraction study. Our study provides a detailed and definitive new interpretation regarding this phase transformation. Further studies of dense ice phases will continue to characterize possible new phase transition behaviors using our combination of theoretical and experimental methods at extreme conditions. Our ultimate goals are far-IR studies above 100 GPa at variable temperatures.

b. Phonon Density of State in Ice VII and Synchrotron Far-IR Spectroscopy up to 80 GPa

Synchrotron far-infrared spectroscopy and linear response density-functional theory provide new insight for the high pressure structural changes and lattice dynamics of ice VII at high pressures. The experimental studies provide detailed high pressure far-infrared absorption data in the pressure range 3 – 80 GPa at 300 K. First-principles lattice dynamics results on ice VIII, the structurally very similar proton ordered form of ice VII,

are combined with the far-infrared data to characterize the details of the lattice dynamics

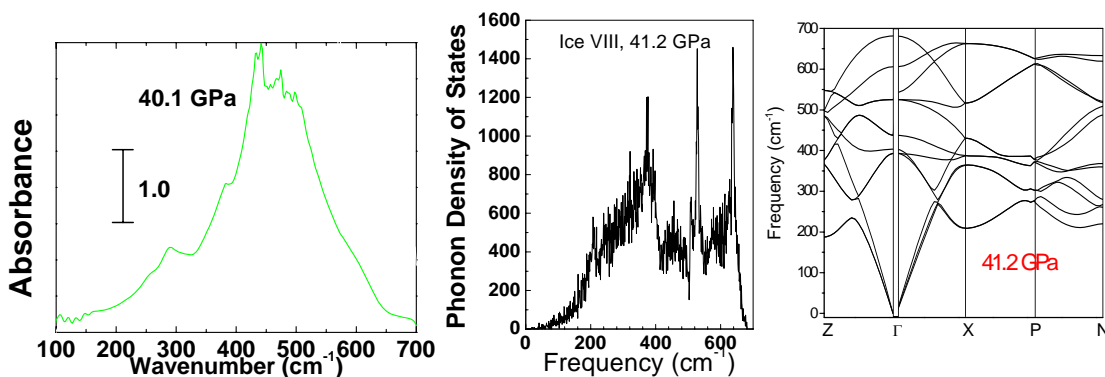


Figure 5. Synchrotron far-IR spectra of ice-VII (right), calculated phonon density of states (center), and the phonon dispersive curve at room temperature and ~ 40 GPa.

of ice VII to pressures that include the formation of ice X where centro-symmetric hydrogen bonds exist. (Z. Liu, Phys. Rev. Lett, to be submitted)

c. Hydrogen-Bond Symmetrization in Methane Filled Ice

Clathrate hydrates are compounds consisting of cages of hydrogen bonded water molecules surrounding guest molecules or atoms. Recent studies revealed the formation of new high-pressure phases of the CH_4 clathrate. This finding has important implications for hydrogen bond research as well as providing a new model for astrophysical studies such as the formation and evolution of the composition of Titan, a moon of Saturn. There has been an intense interest in this area of research. The CH_4 clathrate also has significant importance as a potential energy source.

High-pressure, variable temperature infrared spectroscopy and first-principles calculations on the methane filled ice structure (MH-III) at high pressures are used to investigate the vibrational dynamics related to pressure-induced modifications in hydrogen bonding. Infrared spectroscopy of isotopically dilute solutions of H_2O in D_2O is employed together with first-principles calculations to characterize proton dynamics with the pressure induced shortening of hydrogen bonds. Fermi resonances are identified and shown to dominate the infrared spectrum in the pressure region between 10 and 30 GPa. Significant differences in the effect of Fermi resonance that are observed between 10 K and 300 K are related to the double-well potential energy surface for hydrogen-bonded protons in the lattice and of quantum effects on the proton dynamics. [D. Klug, J. Phys. Chem., submitted].

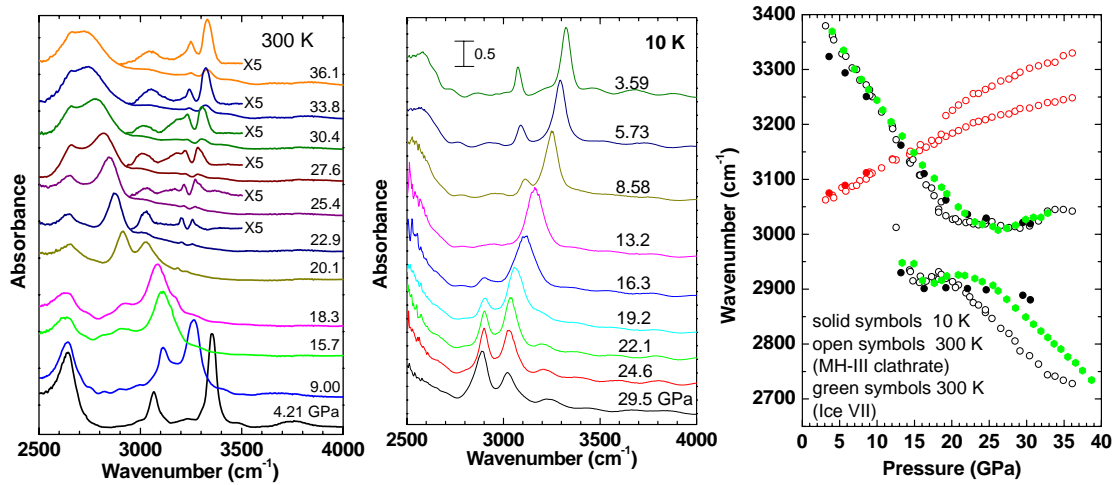


Figure 6. High-pressure synchrotron IR spectra of the CH₄ clathrate at room temperature (right), 10K (center), and pressure dependence of the frequencies of OH and CH stretching vibrational modes (right).

2. Synchrotron IR Spectroscopy of Hydrated Minerals at High Pressure and High Temperature

a. Effects of Water on the Behavior of MgSiO₃-Clinoenstatite at High Pressure

Just 1000 ppm (0.1 wt%) of H₂O stored in major minerals of Earth's upper mantle would represent more than one ocean-mass equivalent of liquid water in the interior. The incorporation of water (as hydroxyl) into enstatite (MgSiO₃) was investigated using synchrotron-IR spectroscopy on the U2A beamline at the NSLS. We monitored the O-H stretching bands in hydrogen-bearing enstatite single-crystals as a function of pressure in a diamond anvil cell (DAC). Special attention was paid to changes in hydrogen bonding on transformation from the low-pressure polymorph (LCEN) to the high-density clinoenstatite (HCEN) at around 6-8 GPa. By studying samples containing different water contents, we mapped out the transformation boundary as a function of water content and pressure, which was also confirmed by in-situ Raman spectroscopy. The use of synchrotron-IR facilitated observation of very weak O-H stretching from low-level concentrations of water on the order of 300-600 ppm by weight.

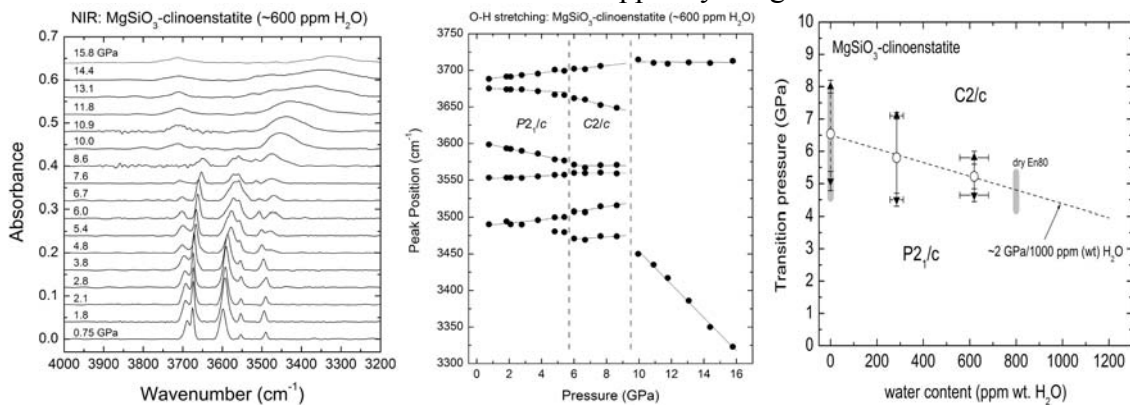


Figure 7. Left: High-pressure IR-absorption bands due to O-H in clinopyroxene measured at U2A. Center: Variation of the O-H stretching band positions with pressure. Here we show how changes in the hydrogen bonding environment at the LCEN ($P2_1/c$) to HCEN ($C2/c$) phase transformation are detected using synchrotron-IR spectroscopy. Right: Summary of the transformation pressures from IR and Raman on compression (up triangles) and decompression (down triangles) for different water concentrations in clinoenstatite. Water shifts the transformation pressure to lower pressures and reduces the hysteresis interval.

Single-crystal samples of LCEN with varying water contents were synthesized in a multianvil press at 16-18 GPa and 1100°C at the Geophysical Laboratory. Samples containing ~300 and ~600 ppm H₂O were selected for the high-pressure IR study. In the hydrous samples, absorption bands in the region of O-H stretching were monitored to 16 GPa (Fig 8). Whereas the two main bands at 3600 and 3675 cm⁻¹ shift to lower wavenumbers on compression, consistent with increased hydrogen bonding strength at high pressure, three other minor bands shift to higher wavenumbers with pressure. At around 6 GPa, a discontinuous change in the slope of dv/dP was observed in most bands, which we attributed to the LCEN-HCEN phase transformation. The transformation pressure was confirmed with in-situ Raman spectroscopy using very fine pressure steps and found to be at 5.8(3) GPa. The transformation at 5.8 GPa on compression is about 2 GPa lower than that observed for the dry sample, which transformed at ~8 GPa on compression (based on the Raman). Upon decompression, all samples back-transformed to LCEN at around 4.5 GPa. The results indicate that hydration narrows the transformation hysteresis and that water reduces the transformation pressure (P_T) by about 2 GPa per 1000 ppm H₂O, if P_T is taken to be the mid-point of the hysteresis. If hydroxyl defects affect the orthoenstatite to HCEN transformation similarly in the mantle, a shift in P_T of 1 GPa would uplift any corresponding seismic discontinuity by about 30 km. Thus, relatively small amounts of water may have seismically detectable effects on the behavior and properties of the upper mantle.

Synchrotron-based IR is highly suitable for studying the behavior of mantle silicates containing relatively low concentrations of water (10's to 100's of ppm by weight). Here we have shown how new synchrotron-IR studies will be carried out on other OH-bearing mantle phases to study hydrogen bonding at pressure and applied to interpretation of mantle velocity structure in potentially hydrous regions of the mantle. [E. Littlefield et al., Eos Trans. AGU, 86(52), Fall Meet. Suppl., Abstract MR41A-0899, 2005)

b. High-pressure vibrational study of dense hydrous magnesium silicate

10Å Phase Dense hydrous magnesium silicates (DHMS) could be important hosts for H₂O in the Earth's mantle and subduction zones, and their dehydration may be responsible for deep focus earthquakes. Among these phases, the so-called 10Å phase (Mg₃Si₄O₁₀(OH)₂.*n*H₂O) is characterized by a phlogopite-like structure which accommodates molecular water in the interlayer. However, the amount of interlayer water, its precise structural position and response upon compression remain uncertain. To better understand the high pressure behavior of hydroxyl groups and interlayer water in 10Å phase, we have conducted *in situ* synchrotron IR and Raman spectroscopic

measurements up to 20 GPa at 300 K. The high brightness and spatial resolution of the synchrotron IR radiation available at the U2A beamline at NSLS, high-quality mid-IR spectra with high signal/noise ratio have been collected in the DAC. The 10Å phase samples were synthesized at 6.5 GPa and 650 °C in a Kawai-type multianvil press using talc and excess water as starting materials. At ambient conditions, the IR spectrum shows a strong band at 3675 cm⁻¹ and two less intense and broader bands at 3258 and 3589 cm⁻¹. With increasing pressure up to 5.5 GPa, the band at 3675 cm⁻¹ displays a linear negative frequency shift. Near 6.2 GPa a new band emerges at 3680 cm⁻¹ and its intensity progressively increases with pressure up to 15.5 GPa, indicating major changes in the hydrogen bonding. All pressure-induced changes in the IR spectra are fully reversible upon decompression and occur without any noticeable hysteresis. On the basis of combined high pressure IR and Raman results, it is possible to reevaluate the assignment of the OH-vibrational bands of the 10Å phase and constrain the response to compression of SiO₄ tetrahedra, MgO₆ octahedra, hydroxyl units, and molecular water [C. Sanchez-Valle et al., AGU Fall Meeting, 2005].

c. Synchrotron Infrared Spectroscopy of Synthetic P2₁/m Amphiboles at High-pressure

Systematic mid-IR measurements of synthetic Na(NaMg)MgSi₈O₂₂(OH)₂ amphiboles up to 30 GPa were carried out by Gianluca Iezzi *et al.* These minerals represent a key double-chain silicate to model the P2₁/m – C2/m phase-transition in A-site filled amphiboles. It has P2₁/m symmetry at room temperature, and reverses to the usual C2/m space-group of monoclinic amphiboles at ~ 257 °C. The spectrum of the Na end-member shows three bands: (A) at 3740 cm⁻¹, (B) at 3715 cm⁻¹ and (C) at 3667 cm⁻¹, respectively. The higher-frequency bands are assigned to two non-equivalent H atoms interacting with ^ANa; this pattern is typical of an amphibole with a P-lattice. The ^BLi-bearing amphiboles show an additional fourth, minor band at 3690 cm⁻¹. With increasing pressure, all bands linearly shift toward higher frequency. At 20.8 GPa, the peak centroid of the main (A) band is > 3800 cm⁻¹. The A and B bands merge into a single broad absorption band, and the P value at which the A-B doublet disappears is a function of the B-site occupancy. For the ^BNa end-member, the A and B bands merge at around 18 GPa; for sample 406, with nominal B-site composition (Na_{0.2}Li_{0.8}Mg₁), the A and B bands merge around 13 GPa. These results show that the Na(NaMg)MgSi₈O₂₂(OH)₂ amphibole undergoes a P2₁/m – C2/m phase transition at high pressure, and that the transition pressure, P_C, is a function of the aggregate dimension of the B-site [G. Iezzi et al., *Am. Mineral.*, **91**, 479 (2006)].

d. High Pressure Far-IR Absorption Spectroscopy of Muscovite

Muscovite is a geologically-important hydrous mineral because it is common in both igneous and metamorphic rocks and is accordingly a significant host for mineralogic water storage in the Earth's crust. High-pressure x-ray diffraction studies suggested a loss of long range crystalline order starting at 18 GPa and pressure-induced amorphization by 27 GPa. To obtain a better understanding of the thermodynamic response of muscovite to compression, Henry Scott *et al.* have used infrared spectroscopy to sample vibrational modes spanning both the mid and far portions of the infrared spectrum using a diamond anvil cell as a both an optical window and pressure generating

device up to 25 GPa. Nine FIR features between 100 and 550 cm^{-1} have been observed that have been previously documented in ambient pressure spectra. All modes shift monotonically to higher frequencies with increasing pressure; there are notable changes in relative intensities, but this is likely a result of enhanced preferred orientation due to the nature of compression in a DAC. Based on the lack of abrupt changes in the lattice modes, it appears that pressure-induced amorphization is not likely to occur over this pressure range and that muscovite can be compressed metastably well beyond its known stability field [H. Scott et al, to be published].

e. FTIR Spectroscopy of the Mixture of N_2 and H_2O under High Pressure and Temperature Conditions

Accurate equations of state (EOS) for mixture reactions using a high pressure and high temperature DAC are essential for understanding the nature of intermolecular forces and the behavior of simple molecules under extreme conditions. The techniques used here to solve for pressure (P), temperature (T), and composition (X), and their results are relevant to a broad range of important processes. For example, interest in fluid-fluid phase equilibria at high pressures and in supercritical fluid mixtures has increased greatly over the last two decades for scientific and practical reasons: intermolecular interactions and general aspects of critical behavior can now be studied experimentally and computational capabilities to describe these systems have been improved. Models capable of accurately characterizing PXT properties can provide important tools for understanding many natural processes occurring in the earth's crust and mantle. Binary systems in which water is one component or systems that consist of one highly polar and one non-polar partner are also of particular interest. Becky Streetman *et al.* studied a mixture of N_2 and H_2O under high pressure and high temperature at U2A, focusing on the measurements of phase separation boundaries of water and nitrogen mixtures over the range of experimentally accessible temperatures and pressures. An externally heated DAC was used for these experiments. At the coalescence temperature, initial results indicate that the mixture is homogeneous and that a clathrate is formed [B. Streetman, to be published].

f. High Pressure Phase Transition and Partial Dehydration of Gypsum

Gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) 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 to 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 the U2A beamline and EDXD and angle dispersive x-ray diffraction (ADXD) at the 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 5 GPa. 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:



[Z. Liu et al, IUCr-COMPRES High Pressure Workshop Non-ambient Crystallography: The Science of Change December 4-7, 2003, at Lawrence Berkeley National Laboratory in Berkeley, California]

g. High-Pressure Infrared Absorption Spectroscopy of Katoite Hydrogarnet

Lager *et al.* studied a D-rich katoite hydrogarnet, $\text{Ca}_3\text{Al}_2(\text{O}_4\text{D}_4)_3$ 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. [Am. Mineral., **90**, 639 (2005)].

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B.1.d Multi-anvil High Pressure Facilities at National Synchrotron Light Source

Operated by Stony Brook University [D.J. Weidner and M.T. Vaughan]

Current funding for 2002-2007: \$1654K

The National Synchrotron Light Source offers the COMPRES community an opportunity for x-ray studies using beamline X17. This superconducting wiggler continues to provide an x-ray source that is competitive with third generation sources for x-ray diffraction studies. In addition, the current plans to build the next x-ray synchrotron at Brookhaven (NSLSII) provides a significant future at the NSLS that requires the presence of COMPRES research to assure access to the NSLSII.

Many high pressure investigations are best carried out in a multi-anvil, large-volume device. Those that require a large sample volume (cubic mm) and/or a uniform temperature environment in the 1000 – 2000K range cannot be done in the diamond anvil cell. The large anvil devices are limited to lower pressure than the diamond anvil cells, but do provide the best sample environment for conditions that match those of the Earth to depths into the top portion of the lower mantle. The diamond anvil cell is the system of choice when matching the environment of the core – mantle boundary or of the core itself.

This beamline is the first beamline in the US with a multi-anvil, large-volume high pressure system. This beamline has pioneered high-pressure, high-temperature acoustic velocity measurements on mineral systems and high-pressure, high-temperature rheology experiments. The beamline saw the first synchrotron measurements of melt acoustic velocity and density (by x-ray absorption) in the US involving synchrotron radiation. This beamline continues to contribute to high pressure crystallography, phase equilibrium, and equations of state.

During these past five years, the facility has benefited from significant financial input from Brookhaven National Laboratories, the Department of Defense, and Stony Brook University. Brookhaven Labs provided the largest share of funds to build new hutches for the multi-anvil system and the diamond anvil system. Previously, we operated from a small hutch, shared with other operations. We needed to move the press into the hutch at the beginning of our beamtime and move it out at the end. This restricted the type of experiments that could be done as well as the size of the press that is used in the multi-anvil system (200 tons). We designed the new hutch so that it was large enough to accommodate a 2000 ton press.

In the new hutch, we have been able to permanently install our existing press, and now we have built a monochromatic side station (funded by the DoD) within the main hutch. This will allow us to run two experiments simultaneously.

In the current mode, the X17B beamline is time-shared 50:50 between high pressure research and material sciences. We built the hutches so that during the high pressure time, the multi-anvil hutch and the diamond cell hutch can operate simultaneously. We hope to nearly double the effective multi-anvil time with the opening of the monochromatic side station (a single bounce monochromator delivers a

separate beam that diverges from the main beam). NSLS now has plans to open a new beamline, X17A. This new beamline will attract the material scientists that use our X17B beamline. We expect that our share of X17B will increase to about 80%. We also plan to use some of the X17A beam time with a portable large-volume system.

We operate with the principle that all user beamtime is allocated on the basis of proposals. These proposals are evaluated by the NSLS evaluation panel. NSLS assigns half of our beamtime on the basis of their evaluation. The recipients of this beamtime include the COMPRES community as well as physicists, chemists, and material scientists (it is dominated by COMPRES users). The rest of the time is assigned by us, on the basis of the NSLS ranking, but with a COMPRES filter. That is, we increase the priority of the COMPRES community relative to others for the final allocation of time. Our assignments are reviewed annually by the COMPRES Facilities Committee to assure that it has been properly executed. All Stony Brook users compete with everyone else for beamtime. We will on occasion retain some time for system development (less than 10%).

A workshop was held in late February, 2006 to assess the science objectives and needs for the high pressure program at the NSLS for the next five years. The plans in this proposal are largely defined by the discussion and recommendations of the workshop.

Scientific Highlights X17B2 in period 2002-2007

- ***Deformation experimental technique breakthrough and scientific research:*** A new high pressure deformation apparatus, called the Deformation DIA (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; the top and bottom ram can advance or retreat independently to deform the sample. The sample stress and strain can be measured by x-ray diffraction and radiograph imaging. X-ray diffraction along different directions relative to the principal stress axis yield an accurate measurement of stress in the sample to 100 MPa, and correlation of strain-mark images on the radiograph provides a precise strain measurement to 10^{-4} . More remarkably, the technique avoids the uncertainty introduced during the deformation and friction modeling of the pistons in the high pressure cell. Therefore the deformation experiments can be carried out far beyond the pressure limits (3GPa) of conventional deformation apparatus. Deformation study on olivine at 10GPa has revealed revolutionary information about the activation volume of this mineral, which is significantly less than what people had believed. This result has an important impact on the understanding of mantle rheology.
- ***Understanding the strength of perovskite:*** The strength of the dominant lower mantle mineral, $\text{Mg}_{0.9}\text{Fe}_{0.1}\text{SiO}_3$, to 10 GPa and 1000K has been measured. The study indicates that perovskite is the strongest mantle mineral. It also has a unique characteristic during stress relaxation: insensitivity to temperature. These findings help us allocate the viscosity jump boundary in the mantle, understand the deflection of the subduction slab at the boundary of transition zone and lower mantle, and illustrate why deep earthquakes occur.
- ***Ultrasonic measurements of non-quenched phases at high pressure and temperatures:*** Studies of acoustic velocities in minerals have moved forward to

- measure lower mantle minerals and non-quenchable phases. Acoustic data for MgSiO₃ perovskite, Al-doped MgSiO₃ perovskite, non-quenchable CaSiO₃ perovskite and high pressure pyroxene phases. The 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 FeS, a possible source of light element in the core. Melting volume of this material has been measured at 4GPa, and the data is used to calculate the slope of melting curve. Acoustic measurements of molten material at high pressure have also been pioneered. Characteristic signals of P-wave and S-wave are observed when phase transitions and melting happens.
 - **Polycrystalline stress field:** MgO data using the D-DIA give new insights into the distribution of stress among the grain subpopulations. Multi-phase aggregates have been the focus of recent studies. Different stresses in different samples show the organization of the grains required to support the stress.
 - **Deformation experimental technique breakthrough and scientific research:** The Rotational Drickamer Apparatus, developed by Shun Karato of Yale has been deployed on our beamline. Karato brings his research team and high pressure apparatus to the hutch. Strain and stress have been successfully measured with this device; it is capable of very large strains because of the rotational mechanism, and very high pressures (in excess of 20 GPa) because of the Drickamer style pressure generation.
 - **High pressure Rheology of olivine:** Olivine continues to be a central theme for D-DIA experiments. Single crystal studies by Raterron demonstrate a pressure induced change in slip systems. Li, *et al.* and Mei, *et al.* are finding a low activation volume for olivine (0-5 cc/mol), while Karato finds evidence for a larger value (15 cc/mol). The story here is still unfolding, but we now have the facilities to address this important issue.

Appendix: X17 Multi-anvil milestones

- 1990 **Experiments began in X17B1** using the Multi Anvil Press (MAP). This involves sharing the high energy, white radiation hutch at the NSLS with Carnegie's Diamond Cell (DAC) high pressure program, as well as medical, material science, and tomography programs. All the equipment for each of these programs must be moved into and out of the hutch during experimental changeover.
- 1994 **High Temperature Rheology experiments.** This technique depends on observing stress relaxation as a function of time during heating. The sample is placed in a high stress environment and heated. The elastic strain is measured using diffraction, and as the stress relaxes, the differential strain is also reduced.
- 1995 **T-cup cell** This is a miniature 6-8 system capable of 29 GPa in a 200 ton press
- 1997 **Acoustic velocity** Simultaneous measurement of acoustic velocities using ultrasonic interferometry and pressure, at high pressure and temperature
- 1998 **Time resolved Monochromatic IP** An imaging plate was installed on a translating stage to enable tracking of phase and/or structural changes during pressure and/or temperature changes

- 2000 **Strain measurements** Measurement of macroscopic, plastic strain using imaging. This entailed use of hard transparent anvils
- 2001 **Beginning of COMPRES**
 - Conical slit stress measurement. This enables measuring elastic strain in two (or more) different directions with respect to non-hydrostatic stress direction, an enhancement over the initial unidirectional strain measurements pioneered in 1994.
 - The possibility of constructing two new hutches dedicated to high pressure science was floated.
 - 38 runs were performed
- 2002 **Construction of new hutches** for Diamond Anvil and Multi-Anvil high-pressure science began.
 - D-DIA (for steady state deformation). This new deformation device enables changing the vertical load on the sample while maintaining a constant pressure. A trial run was made using a borrowed D-DIA, with the pumping system located in the construction area for the new hutch.
 - 32 runs were performed
- 2003 **New Hutch for X17B2** This greatly increased the efficiency of operations by the elimination of re-installation of the equipment for each beamtime, allowing a doubling of the annual number of runs, with no actual increase in beamtime.
 - Funding made available for building and installing a dedicated D-DIA in X17B2
 - NSLS General User Proposal system adopted for all users. This increased the number of proposals, especially from local users.
 - 79 runs were performed
- 2004 **Time-sharing with X17B3** Installation of a removable, evacuated beam pipe through the X17B2 hutch made practical simultaneous use of the DAC and MAP.
 - First experiments on imaging of molten metals for measurement of viscosity
 - Rotational Drickamer Apparatus (RDA) from Yale University temporarily installed and used. A decision made to request funding for a permanent installation of such an apparatus
 - 111 runs were performed
 - 100 runs were performed
- 2005 **Monochromatic Side Station** Funding was obtained from DOD for the construction of an additional beamline, within the X17B2 hutch
 - A MAR345 imaging plate system was purchased for the monochromatic side station
 - Construction began and completed for the table to support the MAR345, a set of slits, and a press.
 - First experiments performed using an under-development monochromator. The ability to perform white radiation experiments simultaneously was demonstrated.
 - 100 runs were performed in the first 9 months of the 2005 fiscal year.
- 2006 (proposed) Completion of installation of the monochromatic side station,
 - Installation of a high-pressure device based on a Tcup inside a Paris-Edinburgh Cell
 - Completion of the monochromator
 - Completion of the installation of an imaging system for the side station

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B.1.e Neutron Studies at National Facilities

Operated by Virginia Polytechnic Institute and State University [N. Ross]

Current funding for period 2002-2007: \$210K

Summary

In COMPRES II, the neutron program will play a proactive role in the development of high-pressure neutron research in the United States. This will be done by establishing a COMPRES “task force” for high-pressure neutron research made up members of the COMPRES community and representatives from SNS, HIFR, and LANSCE. The task force will: (i) identify community needs for neutron research; (ii) encourage exchange of information between high-pressure neutron facilities within the United States and abroad; (iii) identify and submit proposals to COMPRES for workshops for hands-on training; and (iv) develop infrastructure development projects as identified by the community and to target, where appropriate, additional resources through which to leverage COMPRES funds. It is proposed that funds be held at COMPRES central to support travel and subsistence for U.S.-based COMPRES researchers to carry out experiments at high-pressure neutron facilities and to support annual meetings of the task force.

Introduction

The future prospects for high-pressure research at neutron sources at the SNS and LANSCE offer a myriad of opportunities that will fully complement the activities at synchrotron X-ray sources. With the development of new high-pressure cells, neutrons will revolutionize our understanding of the role of hydrogen and carbon in the earth and planetary interiors. Neutron studies will also be influential in addressing global issues related to energy and the environment, from storage of hydrogen in fuel cell materials to carbon sequestration. The powerful combination of neutrons and high pressure will open new pathways for the discovery of novel materials with novel properties. COMPRES is currently supporting a neutron program to cultivate scientific interest in exploiting the new opportunities in this field thereby establishing a high pressure neutron culture in the U.S. that will tap into and harness the broad reach of high-pressure science already existing at synchrotron beamlines.

Summary of the Progress Achieved in the Four-Year Period of COMPRES from May 1, 2002 to April 11, 2007

Unlike the synchrotron X-ray technique that has been widely used for high pressure research over a long period and for which there is a large user base, only a handful of people in the COMPRES community were involved in high pressure neutron research at the beginning of COMPRES I. A considerable effort of the COMPRES neutron initiative has been devoted to growing the neutron community.

One of the first activities that the COMPRES neutron initiative helped to organize and support was 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 to attend the workshop and it is significant that they are now all active in neutron research: Darren Locke is now part of the SNAP team; Kim Tait is finishing her PhD at the Univ. of Arizona based on research conducted at LANSCE; Wendy Mao is now a postdoctoral fellow at LANSCE; Megan Elwood Madden is a postdoctoral fellow at ORNL; and Peter Chupas is a beamline scientist at the IPNS. In addition, many graduate students and post-docs supported with COMPRES travel funds are using neutrons routinely in their research. Other researchers who have been supported by COMPRES are listed in an Appendix. Publications are given below.

During the first year of COMPRES, a website (www.crystal.vt.edu/compres) was designed to keep the community informed of upcoming conferences, proposal deadlines, and to provide a means through which educational materials could be distributed to the community.

Dr. Husin Sitepu joined the neutron team in February 2005. One of the roadblocks he encountered in trying to grow the high-pressure neutron community in the United States is the lack of beam time at neutron facilities for high-pressure research. This also poses a problem for the SNAP team who need to find time at neutron facilities to provide a testbed for their equipment and for data gathering and analysis. Dr. Sitepu, with the help of Dr. Anna Llobet Megias and Dr. Yusheng Zhao of LANSCE, tested a Paris-Edinburgh (PE) cell on the HIPD beamline in November 2005. Many problems were encountered because the PE pressure cell had not been used for many years, but Dr. Sitepu is working with Anna and Yusheng to

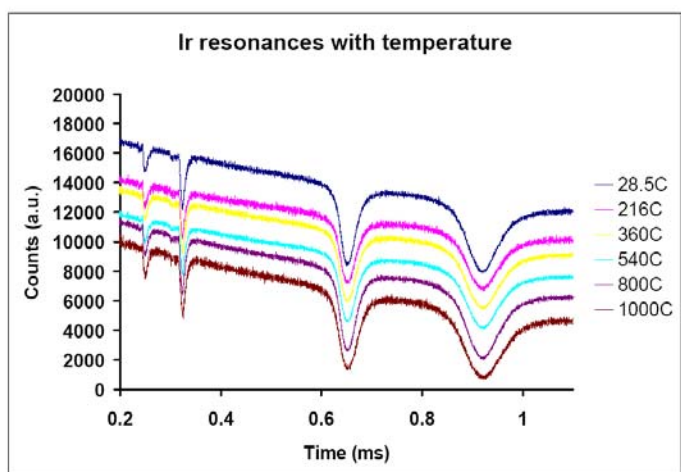


Figure 1: Plot of neutron resonances of iridium (Ir) between 28°C and 100°C.

assess whether this beamline could become viable for high-pressure neutron experiments and whether it can provide a testbed for new equipment development for the SNAP effort. Dr. Sitepu will also be involved with the testing an epithermal neutron resonance detector (preliminary studies have been carried out by Simon Clark at LANSCE) to determine the sample temperature at high pressure using neutron radiography. This technique involves measuring the resonance absorption of high energy neutrons in a thin metal (e.g. Ir) foil held in the centre of the sample. The square of the resonance line width increases with temperature (Fig. 1).

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B.2. Infrastructure Development Projects

B.2.a Pressure Calibration at High Temperature

[Y. Fei, Carnegie Institution of Washington]

Funding for period 2002-2004: \$248K

A correct pressure scale is fundamentally important for interpreting geophysical observations using laboratory experimental data obtained at high pressure and temperature. It also allows us to make comparisons of high-pressure results produced in different laboratories using different experimental and analytical techniques. Metals such as Au, Pt, W, Mo, Pd, Ag, and Cu, whose equations of state are established based on shock compression experiments and thermodynamic data, are commonly used as pressure standards in high-pressure experiments. Commonly used non-metal pressure standards include MgO and NaCl. At room temperature, the ruby fluorescence pressure gauge is extensively used in diamond-anvil experiments. The ruby gauge was calibrated by simultaneously measuring the shift of ruby R_1 luminescent line and specific volume of metal standards (Cu, Mo, Pd, and Ag) as a function of pressure. The established calibration curve based on equations of state of metal standards (Mao et al., 1986) has proven to be accurate, confirmed by direct measurements of pressure by combining Brillouin scattering and X-ray diffraction techniques (Zha et al., 2000).

Accurate determination of pressure at high temperature is more difficult because of large uncertainty in calculating the thermal pressure. Commonly used pressure standards such as Au, Pt, MgO, NaCl generally do not predict the same pressures under the same experimental conditions. In some cases, the calculated pressures based on different standards could differ as much as 4 GPa (Figure 1).

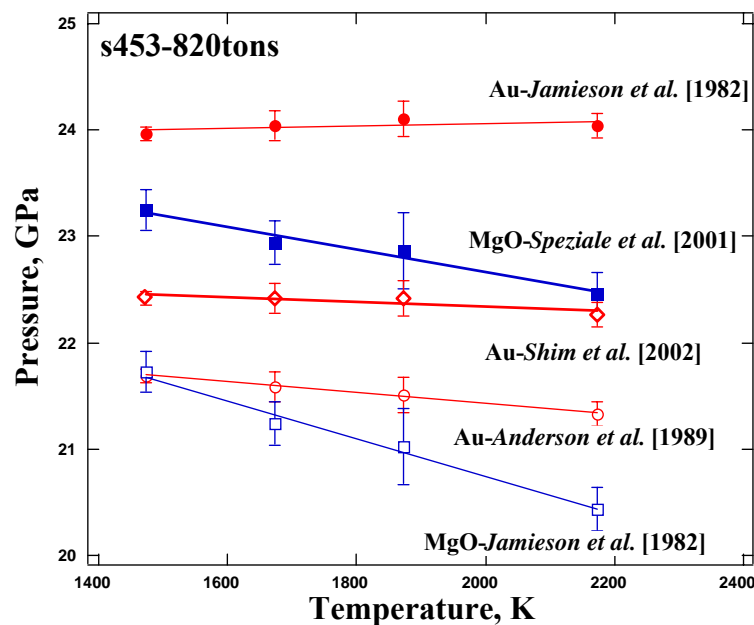


Fig. 1. Calculated pressures using MgO and Au pressure scales at high temperatures. Solid circles, open diamonds, and open circles represent pressures calculated from Au scales by Jamieson et al. (1982), Shim et al. (2002), and Anderson et al. (1989), respectively. Solid and open squares represent pressure from MgO scales by Speziale et al. (2001) and Jamieson et al. (1982), respectively.

The goal of this project is to examine the existing pressure scales at high temperature and to quantitatively determine the relative differences among the different pressure scale.

With recent advances in synchrotron radiation and high-pressure techniques, it is possible to evaluate and compare pressure scales over a wide range of pressure and temperature. Our strategy to attack the pressure scale problem at high temperature is first to establish a self-consistent pressure scale through in situ X-ray diffraction measurements of the primary pressure standards such as MgO, Au, and Pt in a multi-anvil apparatus up to 28 GPa and 2300 K and in a externally-heated diamondanvil cell up to 100 GPa and 1100 K. This effort was led by Y. Fei at the Geophysical Laboratory. The recommended model parameters for the thermal equation of state of MgO, Au, and Pt are listed in Table 1. These equations of state predict consistent pressures. Details are described in recently published paper by Fei et al. (2004).

Table 1. Model parameters for the equations of state of MgO, Au, and Pt

| Parameters | MgO ¹ | Au ² | Pt ³ |
|----------------------|------------------|-----------------|-----------------|
| $V_0, \text{\AA}^3$ | 74.71(1) | 67.850(4) | 60.38(1) |
| K_{0T}, GPa | 160.2(2) | 167(3) | 273(3) |
| K_{0T}' | 3.99(1) | 5.0(2) | 4.8(3) |
| θ_0, K | 773 | 170 | 230 |
| θ_0' | 1.524(25) | 2.97(3) | 2.69(3) |
| q_0 | 1.65(40) | 0.7(3) | 0.5(5) |
| q_1 | 11.8(2) | 0 | 0 |
| $3R, \text{J/gK}$ | 0.12664 | 0.12500 | 0.12786 |

¹All parameters are from Speziale et al. (2001) ($q = q_0(V/V_0)^{q_1}$). ²All parameters except q value are from Shim et al. (2002). ³Fei et al. (2004).

COMPRES PUBLICATIONS

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B.2.b Multi-anvil Cell Assembly Initiative: New Developments and Production

[K. Leinenweber, J. Tyburczy, T. Sharp, Arizona State University]

Current funding for period 2002-2007: \$743K

Previous Developments

The purpose of the COMPRES multi-anvil cell development project is to coordinate an interactive and vibrant community effort to develop novel, well characterized assemblies for conventional and *in-situ* experiments in multi-anvil devices that enable new capabilities and to manufacture and distribute such assemblies for use in the community. The effort now involves 17 of the multi-anvil laboratories in the United States (Table 1). The project has resulted in the development of four cell assemblies for use in conventional multi-anvil laboratories, and four modified assemblies for use in *in-situ* experiments at x-ray beamlines (Table 2). The project has led to the development and introduction of a significant number of novel materials and techniques into multi-anvil research, including injection-molded octahedra, porous mullite pressure media, forsterite thermal insulation sleeves, laser-cut rhenium furnaces (Figure 1), and new methods of putting x-ray windows into multi-anvil assemblies (Figures 1-3). The COMPRES lathe that was purchased in the initial stages of the project has allowed the development of automated notching and slitting of ceramic parts for thermocouples and x-ray access (Figure 2). The thermocouple grooves in the octahedral pressure media are notched on an automated mill. These are all tasks that researchers previously had to do by hand. These developments are documented in two summary publications, one in preparation and one submitted (1, 2). A preliminary report was presented in the COMPRES Newsletter (3). The thermal models that were used in development and characterization of the COMPRES assemblies are detailed in reference (4) (see Figure B2 for an example of a thermal profile calculated using this model).

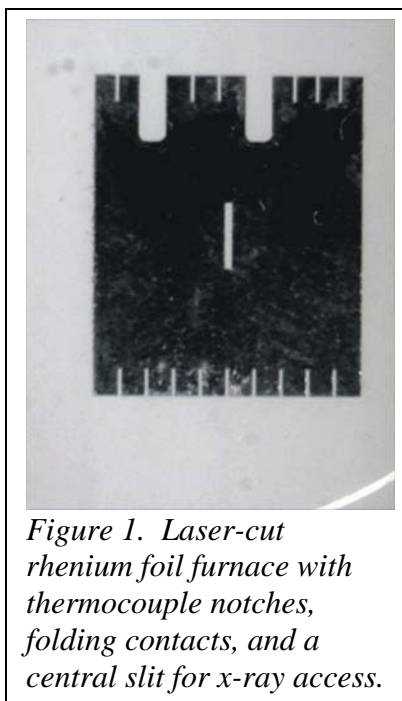


Figure 1. Laser-cut rhenium foil furnace with thermocouple notches, folding contacts, and a central slit for x-ray access.

By engaging in these activities, the COMPRES Multi-Anvil Cell Development Project has created a new forum for discussion and comparison of experiments in the multi-anvil community. Because they are available in their complete form to any interested laboratory, the COMPRES cell assemblies represent the first time that identical assemblies and materials have been used jointly by many different laboratories, aiding interlaboratory comparison. This project has also aided rapid transfer of multi-anvil technology throughout the community and made possible rapid startups for new laboratories. Community feedback and

discussion has led to new ideas and improvements that have been incorporated into the assembly designs. The overall result is a set of assemblies that are easy to learn and use, are well-characterized, have high success rates, and have a broad user base. The assemblies cover a wide range of P-T capabilities (Figure 4) with large volumes.

The building of a community has been promoted by the efforts of the PI's to communicate with many researchers on an individual basis about the techniques and the transfer of the technology, by training visitors at the ASU laboratory, by posters and talks at the COMPRES meetings (5, 6) and at AGU meetings (7), and by input from the COMPRES Facilities Committee and COMPRES Central. The beam line assembly technology has been transferred through contacts with Yanbin Wang at GSECARS. On March 1-3, 2005, a COMPRES-sponsored Workshop on Multi-Anvil Techniques was held at APS with 25 participants. The workshop demonstrated multi-anvil techniques, the new series of cell assemblies, and the use of *in-situ* diffraction and radiographic techniques. As part of this hands-on workshop three *in-situ* experiments were performed (8).



Figure 2. LaCrO₃ sleeve with thermocouple slots and x-ray window cut on the COMPRES automated lathe. The slit in the rhenium furnace from Figure 1 is also visible.

Parallel development of multi anvil cells for *in-situ* synchrotron work that resemble the conventional cells (Figures 1-3 show the 10/5 assembly for this purpose) has been pursued. A primary motivation for this is to make it easier for researchers at conventional laboratories to use synchrotron radiation. The Large-Volume Press (LVP) facility at the Advanced Photon Source allows multi-anvil experimentalists to have direct access to *in-situ* experiments because the tooling at that press can accept the standard multi-anvil second stage of carbide cubes. The four *in-situ* cells from this project (Table 2) are designed to take advantage of this because they are based on the familiar designs from the four conventional cells with the simple addition of x-ray windows. New beam line users can focus on

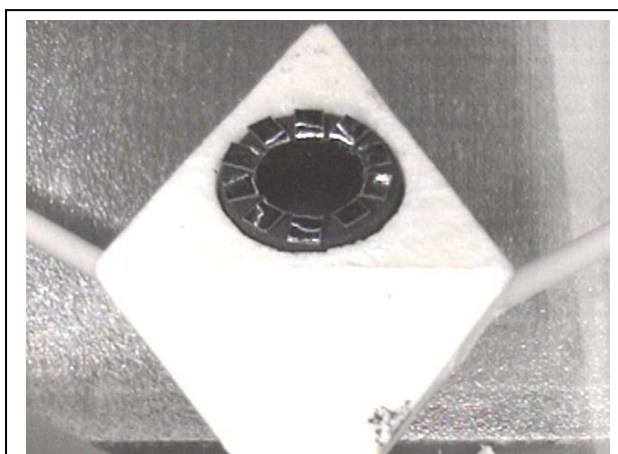
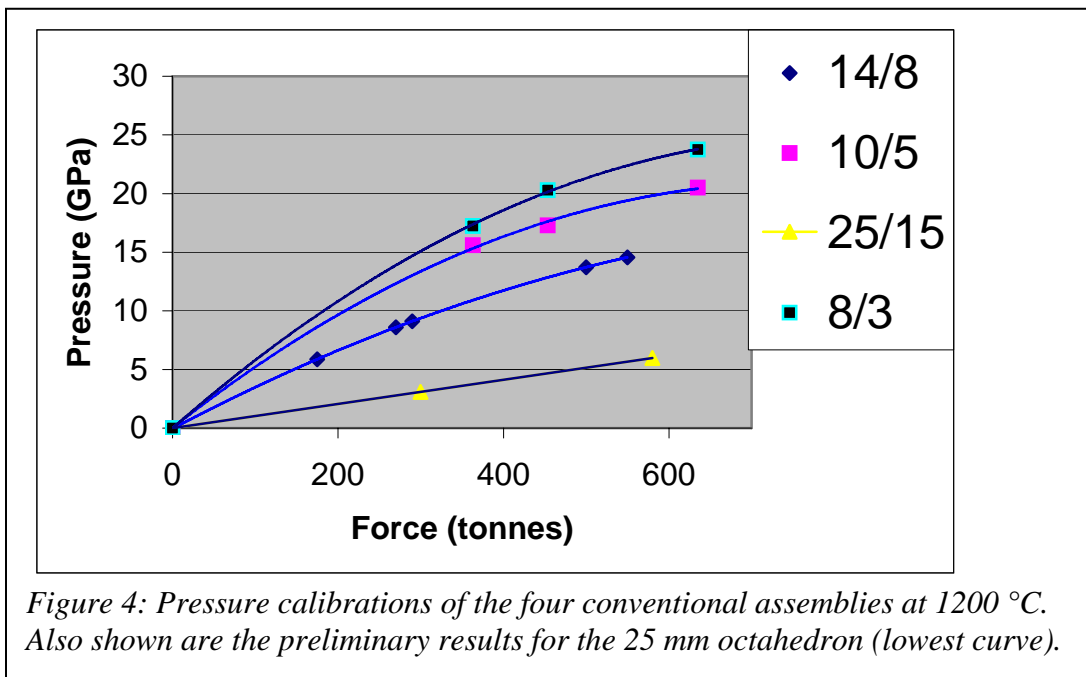


Figure 3. 10/5 in-situ assembly ready to load. The pencil smudge (lower right edge of octahedron) marks the x-ray path.

learning the *in-situ* x-ray techniques rather than worrying about an entirely new line of pressure cells.

Certain developments, in particular the porous mullite pressure media, have been beneficial to the DIA and D-DIA programs for *in-situ* diffraction and deformation experiments using cubic pressure media. The porous mullite ceramic from this project has been heavily developed as a replacement for boron epoxy by the Stony Brook group. Boron epoxy, though it is a highly effective thermal insulator and has low x-ray absorption, has recently been found to introduce H₂O to the sample and assembly, which may cause water weakening in deformation experiments, and simultaneously limits the temperatures that graphite furnaces can reach. Mullite has good thermal insulation capabilities and is reasonably x-ray translucent, but does not introduce H₂O. This has allowed more reliable dry or controlled H₂O deformation experiments, and has raised temperature capabilities in the DIA by several hundreds of degrees Celsius (9), a major advancement in capabilities.



Proposed New Phase of the Project

In 2005-2006, the Multi-Anvil Cell Development Project entered a new phase at the request of the COMPRES Facilities Committee, in which the eight standard assemblies (four conventional and four *in-situ*) are being supplied to the community for the cost of materials and supplies plus a modest service charge (25%), rather than *gratis* as they were during the early stages of testing. The Multi-Anvil Cell Development project and Arizona State University continue to provide the personnel (machinist) and infrastructure (lathe and machine shop) to produce the assemblies. The tasks of overseeing the purchasing, quality

control, and testing of cells, as well as interpreting the feedback from other laboratories, is performed by K. Leinenweber. The recharge system for materials has freed up COMPRES resources for new designing and testing, for sending out trial batches of the standard assemblies for new users, and for distributing new, untested parts and designs for trials, calibrations, and community input. The details of purchasing, making and shipping the standard assemblies have been developed and several laboratories have been buying and using the assemblies since October, 2005. The assemblies have been used for such projects as *in-situ* determination of equation of state of metal-metal oxide pairs for oxygen fugacity studies (10), the kinetics of the olivine to ringwoodite transition in the presence of H₂O (11), and the phase boundary determination of the clinopyroxene to ilmenite transition in ZnSiO₃ (12).

Based on experience gained during this initial period, we have established that the materials and supplies budget for the eight standard assemblies can be self-sustaining by this method of providing fabricated assemblies to and sharing design development with the community. The benefits to the community are that money is saved in the procurement of the assembly materials because the shared project can buy them in larger quantities, that individual laboratories save time and effort previously spent on assembly manufacture, and that the materials are carefully vetted through the COMPRES project. The savings in money, time and effort are compelling for many groups who use the COMPRES cells.

This project significantly benefits the high-pressure community and so we seek continuation as part of the COMPRES renewal. We will continue to make the cells readily and quickly available to the community, to extend the performance documentation of these cells and to develop additional new designs through further testing and feedback from the community. We will also continue developing the manufacturing techniques, reducing the costs and increasing the supply of the most time-consuming parts which are the limiting factor in production capacity. Because the feedback from the community and new data on cell assembly performance (thermal gradients, *in-situ* results, etc) will result in design improvements, we have the capacity to make new developments in order to respond to this feedback.

The COMPRES-supported machinist is a key element of this project. The machinist set up the COMPRES automated lathe and developed all the techniques and program codes for machining complex parts out of zirconia, lanthanum chromite and other ceramics, as well as metals. He also developed programs, tooling, and fixtures to fit on an automated milling machine (which is housed in the ASU Machine Shop and does not belong to COMPRES) to machine the pyrophyllite gaskets (12 per cell) and to cut all the thermocouple slots in the various sizes of octahedra, which requires three-dimensional programming of the thermocouple pathway. He also performs all the storekeeping, packing and shipping for the project.

One month of salary per year for the lead P.I., Kurt Leinenweber, is requested. The actual commitment of time that has been made and will continue to be made for this project is significantly more than one month per year. The purpose of adding this salary is to begin to address a new directive from the president of Arizona State University, Michael Crow, that

Research Specialists on State salary lines (such as Kurt Leinenweber) contribute to their salaries.

We request a much-reduced budget (\$4K per year) for materials and supplies, which will be used for purchasing and testing experimental materials such as new ceramics that are not covered by the sales of cell assemblies. We also ask for \$2K per year in travel which will be used for beam line experiments pertaining to the cell assemblies, and for national meetings in which the results are presented.

We also request a one-time capital budget of \$9 K in year 1 for the purchase of an enclosed mini-mill for the production of pyrophyllite gasket pieces and the cutting of thermocouple slots in octahedra. The current mill is the property of the machine shop (not COMPRES) and is used for many other milling jobs besides the gaskets, which has become a scheduling issue. For these reasons we are requesting the dedicated mini-mill to which we will attach a customized hazardous dust removal system.

Through these efforts we will maintain the COMPRES multi-anvil cell development effort as firmly established part of the multi-anvil community, which will contribute to the COMPRES presence both in university laboratories and at the synchrotron facilities.

Targets for the Standard Assemblies

The eight standard assemblies will be joined by several more in the proposal period; the 18/11 and 25/15 assemblies, and a new low thermal gradient assembly. Even the well-established standard assemblies will benefit from further development, although great care must be taken not to reduce their capabilities or to drift away from the pressure calibration. The main targets are to use the same materials but find ways to fabricate the pieces that are less costly and which reduce the repetitive tasks performed by the COMPRES machinist. For example, the metal rings (moly or TZM cylinders with thermocouple notches) for the four 14/8 assemblies are all currently made by the COMPRES machinist; however, there are outside shops that can make these at a competitive price if quantities of 1000 or more are ordered. We are exploring ordering these in the first and second year of the proposed period, when the recharge budget can handle the large orders. We are also looking at extruded or pressed zirconia for the 14/8 assemblies, which will save a great deal of money on each assembly relative to the current method of buying zirconia blocks, slicing and turning them (the zirconia is currently the single most viable target for significantly reducing the prices of the standard assemblies). This requires some materials budget for testing and for ordering large batches (again in the neighborhood of 1000 pieces per order) and may require calibration checks or even re-calibration. In the long run these improvements will be advantageous. Standard pyrophyllite gaskets will be outsourced to a laser-cutting company. If possible, we will also obtain new molds for the standard octahedra that have the thermocouple notches already molded in. The outsourcing of these and other standard pieces will allow us to increase the overall supply of assemblies, and will free up time for important new developments.

Targeted Goals for New Development

Here we present plans for new developments. Many of these plans are based on input from the high-pressure community.

New 6-8 Assemblies

A large-volume assembly, the 25/15 assembly, needs to be designed, tested, and calibrated, from already existing molded octahedra made by the COMPRES project. We plan to design the assembly around the 5 mm tubing normally used with piston-cylinder experiments, which will provide a very large volume capability up to 8 GPa. There has heretofore been no 18/11 assembly, although the octahedra for this size also already exist. This is the last size slated for development; it is needed to allow larger sample volumes in the 8-11 GPa range.

A near-zero thermal gradient 14/8 box heater will be developed. The basic design has been modeled with the thermal gradient package of Hernlund *et al.* (4). The gradient is less than 5 °C in a region 25 mm³ in size. A gradient this low is expected to change the phase stability greatly in experiments with mobile components, where diffusion through thermal gradients is commonly a problem. It will also allow the synthesis of single-phase materials, such as hydrous phases, that were not before possible because of sample layering. This development will be pursued in collaboration with G. Gwanmesia at Delaware State University.

DIA Assemblies

We have been helping with the development of some DIA technologies, in particular the new mullite cubes for pressure media. We will increase our involvement in the design and production of cubic DIA assemblies. This will enable us to become further involved in *in-situ* designs and technologies beyond the 6-8 octahedral designs, and also to include new techniques such as deformation, for which the primary recent achievements have been in the deformation DIA, or D-DIA. We will coordinate with groups from SUNY Stony Brook, Lawrence Livermore National Labs, Yale University and UC Riverside, where the DIA development has been pursued strongly, identify the best cube sizes and furnace designs, and test them and make them available for testing and use in a fashion similar to what we have accomplished with the octahedral designs.

Calibrations

In-situ load-pressure calibrations of the conventional assemblies, such as have resulted in very detailed calibration curves for the 10/5 and 8/3 assemblies and curves of lesser but still very enlightening detail for the two 14/8 conventional assemblies, will continue at the beam lines. The calibration and testing of the beam line assemblies will occur as a normal consequence of their use at the beam line, but the conventional assemblies require dedicated experiments for their calibration because they would not normally be run on a beam line.

The travel and materials and supplies budget for these experiments are provided for in the COMPRES budget and the data will be published for reference by the community.

Encapsulation

The COMPRES cells are currently supplied “empty,” without noble metal capsules or samples. The original intention was to leave the encapsulation completely up to the user, because it is so sample-specific. However, many of the assembly end users have strongly encouraged us to address as a community effort the problems of encapsulation. In response to this interest, and in combination with testing by end-users, we have recently added several choices of ceramic capsules to the assemblies (BN, graphite, MgO). A goal of the project is now to work on the problem of noble metal capsules. Engelhard has closed its tube manufacturing plant in New Jersey, which has caused extreme difficulties in economically obtaining noble metal tubing, especially the alloys such as gold-palladium, but also the pure metals such as gold and platinum. In effect, some of the savings for researchers of using COMPRES project assemblies are going to be lost in the price increase of noble metals. The COMPRES Multi-Anvil project will work with the community and with companies on joint purchases and on locating alternative suppliers of noble metal tubing and foil.

Accessories

We will increase the supply of special jigs, dies, and nesting plates for assembly construction. These have not been an official part of the COMPRES supplies and have simply been sent to laboratories for free when available, but in many cases the jigs not only significantly reduce the labor involved in assembly-building, but they can contribute to the success rates of runs. Also, the demand has grown rapidly, especially since they are featured in the instruction manuals for the assemblies. For these reasons, we plan to officially introduce them as COMPRES supplies.

We will commission a mold for injection-molding the mullite octahedra for the 14/8 “Bay-Tech” in-situ assembly described in reference (2) (the octahedra are currently machined one at a time). We are also currently pursuing molds that have thermocouple grooves included. These molds, once made, can produce in the range of 10,000 reproducible pieces before they need to be replaced.

Table 1

- A. A list of the laboratories using COMPRES cells:
- Argonne National Laboratories
 - Arizona State University
 - Delaware State University
 - Geophysical Laboratory
 - Lawrence Livermore National Laboratories
 - NASA Johnson Space Center
 - Stony Brook University
 - University of Arizona
 - University of California at Davis
 - University of California at Riverside
 - University of New Mexico
- B. A list of additional laboratories testing COMPRES materials:
- American Museum of Natural History
 - Brookhaven National Laboratories
 - California Institute of Technology
 - Georgia State University
 - University of Hawaii
 - University of Minnesota
- C. Foreign affiliates:
- Daresbury Laboratory
 - University College London
 - University of Western Ontario

Table 2. A summary of the standard COMPRES multi-anvil assemblies.

| Assembly name | Peak pressure | Proven temperature | Design |
|-------------------------|---------------|--------------------|--|
| 8/3 | 25 GPa | 2319 °C | Rhenium furnace |
| 10/5 | 20 GPa | 2000 °C | Rhenium furnace |
| 14/8 “G2” | 13 GPa | 1200 °C | Graphite box furnace |
| 14/8 “Bay-Tech” | 15 GPa | 1400 °C | Graphite/LaCrO ₃ step furnace |
| 8/3 in-situ | 25 GPa | 2000 °C | Slitted rhenium furnace |
| 10/5 in-situ | 20 GPa | 2000 °C | Slitted rhenium furnace |
| 14/8 “G2” in-situ | 13 GPa | 1200 °C | Graphite box furnace, forsterite sleeve |
| 14/8 “Bay-Tech” in-situ | 15 GPa | 1500 °C | Graphite step furnace, MgO equatorial window, mullite octahedron |

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B.2.c Brillouin Spectroscopy at Advanced Photon Source

**[J. Bass-University of Illinois at Urbana-Champaign, G. Shen-
University of Chicago]**

Current funding for period 2002-2007: \$429K

During past year, a great amount of progress has been made on the installation and commissioning of a Brillouin spectrometer has been installed at the APS on the GSECARS sector 13-BM-D. In short, a working system is now in place and the first data have been collected. Photographs of the new system and some representative data collected with it are attached. Initial testing and our first commissioning experiments of the Brillouin spectrometer was carried out during two beam time allocations during the last year, and these tests were successful. The first data collected are presently being analyzed and will be written up for publication. With the installation and testing of the Brillouin system in the synchrotron hutch, the primary goals of this project are essentially complete.

As described in the original COMPRES proposal, one of the main purposes of this project was to simultaneously determine the density and sound velocities in a variety of materials that could be useful as calibration standards in high P-T experiments. By simultaneous velocity and density measurements, one obtains the pressure on a sample absolutely. Thus, one of our primary goals is to determine a number of primary pressure standards for use in high-pressure-temperature research. Our long range goals are to perform such velocity-density measurements under a wide range of P-T conditions. The high temperatures can be provided by resistance and/or laser heating techniques.

Our initial experiments will included the following:

- NaCl in B1 phase to 30 GPa in a diamond anvil cell.
- MgO to 30 GPa with the DAC.
- Aggregate acoustic velocities, elastic moduli and equation of state of polycrystalline NaCl in B2 phase to 70 GPa.
- MgO and NaCl at high pressure and high temperatures (by resistance heating) up to 400° C.

In all absolute pressure scale experiments gold + ruby (+ platinum + powdered NaCl) were added to experimental charges to cross calibrate these pressure standards against absolute equations of state of NaCl and MgO.

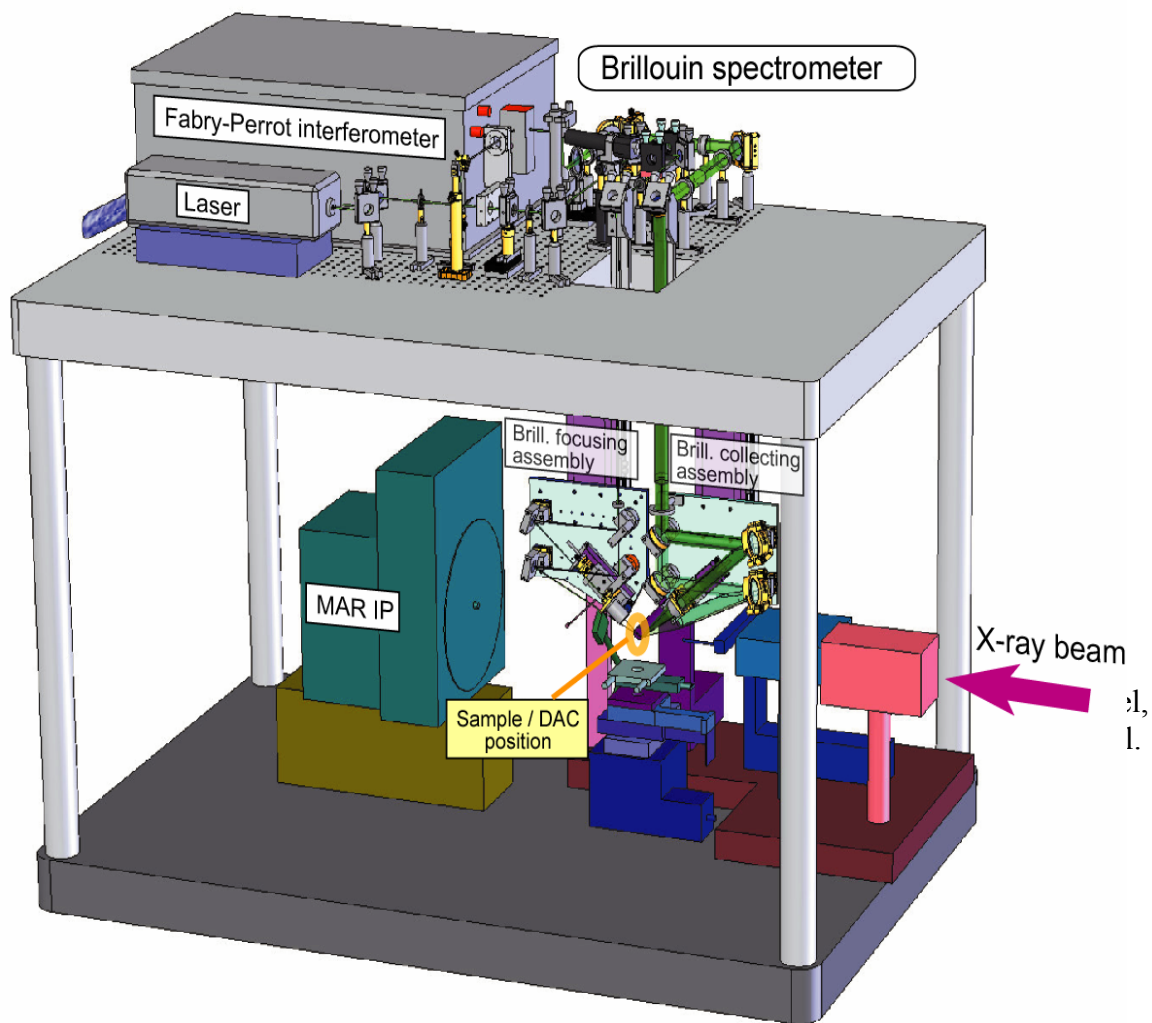
A second goal of this proposal was to provide a centralized facility for Brillouin scattering studies. We have provided a state-of-the-art facility that is open to the entire scientific community and not widely available elsewhere (except in a few specialized labs). We are already working with other groups that have expressed interest in using the system (e.g., U Nevada Las Vegas, Arizona State).

To summarize the activities on this project in all years, the first 3 years were spent on design of the system, ordering equipment, building prototypes at Illinois, testing the main components, experimenting with various optical configurations that might be used at APS, and working to make the optical set up more compact. The APS Fabry Perot and control electronics differed substantially from the system we used in Urbana (which was purchased 20 years ago), and we spent time gaining experience with the new components. In the last 2 years the system was installed at sector 13 and initial testing/commissioning of the system was performed on samples of MgO and NaCl in the diamond cell.

This project required the participation of many people. Most important was Stanislav V Sinogeikin (formerly UIUC, now at HPCAT), who did much of the detailed design and handled the installation of the system. Others who assisted in various aspects of the project included Dmitry Lakshtanov (grad student, UIUC), Guoyin Shen (former co-PI, GSECARS; now at HPCAT), Vitali Prakapenka (GSECARS), Carmen Sanchez-Valle (post doc, UIUC), Jean-Philippe Perrillat (post doc, UIUC), and Jingyun Wang (grad student UIUC).

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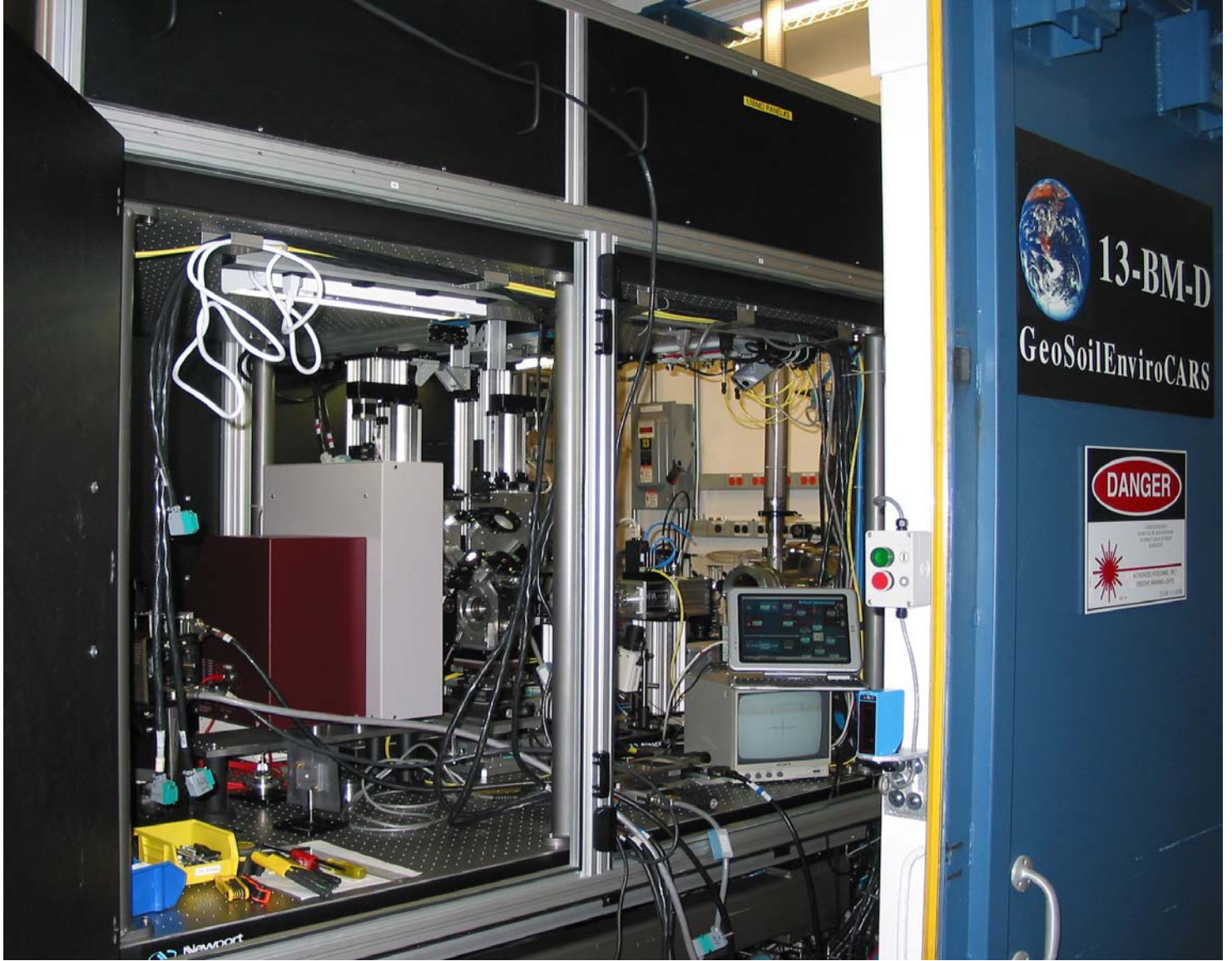
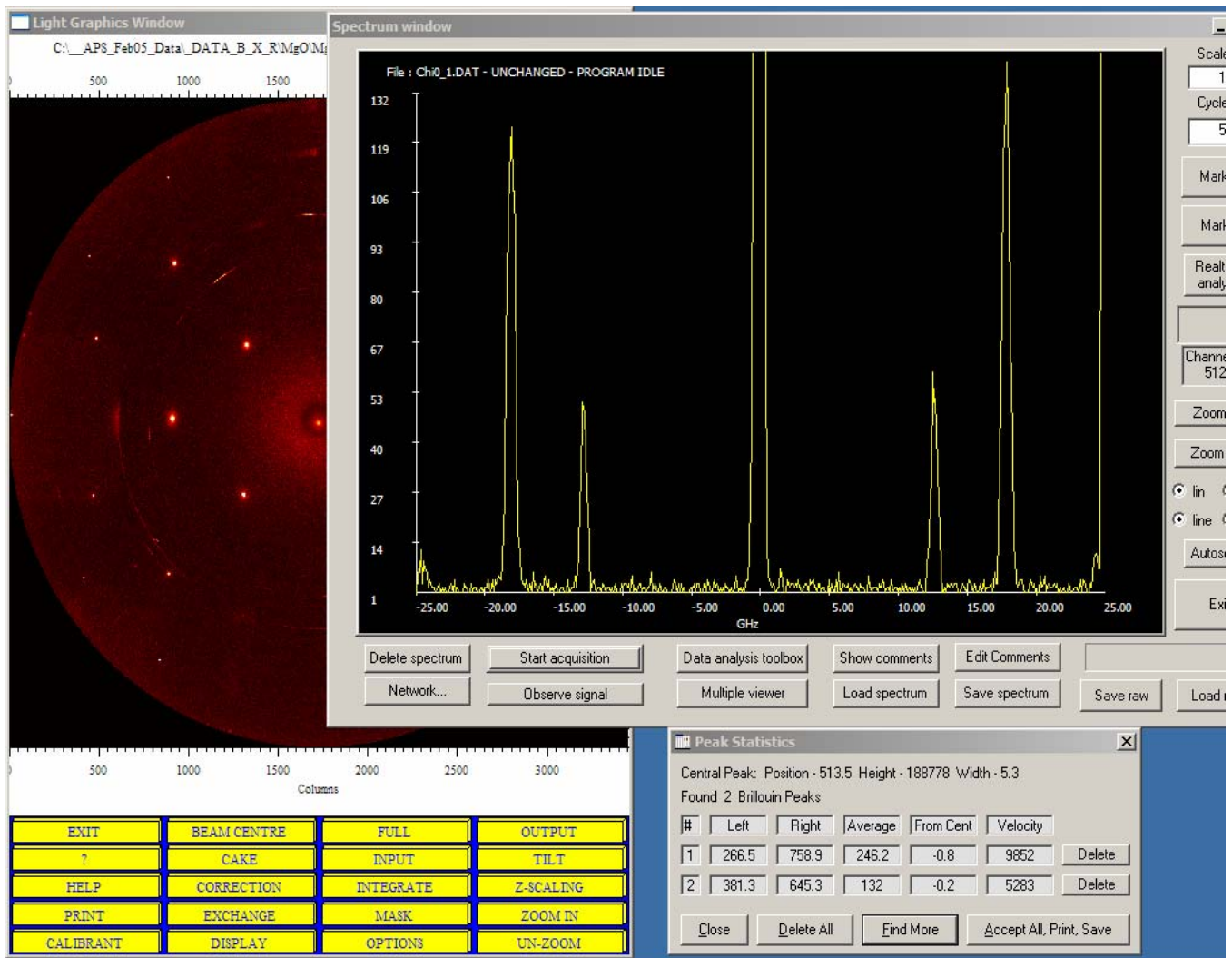


Figure 2: Side view of the Brillouin system at the APS. The upper optics are in the black enclosure at the top of the photo, to comply with APS laser safety regulations.

Single crystal X-ray diffraction and Brillouin spectra of MgO at ambient pressure



26 July 2006

B.2.d Nuclear Resonant Inelastic Scattering at High Pressure & Temperature

[W. Sturhahn-Argonne National Laboratory, J. Bass- University of Illinois at Urbana-Champaign, G. Shen-University of Chicago]

Current funding for period 2004-2007: \$185K

Progress Report for 2004-2006

Report Summary

We report here on the activities to date of Years 1&2 of a 3-year infrastructure development project on Nuclear Resonant Scattering (NRS) at high P and T. We include here a description of activities to date and planned activities for the third year.

Nuclear resonant scattering techniques are relatively new applications of synchrotron radiation for determining the properties of condensed matter. Our infrastructure development project 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. We are pursuing the development of two related techniques: 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.

In the first two years of our infrastructure development project, we focused on the hiring of a full-time postdoctoral researcher to support the goals laid out in the original proposal text and on the improvement of the experimental capabilities of the NRS beam line (sector 3-ID) of the Advanced Photon Source (APS) to enhance its performance in high-pressure research and to make it more accessible to the COMPRES community. Outreach activities, e.g., an upcoming workshop on NRS and various presentations at meetings and conferences, have broadly disseminated information on applications of NRS to understand Earth materials. In particular, we accomplished the following tasks:

- Hiring of a full-time postdoctoral researcher;
- Development of refined high-pressure equipment for NRS;
- Improvement of the laser-heating system at sector 3-ID of the APS;
- Organization of the workshop “Nuclear Resonant Scattering on Earth Materials using Synchrotron Radiation”, February 12-13, 2005 at the APS.
- Organization of the workshop “Evaluation of Synchrotron Mössbauer

Spectroscopy Data using the CONUSS Software”, October 29-30, 2005 at the APS.

- Procurement of a new focusing mirror for increased x-ray intensity;
- Installation of a DAC loading facility for users of the NRS beam line;
- Procurement of image plate device for NRS with x-ray diffraction;
- Generation of numerous new proposals for sector 3-ID of the APS by COMPRES members.

Some of the individual items are described in more detail below.

Improvement of the Laser-heating System

A laser-heating system was purchased earlier and integrated at sector 3-ID of the APS. The logical continuation of this effort now consists of the full integration of this system with NRIXS experiments using the DAC. We added several automated controls, e.g., the heating power distribution over the two sides of the DAC, to obtain a more user-friendly system. The integrated laser-heating system was tested with metallic iron samples. Stable conditions were achieved over 12h and more depending mostly on sample temperature and pressure. We accommodated such situations that require a very well-defined x-ray focus to minimize background by the addition of clean-up slits into the x-ray beam path. This instrument upgrade facilitates the removal of scattering contributions that originate from the tails in the intensity distribution of the x-ray focus very effectively.

Workshop Organization

We organized the first workshop on “Nuclear Resonant Scattering on Earth Materials using Synchrotron Radiation” on February 12-13, 2005 at the APS. Participants will learn about the capabilities and the theoretical background of NRS methods, visit sector 3 at the APS where NRS is performed, and obtain some hands-on experience. The goals were formulated as follows: provide a basic introduction of NRS to the Earth science community; define the state-of-the-art of NRS especially at high pressure; discuss the applications to important geophysical problems; develop productive collaborations; address common experimental issues confronting users. We were also organizing a workshop on “Evaluation of Synchrotron Mössbauer Spectroscopy Data using the CONUSS Software” on October 29-30, 2005 at the APS. Participants learned about the strategies to successfully evaluate and interpret SMS data collected at sector 3-ID or sector 16-ID. The goals were: provide a basic introduction of SMS to the Earth science community; introduce the CONUSS software for SMS data evaluation; provide “hands on” training in the use of the CONUSS software; address common experimental issues confronting users. Both workshops provided ideal formats to collectively address possible solutions to experimental problems and will help to build a viable COMPRES user base for this facility. Details on the workshop agendas can be obtained at the following websites:

<http://www.nrs2005.aps.anl.gov> and

http://www.aps.anl.gov/News/Conferences/2005/Mossbauer_Data_Workshop.

Procurement of a New Focusing Mirror

Experiments with small samples require a small x-ray beam. In particular, high-pressure studies with sample sizes of 50 micron or less benefit tremendously by focusing of the x rays. At sector 3 Kirckpatrick-Baez mirrors are implemented for this task. The spatial acceptance of the system is determined by mirror size, incident angle of the x rays, and energy of the x rays. At 14.4 keV the vertical and horizontal acceptance is about 300 micron and 700 micron, respectively, but the size of the SR beam at the mirror location is about 350 micron vertical and 2 mm horizontal. Therefore, an improvement of photon flux incident on a pressurized sample mounted inside a diamond anvil cell can be achieved by a longer horizontally focusing mirror. With funds of Sturhahn's group at the APS amounting to about \$200k, we procured a horizontally focusing mirror of 60 cm length. This mirror has a piezo-electric bending mechanism build into the mirror itself for optimal shape adjustment and will capture most of the x-ray beam at 14.4 keV. We expect an increase the x-ray flux on the sample by a factor of two to three. This increase in flux directly translates to enhanced capabilities, either by reduction of data collection times (crucial for experiments at very high temperatures) or by increased statistical accuracy. Also the persistent oversubscription of sector 3-ID can be more effectively addressed.

Installation of a DAC Loading Facility

A facility for loading diamond cells is indispensable if sector 3-ID is to be used regularly for high-P studies by the entire COMPRES community. This is especially true for SMS and NRIXS experiments since they require special types of high-pressure cells and loading techniques that are quite different from other x-ray methods, e.g., the development and implementation of Be gaskets for NRIXS. We set up a DAC loading facility in one of the laboratories of Sturhahn's group near sector 3. The facility is now available to COMPRES members working at the sector 3-ID beam line.

Procurement of Image Plate Device

With \$120k in funds from Sturhahn's Inelastic X-ray and Nuclear Resonant Scattering (IXN) group at the APS we procured an image plate device. This is the key component for the planned enhancement of the NRS beam line by adding an x-ray diffraction capability. Once operational, the added diffraction capability can provide us with structural confirmation as well as with an equation-of-state during NRIXS data collection. The possibility to perform NRS (for sound velocities and elastic parameters) and x-ray diffraction (for density, elastic parameters, and structure confirmation) simultaneously under high pressure and temperature conditions will be groundbreaking.

Generation of New Proposals

In large part as a result of the first workshop on "Nuclear Resonant Scattering on Earth Materials using Synchrotron Radiation" that took place on February 12-13, 2005 at the APS, we were able to catalyze nine new beam time proposals for NRS studies at sector 3-ID from COMPRES member institutions for the October-November-December operations cycle of the APS. Experiments based on proposals over the last year have produced novel results as shown in the separate list of publications. The following scientists of COMPRES member institutions have lead proposals that

received beam time at 3-ID between May 2004 and today: J.D.Bass (UIUC), B.Fultz (CalTech), A.Goncharov (LLNL&GL), J.M.Jackson (UIUC&GL), J.Li (UIUC), J.-F.Lin (GL&LLNL), H.-K.Mao (GL&UofC), W.Mao (UofC&LANL), G.Shen (UofC&GL), V.V.Struzkhin (GL), O.Tschauner (UNLV), C.-S.Yoo (LLNL). Lead scientists of active proposals to be scheduled are: J.Crowhurst (LLNL), H.Cynn (LLNL), H.Giefers (UNLV), J.-F.Lin (LLNL), C.-S.Yoo (LLNL).

With improvements of the x-ray intensity, e.g., by the mentioned mirror upgrade project, we are addressing the present oversubscription of sector 3-ID. The HRX group also plans to effectively increase the total amount of NRS beam time in sector 3-ID by 20 % with the beginning of operations at the new IXS beamline 30-ID.

Planned Activities

In the last year of our infrastructure development project, we will continue the outreach effort to the COMPRES community by assisting interested groups in design, preparation, execution, and evaluation of NRS experiments. We will organize a second tutorial workshop introducing NRS and its applications for studying planetary interiors. For those who wish to perform experiments in the near term, we will assist the COMPRES community in the preparation of proposals for beam time. On the instrumental side, we will proceed with the installation of the new focusing mirror system for increased x-ray intensity and the integration of the capability of x-ray diffraction with NRS experiments. The added diffraction capability will provide us with structural confirmation as well as with an equation-of-state during NRXS data collection. The possibility to perform NRS (for sound velocities and elastic parameters) and x-ray diffraction (for density, elastic parameters, and structure confirmation) simultaneously under high pressure and temperature conditions will be groundbreaking. We expect that more proposals for NRS experiments on sector 3-ID will likely result from the workshop, and that we will work with the PIs to develop effective proposals that will be very competitive for beam time. In effect, COMPRES will continue to have its own experts to help write proposals, consult on technical aspects of experiment design, and to help run experiments.

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Illustrations

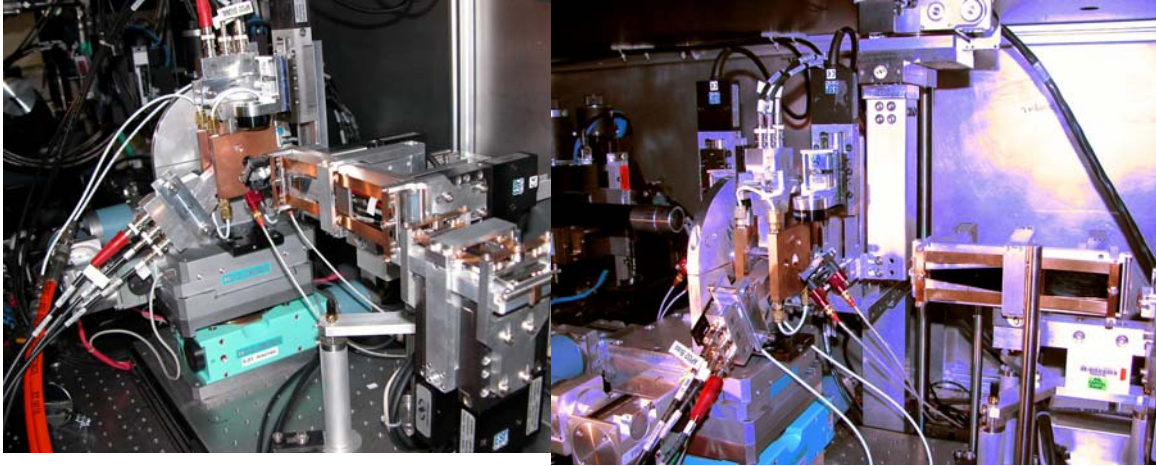


Figure 1a: DAC environment for NRS experiments with laser heating before the addition of clean-up slits. Figure 1b: DAC environment for NRS experiments with laser heating after the addition of clean-up slits. The distance between x-ray focusing mirrors (right) and DAC (middle) was increased, and a clean-up slit assembly was inserted. On the left, a microscope for DAC alignment is visible.



Figure 2: New focusing mirror for high pressure experiments. The mirror is 10 cm long and 1 cm wide. The ray intensity is increased by a factor of 10. Courtesy of the Advanced Photon Source.

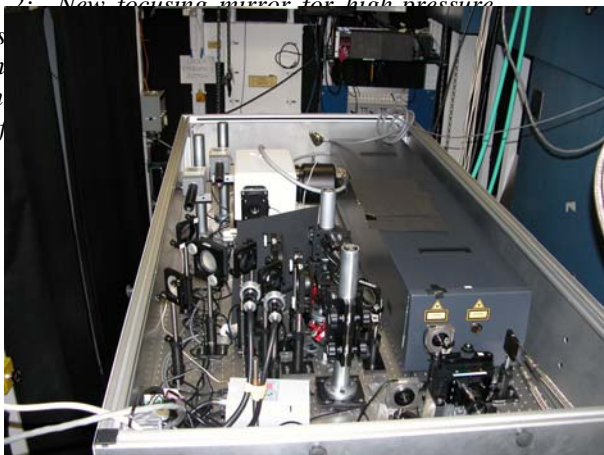


Figure 3: Laser-heating system for NRIXS studies at beam line 3-ID of the Advanced Photon Source.

B.2.e Development of the CO₂ Laser-Heated diamond Anvil Cell

[T. Duffy-Princeton University, G. Shen-Carnegie Institution of Washington, D. Heinz-University of Chicago]

Funding for period 2002-2007: \$493K

CO₂ laser heating system development

A goal of this project is to develop an on-line CO₂ laser heating system in the 13-ID-D station of the GSECARS sector at the Advanced Photon Source. A bench-top system has been designed, constructed, and is currently undergoing testing (Fig. 1). The system is built around a Synrad f201 200-W CO₂ laser with linear polarization that is operated in CW mode. Laser power is controlled through a rotatable Brewster window device. The bench top set-up also includes a laser-alignment system, attenuator, sample stage, focusing and imaging optics, and APS-approved safety shutter system. The system has been used to heat diamond cell samples to 40 GPa. On-going upgrades include incorporation of IR detectors for sample imaging, improvements in optical layout, and motorizing components. The current timeline is as follows: bench-top testing will be completed in the summer 2006, and the system will be moved to ID-D in Fall 2006. Commissioning will be carried out in the first part of 2007, and the system will be open to users by the end of 2007.

Technical development of laser-heated diamond cell

We have also carried out experiments and simulations designed to improve fundamental understanding and capability of the laser-heated diamond cell. Finite element simulations of the temperature field in the laser-heated diamond anvil cell have been used to evaluate the parameters that control axial and radial temperature gradients (Fig. 2). We have performed simulations for a typical experimental geometry consisting of an optically thin sample separated from the diamond anvils by an insulating medium of varying thickness and heated by the Gaussian mode from an infrared laser (Kiefer and Duffy, 2005). More recently this effort has been expanded to consider a range of realistic sample geometries that are used in practice including single- and double-sided heating designs for both metals and insulators (Kiefer and Duffy, in prep.). As examples of other projects, we have carried out experiments to quantitatively examine thermal pressure in the laser-heated diamond cell (Kubo et al., 04), demonstrated the feasibility of using x-ray fluorescent crystals as x-ray-beam markers (Shieh et al., 2005), and carried out inter-calibrations of pressure standards using alkali halide crystals.

Personnel and outreach activities

A workshop on the laser-heated diamond anvil cell was held on May 19-20, 2004 at the Advanced Photon Source. A total of 41 persons attended the workshop including participants from Japan, Europe, and the US. The format of the workshop included presentations from leading researchers in the field, as well as a series of open-ended discussions regarding future needs for the high-pressure laser heating community. A broad cross-section of the high-pressure laser-heating and synchrotron communities was represented at the meeting.

http://www.compres.stonybrook.edu/Workshops/March_04_Laser_Workshop/laser_workshop_presentations2.htm

Dr. Andy Campbell (Chicago) participated in this project at 50% level from July 2004-2005 before he left to join the faculty at Maryland. Dr. Alexei Kuznetsov (GSECARS) was then hired and began in April, 2006. Partial support was also provided to Dr. Boris Kiefer (Princeton and New Mexico State) for work on finite element simulations of the laser heated diamond cell and Atsushi Kubo (Princeton) and Sean Shieh (Princeton, now Asst. Prof., Western Ontario) for work on technical developments in the diamond anvil cell.

Publications:

Duffy, T. S., Synchrotron facilities and the study of deep planetary interiors, *Reports of Progress in Physics*, 68, 1811-1859, 2005.

Kiefer, B. and T. S. Duffy, Finite element simulations of the laser-heated diamond anvil cell, *Journal of Applied Physics*, 97, 114902, 2005.

Kubo, A., T.S. Duffy, S.R. Shieh, G. Shen, V.B. Prakapenka, Thermal pressure in the laser-heated diamond cell, Advanced Photon Source Activity Report 2003, ANL-04/16, December 2004.

Shen, G., V. B. Prakapenka, P. J. Eng, M. L. Rivers, S. R. Sutton, Facilities for high-pressure research with the diamond cell at GSECARS, *Journal of Synchrotron Radiation*, 12, 642-649, 2005.

Shieh, S. R., T. S. Duffy, and G. Shen, X-ray diffraction study of phase stability in SiO₂ at deep lower mantle conditions, *Earth and Planetary Science Letters*, 235, 273-282, 2005.

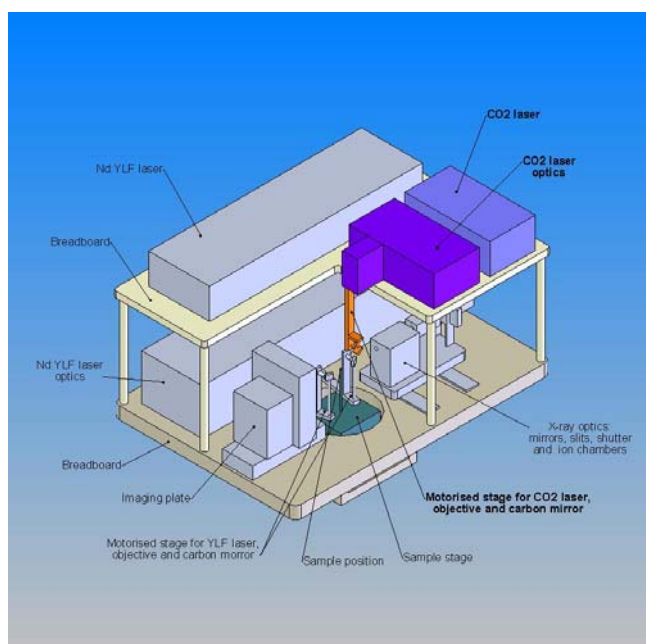


Fig.1. Experimental set-up for CO₂ laser heating in 13-ID-D station of GSECARS.

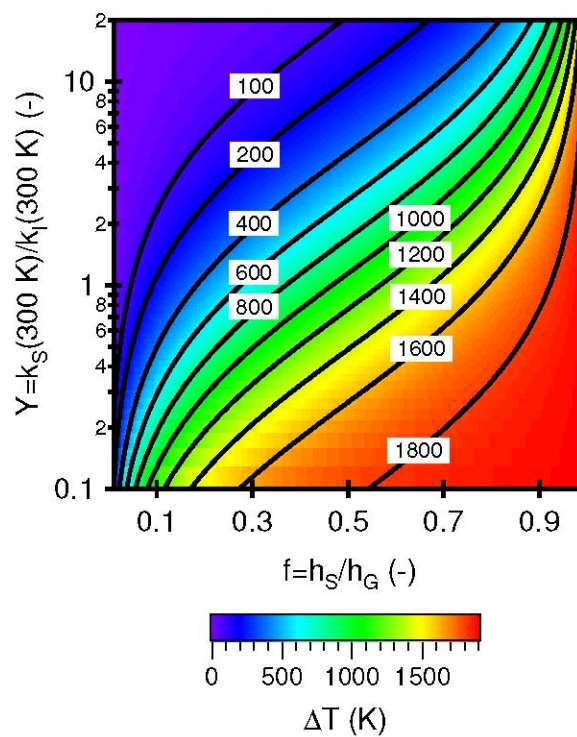


Figure 2. The axial thermal gradient in the laser-heated diamond cell depends on two major factors: the ratio of thermal conductivities of the sample and insulator and the ratio of sample thickness to anvil gap (h_s/h_G). The figure above shows contours of axial temperature decrease across the sample (for a peak temperature of 2200 K) as these two parameters are varied. For a typical thickness ratio of 0.5, a thermal conductivity contrasts of ~ 10 is required for the axial temperature drop to be less than 10% of the peak temperature. From Kiefer and Duffy (2005).

B.2.f Absolute Temperature Calibration using Johnson Noise Thermometry

[I.Getting-University of Colorado, and Y-b. Wang, M. Rivers-University of Chicago]

Funding for period 2002-2007: \$367K

This component of the COMPRES Infrastructure Development program seeks to establish accurate temperature measurement within the high pressure community based on sound metrological practice. Temperature measurement has proven very difficult in high pressure environments. Decades of consideration have failed to yield realistic calibration for thermocouples. Temperature can be measured accurately by Johnson noise thermometry in a high pressure environment, however. Johnson noise is the very small, fluctuating voltage noise which appears across any resistor at temperatures above absolute zero. For an open circuit resistor in thermal equilibrium, the relation between the mean square noise voltage across the resistor, $\langle E_R^2 \rangle$, the resistance, R , and the absolute temperature, T , is given by

$$\langle E_R^2 \rangle = 4kBR T \quad (1)$$

where k is Boltzmann's constant and B is the electrical bandwidth over which the noise voltage is observed. This random fluctuating voltage has Gaussian-distributed amplitude, a zero mean, and a white power spectrum. 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.

The Johnson noise signal in a practical noise thermometer has a typical RMS value of less than 1 μ V. To achieve the desired temperature resolution of ~ 0.1 % this signal must be resolved to about 1 nV. This is a demanding electronic challenge. The measurements must be restricted to the Johnson noise itself. Any spurious noise in the signal corrupts the measurements by reducing the sensitivity and by introducing time varying errors. These errors must be eliminated by making the thermometer circuits and cables sufficiently insensitive to the ambient electromagnetic environment. This is achieved by having sufficiently good isolation and shielding of the circuits and cables and by having an electromagnetic ambient which is sufficiently quiet.

In an effort to address this long-standing high pressure temperature measurement problem Ivan Getting and Dr. John Hall constructed a Johnson noise thermometer over a several year period. John is a world renowned metrologist and Nobel Prize recipient at JILA (<http://jilawww.colorado.edu/>), a NIST co-sponsored research institute at the University of Colorado. John provided all the critical circuit design for this project.

When COMPRES support for this project began in the fall of 2002 substantial progress had been made. The pre-amplifier, main amplifier, control relay system, and

rudimentary software had been constructed. At that time all of these system components required further refinement. These refinements were accomplished under COMPRES funding in the three following years. By the spring of 2005, the following had been achieved (Fig. 1):

- 1.) The pre-amplifier had been modified to make it stable over an appropriate range on source impedances.
- 2.) The main amplifier and analog filters were settled in their final design.
- 3.) The relay control system was thoroughly tested for parasitic emfs, spurious noise was eliminated, and complete control was achieved.
- 4.) The software was dramatically improved allowing detailed inspection of the power spectrum of the Johnson noise signal and refined presentation of the real time results.
- 5.) A dramatic reduction in extraneous electrical noise was achieved rendering the instrument significantly more stable with resolution very close to the theoretical limit.
- 6.) The relevant GSECARS hutch at APS in Argonne National Laboratory was tested for ambient electrical noise leading to the decision to migrate the instrument to that environment in anticipation of Ivan Getting's termination of participation in the project.

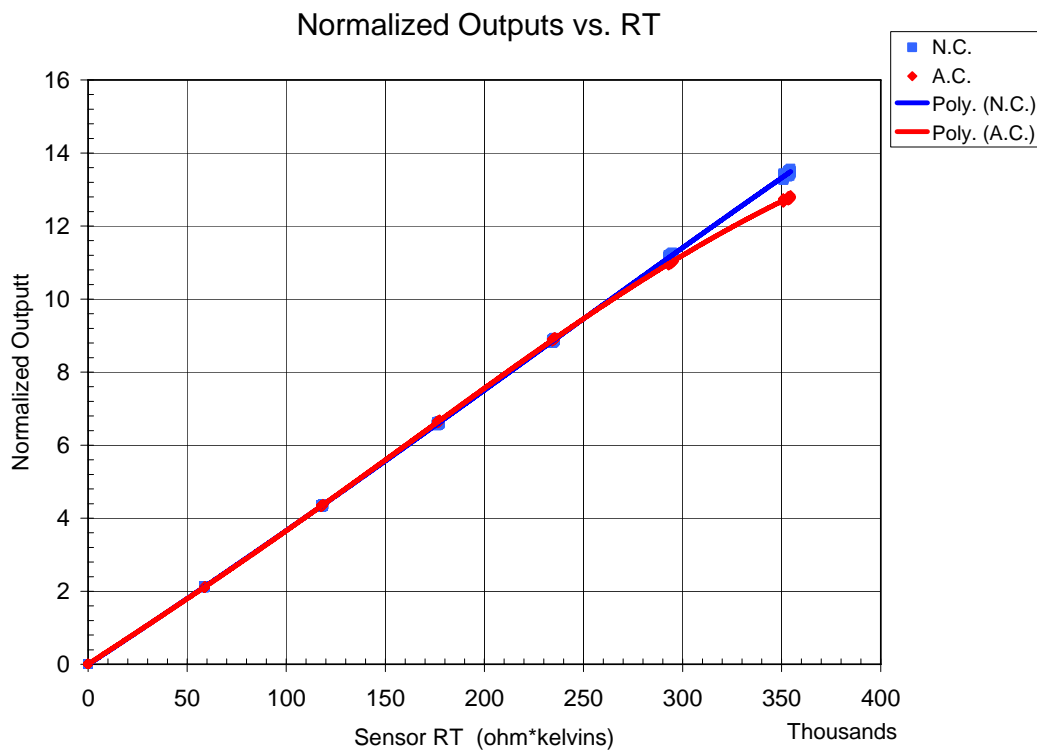


Figure 1. Johnson noise thermometer output vs. the sensor RT product, where R = sensor resistance, T = sensor temperature. In this room temperature study, the sensor resistance is changed in 200 ohm intervals simulating the RT range intended with a nearly constant 100 ohm sensor over a wide temperature range. With the intended sensor, room temperature corresponds to $RT = \sim 50$ thousand. $RT = \sim 350$ thousand corresponds to ~ 2000 K. Both numerically correlated and analogue correlated outputs are shown. The analogue correlator begins to clip significantly near full scale resulting in the artificial, but reproducible curvature shown.

In June of 2005 the first high pressure tests were made at the University of Colorado. Yanbin Wang and Norimasa Nishiyama brought the DIA apparatus to Colorado with a successful noise thermometry cell (Fig. 2). AC contamination of the noise signal from the heater current proved to be quite low in the carefully designed high pressure cell, about an order of magnitude lower than that associated with typical cell designs, posing no problems to the noise thermometer circuits.

Another trip to Colorado in January of 2006 was aimed at conducting a real JNT temperature measurement under pressure in the DIA. However, it was discovered that a DC bias on the noise signal, associated with the thermoelectric effect, saturated the JNT pre-amplifier. John Hall provided a filter design for the pre-amplifier which was implemented by Ivan Getting.

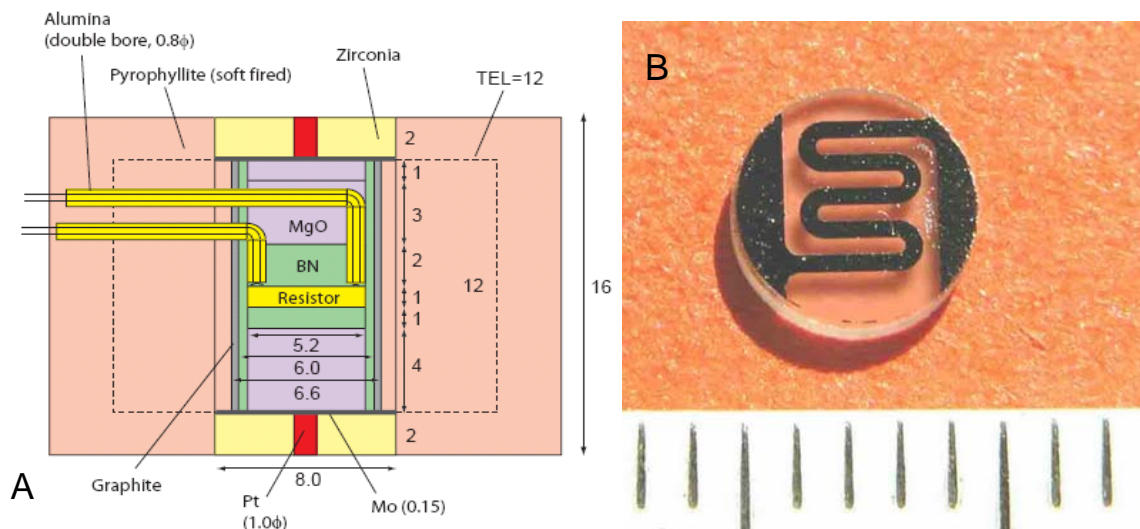


Figure 2. (A) Cell assembly for the first JNT test. All dimensions are in mm. Tests were conducted using the DIA apparatus, with 12 mm anvil truncation. (B) 5 mm diameter resistor for JNT temperature measurements. Ta and Pt layers are vapor deposited on a polished Lucalox substrate to form a mechanically robust and thermally tolerant resistor. Mechanical/electrical contact with the four thermal element leads on the pads at the ends of the serpentine filament (see A). The cell design has been successfully tested in the DIA apparatus and proven to be applicable for high pressure JNT measurements.

In March of 2006 the instrument was moved to Argonne National Lab with Yanbin Wang and Mark Rivers assuming the responsibilities of Principal Investigators. Ivan Getting and Yanbin Wang installed the noise thermometer and reestablished the very high level of performance achieved in Colorado. These tests were made while the synchrotron was running and the x-ray beam was present in the adjacent hutch. This achievement represents a great step forward for this instrument. Ambient electromagnetic noise can raise havoc with noise thermometry. The persistent efforts of the several prior years produced sufficient immunity to reproduce the best measurement in the synchrotron hutch environment.

When the noise thermometer was connected to a high pressure cell at GSECARS, classical transmission line and electromagnetic interference problems were encountered:

- 1.) Large spikes occurred on the Johnson noise power spectrum from harmonics of the quasi-sinusoidal heater power supply. A more rudimentary power supply based on a simple autotransformer and an isolation power transformer is under construction.

2.) Ground loops, associated with a weak definition of the press body potential gave rise to other corrupting components in our signal.

3.) Inadequate shielding in the leads emanating from the cell led to additional pick-up.

Most of these issues are specific to the hutch at GSECARS and must be addressed with the assistance of knowledgeable electronics personnel. An effort to bring John Hall to the GSECARS lab in May, 2006 was thwarted by his responsibilities associated with his recently receiving the Nobel Prize. With Ivan Getting's retirement, he will be unavailable again until late fall of 2006 at which time we hope to engage John Hall at GSECARS. In the meantime, an appropriate post-doctoral level person is being identified at GSECARS to carry this project forward. Current expectation is that such a person will be engaged in the project by fall of 2006.



Figure 3. John Hall (right), Ivan Getting (middle) and Yanbin Wang during the high pressure testing of the JNT at U. Colorado, Boulder. Jan., 2006.

We are also in the process of identifying a collaborating electronics engineer at the APS to address noise and ground loop issues. We anticipate solving the ground loop problem during the summer. With the post-doc on board in fall of 2006, we will concentrate on high-pressure temperature measurements. The current 5 mm resistors are adequate for relatively low pressure measurements, which will establish the baseline of

pressure effects. Other types of resistor sensors are being sought with much smaller physical dimensions so that they can be used in cells at pressures of 20 GPa level.

B.2.g Development of CEAD (COMPRES Environment for Automated Data Analysis)

[S. Clark, P. Adams-Lawrence Berkeley National Laboratory, J. Parise-Stony Brook University, M. Rivers-University of Chicago, R. Angel and N. Ross-Virginia Polytechnic Institute and State University]

Funding for 2005-2007: \$170K

The goal of this project is to develop an automated data analysis environment aimed specifically at the needs of the COMPRES community in a code named CEAD (COMPRES Environment for Automated Data analysis). This environment will allow the automation and linking together of existing computer codes. This will allow a massive gain in efficiency of data processing and analysis. It will also allow the direct comparison of data processing strategies and software and enable the rapid development of new data analysis procedures and computer codes. We propose to build on an existing automated crystallographic computing environment, PHENIX, in order to allow rapid and cost-effective development. The resources required for this development are salary for one post doc with programming skills, support for two workshops and travel funds for management meetings and site visits. The duration of the project will be two years. In that time we will: recruit a suitable programmer, hold a community workshop, build the software environment, integrate a range of existing program packages, roll out the software to the COMPRES community with a “hands on” workshop and apply to NSF for funds for the maintenance and the further development of CEAD. The applicability of automated data analysis procedures goes well beyond the COMPRES community. This proposal offers the opportunity for COMPRES to show leadership not only to the high-pressure Earth Science community but also to the wider Earth science and crystallographic communities and beyond.

Background

As the methods and techniques for high-pressure studies using modern synchrotron and neutron sources have matured, the time required for data collection has drastically dropped. The bottleneck for most studies is now in the processing and interpretation of that data. Many computer codes exist to aid in this task. Most experimenters use a number of different programs, some self developed, some shareware or freeware and other proprietary, in the processing and interpretation of their data. These programs usually use different input and output formats and various conversion programs exist to enable data flow between the various units. These systems are highly fragmented and this makes it difficult to add automation to the process, to ensure uniformity of standards and to directly compare specific computer codes or generic algorithms. This situation has not only created great inefficiencies in our community, with students and post-docs spending many months a year processing data that should take only a few hours to

process, but is also restricting the general development of the algorithms and software that we need to fully exploit the power of our data. The current computer codes that we use took many hundreds of man years to develop and rewriting these from scratch is extremely unattractive and may even be counter productive. What we propose here is to build a software environment that will allow us to run these existing codes in a highly automated manner. Such environments have already been developed by the protein crystallography community, such as ccp4i [1], Elves [2], PHENIX [3], and have provided greatly increased efficiency, for example, reducing data processing times from a few months to a few hours allowing data to be processed during data acquisition. This last point is extremely important for us as a community. The ability to determine lattice parameters, unit cell volumes and even atomic positions in real time during an experiment will greatly improve our ability to direct our experiments ensuring that we get the data we need in one visit to the synchrotron or neutron source. Also, the added automation will allow us to subsequently reprocess data without the overhead of many days or weeks stuck in front of a computer screen. This will greatly add to the quality of our measurements and provide added impetus to our scientific progress.

Aims and Methods

The overall goal of this Project is to build a new software system, CEAD, which is capable of routinely and automatically performing data processing and analysis tasks for the COMPRES community. We have assembled a high caliber team of scientists with the appropriate training and experience to deliver this system within the time period of this proposal. Simon Clark is a high-pressure crystallographer and an expert in project management; he will oversee the project management for the work carried out at LBNL and be responsible for the powder diffraction component of CEAD. Paul Adams is the program manager for PHENIX, he will coordinate the development of the basic CEAD package. John Parise is a Professor of Geosciences and Materials Science at the State University of New York, Stony Brook. He is an expert on materials science and x-ray and neutron diffraction. He will be responsible for the neutron data analysis capabilities of CEAD. Mark Rivers is joint head of the GSE-CARS facility at the Advanced Photon Source and an expert in data acquisition and analysis software. He will be responsible for x-ray data processing other than diffraction. Ross Angel is Research Professor of Crystallography at Virginia Tech. He is an expert on high-pressure single-crystal diffraction and has written a number of data collection and reduction software package for single-crystal diffraction that are available to the community via: <http://www.crystal.vt.edu>. He will be responsible for these areas of CEAD for both lab and synchrotron data. We have selected Virginia Tech to be CEAD's academic home. Nancy Ross is Professor of Mineralogy at Virginia Tech and Associate Dean of Research. She is experienced at coordinating developments with in the COMPRES community. She will lead this project from VT and provide overall personnel and financial management. This team of principle investigators is dedicated to establishing an automated system for data analysis and is already fully committed individually.

An extra person is needed to focus on this project and to turn our ideas into a practical reality. Our first step was to hire a postdoc to develop the necessary user interface in

PHENIX working with Paul Adams at LBNL. We will organize a workshop to document the various data analysis tasks performed by COMPRES groups, to list the most appropriate software packages and to prioritize their implementation in the CEAD package. We will then start the implementation process. This process will take about a year to complete. During the second year the programmer will work with COMPRES user groups around the country, starting with major hubs such as the synchrotron and neutron sources, training them in the use of CEAD and helping to set up the necessary strategies for their own particular data analysis requirements. A "training" workshop for the COMPRES community will be held in year 2. As CEAD is distributed to the community at large, there will be a need for support services. The package will be written with ease-of-use as a primary goal, including comprehensive on-line documentation. Despite this, some amount of support will be required. A web-based problem tracking database/bulletin board will be implemented as part of the software distribution web site. This will reduce repeat questions and allow for a significant level of community-based support. The main focus of the Project manpower will be allocated to handle the remaining questions/problems once the Project is released to the COMPRES community.

This project will provide the basis for a larger-scale proposal to NSF for funds to maintain and further develop CEAD. The applicability of automated data analysis procedures goes well beyond the COMPRES community. This proposal offers the opportunity for COMPRES to show leadership not only to the high-pressure Earth Science community but also to the wider Earth science and crystallographic communities and beyond. The schedule for the 2-year project is as follows:

| | |
|---------------|--|
| <i>Year 1</i> | Hire programmer Build basic CEAD package Hold COMPRES community meeting |
| <i>Year 2</i> | Prepare CEAD web site including: documentation, CEAD download and strategies exchange capabilities. Roll out CEAD package with users; "training" workshop Apply to NSF for funds to maintain and expand the basic CEAD package |

Progress through May 2006

The CEAD project was approved by the infrastructure committee of COMPRES to start May 2005. The main resource requirement for this project is a skilled programmer who can put the necessary software together. Finding a person of with the appropriate skills turned out to be very difficult, but after an extensive and exhausting search we managed to fill the post. Jinyuan Yan and he took up the appointment in February 2006. Jinyuan is now embedded in the Computational Crystallography Initiative (CCI) at the Lawrence Berkeley National Laboratory in line with our project plan. On his arrival we reviewed his skill set and identified areas where he needs training or skill strengthening. Members of CCI are providing this training with in the context of producing an identified simple automated data analysis package which will form the basis for development of the full CEAD implementation. Jinyuan has shown great aptitude for this work and is currently over 80% of the way through our planned training schedule and we expect that he will be

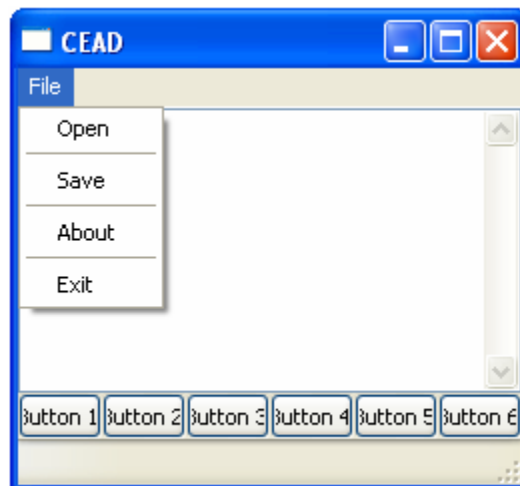
in a position to demonstrate this initial simple analysis package at the COMPRES Snowbird meeting in June 2006. Also, at Snowbird we plan to organize a meeting of the PIs and other interested parties to review progress and to make important decisions on development priorities and roll out strategy. If we assume a two year period of support from Jinyuan's start date then we would confidently predict completion of all or most of our original project goals based upon progress to date.

References

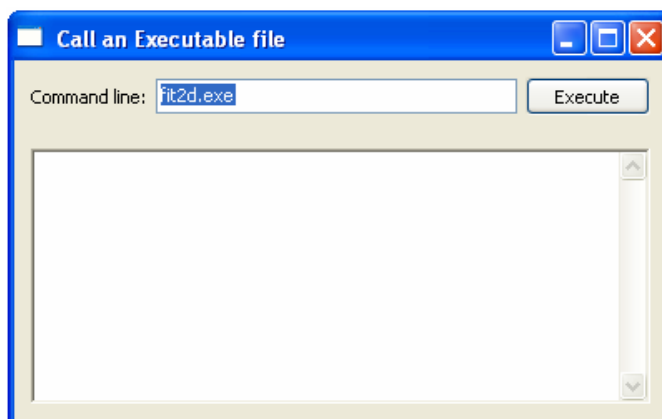
- 1 Collaborative Computational Project, Number 4. **The CCP4 Suite: Programs for Protein Crystallography**. *Acta Cryst.* **D50** 760-763 (1994).
- 2 J. Holton, T.R. Ioerger, J.A. Christopher and J.C. Sacchettini, *Acta Cryst.* **D55** 1309-1319 (2000).
- 3 P.D. Adams, R.W. Grosse-Kunstleve, Li-Wei Hung, T.R. Ioerger, A.J. McCoy, N.W. Moriarty, R.J. Read, J.C. Sacchettini, N.K. Sauter and T.C. Terwilliger, **PHENIX: building new software for automated crystallographic structure determination**, *Acta Cryst.* **D58** 1948-1954 (2002).
- 4 S.R. Hall, D.J. du Boulay and R. Olthof-Hazekamp Eds. **Xtal3.7 System**, University of Western Australia (2000).
- 5 Du Boulay & Olthof-Hazekamp (2000).
- 6 M. Lutz and D. Ascher, **Learning Python**, O'Reilly & Associates, California, USA (1999).

Appendix: Some of the specific milestones achieved so far.

1. Acquire a basic understanding of python and wxpython which underline the PHENIX system. Write and run a small python program to open a text file saved on PC, in the windows operation system, edit the text file and save it to a different directory.



2. Develop a small python program to load the executable program fit2d.exe in the windows operating system, and execute the fit2d program.



3. Learn how to use fit2d and GSAS.
4. Learn the basic infrastructure of PHENIX under UNIX.
5. Produce an automated data stream to take raw 2d diffraction images, process them with fit2d to give 1d diffraction patterns and then process these with GSAS to give lattice parameters that are then plotted in EXCEL.

B.2.h Technical Support for a Dual Beam Focused Ion Milling Facility for TEM

[Harry Green, University of California at Riverside]

Funding for period 2005-2007: \$70K

PROGRESS REPORT ON FOCUSED ION BEAM (FIB) PROJECT

This project proposes to establish a nation-wide facility for preparation of thin foils of experimental Mineral Physics specimens for transmission electron microscopy (TEM) using FIB technology. This technology allows preparation of small foils (of order 15 μ m long X 5 μ m wide X 50nm thick) from specifically chosen areas of any inorganic material. Preliminary studies have shown it to successfully prepare excellent electron-transparent foils of diamond, olivine, pyroxene, majoritic garnet, micas, and other geophysically relevant phases, including those synthesized in and recovered from pressures in excess of 13 GPa. The PI and his colleagues have already published several papers using this technique for preparation of TEM foils both from natural “Ultra-High Pressure” metamorphic rocks and from multianvil experiments. Use of the technique is growing in the multianvil and diamond cell communities as a stand-alone technique and as a powerful supplementary technique to *in situ* synchrotron observations.

This project was funded by COMPRES on a 1-year basis, predicated on the PI successfully obtaining matching monies directly from the EAR IF Program of NSF. A proposal for two years support was submitted to NSF in January, 2005, with a budget request that inadvertently exceeded the maximum award level of the program. After discussions with the Program Director and submission of a revised budget, a three-year award was made at the maximum award level (\$75K) for the program, with a start-date of 1 July 2005. The funds are for support of a technician dedicated to the FIB. The technician will serve both this geophysics program and the nanomaterials program that has purchased the instrument.

Unfortunately, a series of delays have prevent launching of this program. The instrument is still not available on campus because of long over-runs in preparation of the facilities where it will be installed and bureaucratic delays associated with our administration establishing how this unique facility will be organized and run. The delays are frustrating both at UCR and for others in the community who have asked when it will be up and running. It appears that we now have passed the most crucial barrier with the financial office. The process of hiring the technician has been initiated and, with the strong backing of the new Dean of Engineering, I expect to begin operation by the end of summer, 2006. To date, no funds have been expended from either the COMPRES grant or from the NSF grant.

The total funds from COMPRES and NSF are sufficient to support this program for 3 years. Accordingly, no further funding is requested for this program at this time. The period currently funded will serve as a test of whether such a national facility is advantageous to the COMPRES community. I will be discussing with the Executive Committee of COMPRES whether it would be appropriate to put place-holder funding in the renewal budget for Years 4 and 5 of COMRES II, with such additional funding predicated on the recommendation of the Infrastructure Development and Facilities Committees.

B.2.i A gas loading system for diamond anvil cell at Advanced Photon Source

[M. Rivers, G. Shen, V. Prakapenka, University of Chicago]

Funding for period 2005-2007: \$118K

Introduction

With funding from COMPRES, a gas-loading system is under construction at the Advanced Photon Source, which allows for loading diamond-anvil cells (DAC) with various kinds of high-pressure gases, gas mixtures, or liquid-gas mixtures at room temperature and at low temperatures down to $-30\text{ }^{\circ}\text{C}$.

Workshop

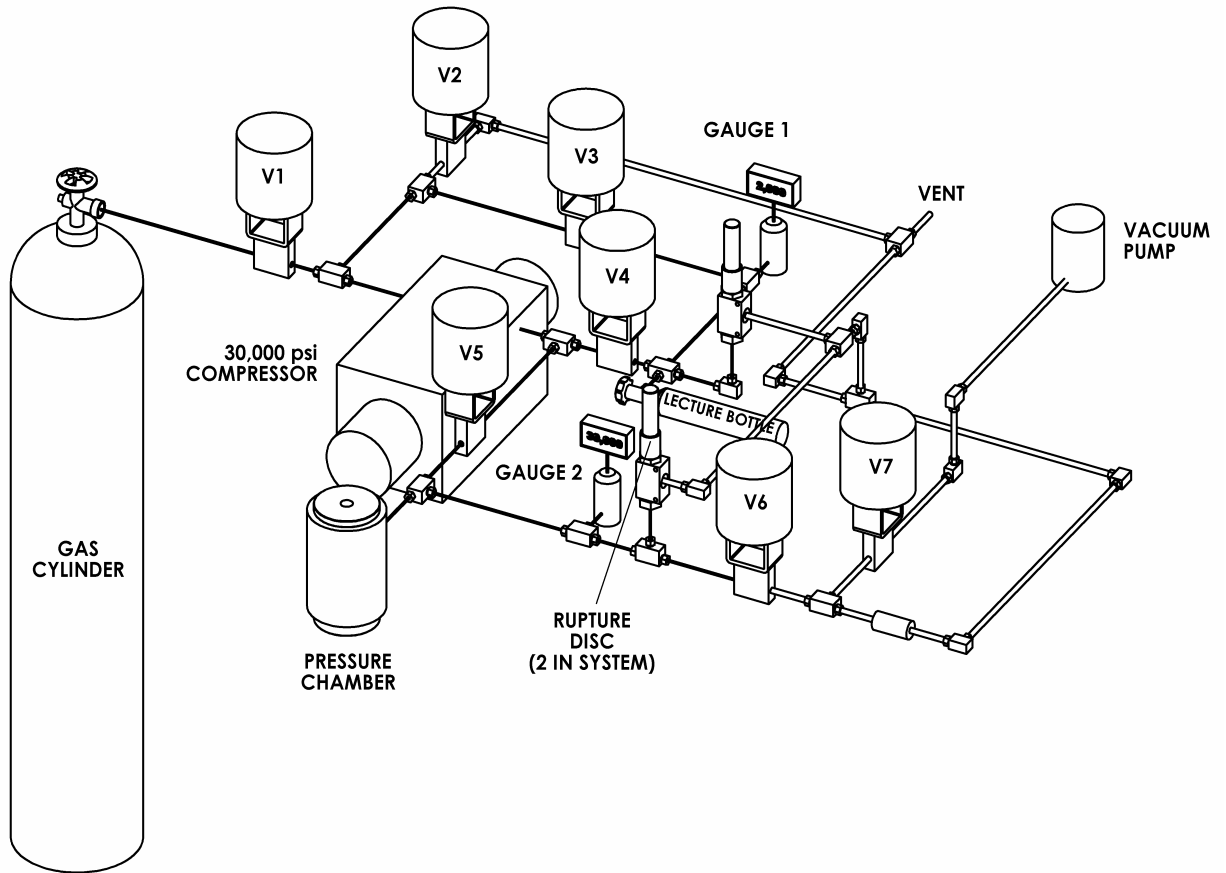
- In order to incorporate input from the community in the design of the system a workshop was held at the APS on April 28, 2006, with 16 specialists from throughout the U. S. attending.

System Design Goals

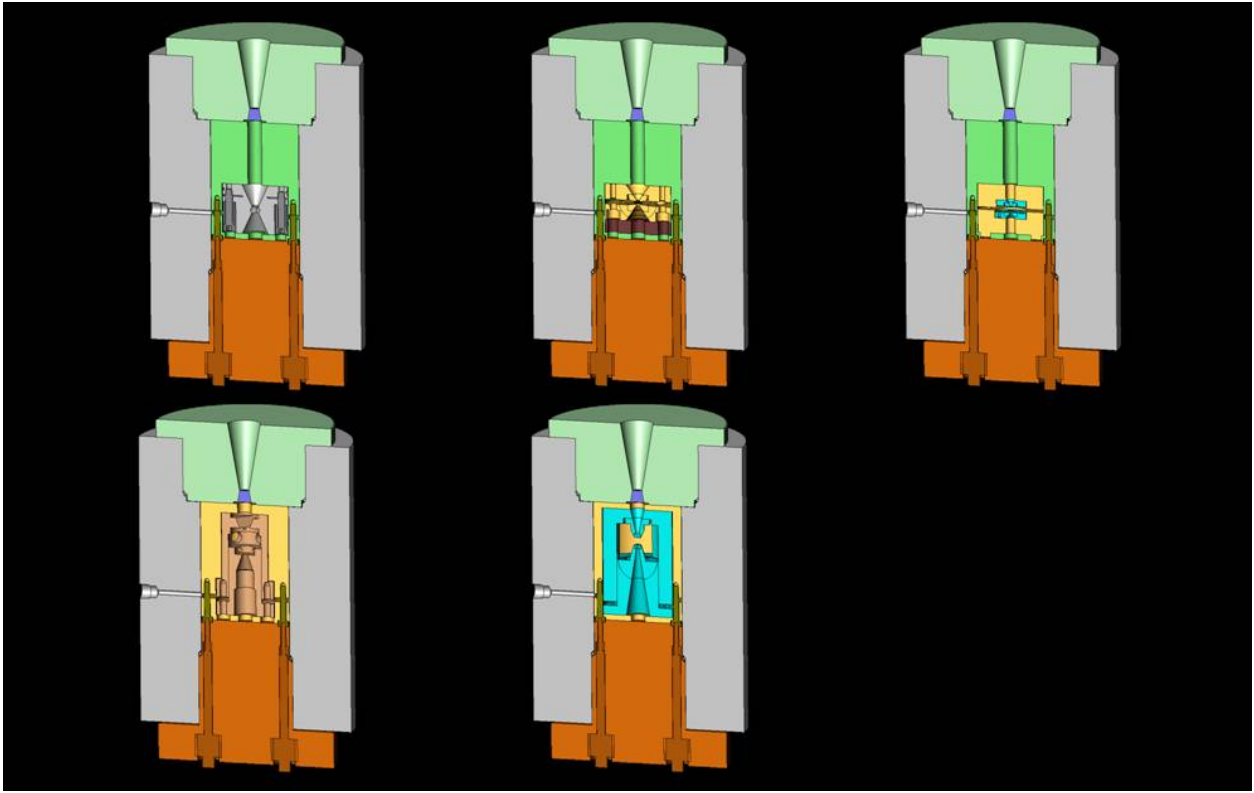
Based on the workshop discussions, the following key system design goals were identified:

- Able to load many kinds of cells
- Closure mechanism (motor driven screws) will close a clamping device, not the cell itself. Easy to add new cell designs, just a different clamp or different spacers
- Optical access (if possible).
- Vacuum pump to clean system before loading
- No electrical parts except pressure transducers in high-pressure enclosure
- Allows flammable gas operation in future
- Easy to safely operate

The schematic design of the system is shown in the following figure.



The design of various cells and cell clamps is shown in the following figure:



Safety Features

The following key safety features are planned:

- We have started with an approved LLNL design
- Many new features to improve ease of use and interlocks for safe operation
- Interlocked heavy-duty enclosure (1/2" aluminum). All high-pressure components contained inside enclosure
- Gas loaded from lecture bottle to limit total mass (energy) that can be compressed in pressure vessel.
 - Protects against user forgetting to load cell or filler parts.
- Pressure meters on both low and high pressure systems which will vent before rupture disk fails
- Gas-operated valves and motor operated drives for remote operation
- PLC for interlock control and computer for routine operations control

The system will include the following interlocks:

- Prevent over-pressuring low-pressure side with lecture bottle
 - Gauge 1 > 1700psi: Vent low pressure side
 - Close V1, V4
 - Open V2, V3
 - Turn off compressor
- Prevent over-pressuring high-pressure side with pressure vessel

- Gauge 2 > 30000psi: Vent high pressure side
 - Open V6
 - Turn off compressor
- Prevent pressurizing more gas than lecture bottle contents
 - V1 open: Isolate compressor output
 - Close V5
- Prevent adding a second lecture bottle contents to already pressurized vessel
 - Gauge 2 > 1700 PSI:
 - Close V1
 -

Computer control will be used to do the following types of routine operations:

- Pressurizing lecture bottle up to 1700psi
- Pressurizing pressure vessel up to 30000psi
- Venting high and low pressure systems
- Pumping high and low pressure systems with vacuum pump

B.2.j Development of a Next Generation Multianvil Module for Megabar Research

[Y.-b. Wang, University of Chicago]

Funding for period: 2006-2007: \$68K

Design Team: Yanbin Wang, Charles E. Leshner, Harry W. Green, II, Yingwei Fei, Guoyin Shen, Carl Agee, William B. Durham, and Murli, H. Manghnani

This component of the COMPRES Infrastructure Development program seeks to construct a new large-volume high pressure module, in order to break the megabar (100 GPa) barrier in large-volume high pressure research, with added flexibility and capabilities. The idea was initiated by interactions with beamline users at GSECARS, and presented at the last COMPRES annual meeting in June, 2005 in New York, where a short workshop was held with more than 30 participants. We proposed to develop a new multianvil module to achieve this goal.

A proposal was submitted to the Infrastructure Development Committee in Nov., 2005 requesting funds for the hardware only. We received 50% funding from COMPRES in Jan. 2006; the other 50% is provided by GSECARS. This report summarizes all the development so far, mostly of which has been supported by GSECARS, as the COMPRES support for hardware was just committed.

The fundamental design concept is based the experience gained during the development of the D-DIA apparatus (Wang et al., 2003), but with much larger first-stage WC anvils capable of compressing a 30×30×30 mm volume (hence the name DDIA-30).

Figure 1a illustrates the mechanism. The 27000 mm³ compressed volume will provide a range of possible high-pressure configurations:

1. A double-stage 6/8 configuration with the second-stage anvils being 14 mm sintered diamond cubes. This configuration is the standard ultra-high pressure module in Japan (e.g., Ito et al., 2001) and has been used to generate up to 62 GPa (Ito et al., 2005) at SPring-8 synchrotron beamline BL04B1.

2. A double-stage 6/2 configuration with the second-stage anvils being Drickamer- or diamond-anvil type. With large (10 mm or more in diameter) second-stage anvils, we expect this configuration to generate megabar pressures at high temperature. This configuration has been tested by various groups using a smaller DIA apparatus and a maximum of 90 GPa has been reached (e.g., Utsumi et al., 1986; Li et al., 2001).

With newly developed diffraction techniques, these two configurations will make it possible to conduct structural refinements at P-T conditions corresponding to the deep lower mantle.

3. The deformation capability of the DDIA-30 will allow us to carry out deformation experiments on large samples for stress-strain curve measurements as well as acoustic emission studies.

This DDIA-30 module will be compressed by the 1000 ton press installed at the 13-ID-D beamline with white or monochromatic radiation as needed. This device will allow us to conduct routine experiments at pressure and temperature conditions corresponding

to hydrostatic pressures over the entire lower mantle and into the core. Flow and faulting properties will be conducted well into the Earth's lower mantle.

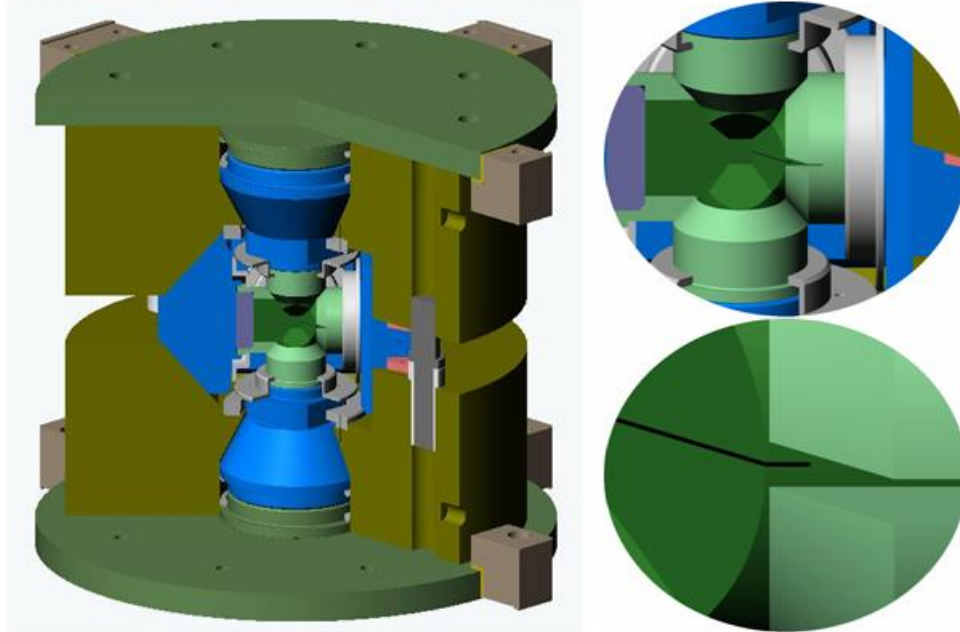


Figure 1. A schematic illustration of the DDIA-30. Horizontal overall dimension 500 mm; vertical height 550 mm. The first-stage DDIA anvils have a maximum of 30 mm truncation, capable of compressing a set of eight 14 mm edge length sintered diamond anvils for 6-8 pressure generation configuration. The top and bottom anvils have built-in differential rams, which drive the two anvils independently from the main hydraulic ram of the 1000 ton press. X-ray access is made into the wedges and anvils for diffraction in the horizontal plane. These slots can be made in semi-conical shape to allow more diffraction access.

To test and demonstrate the ultra-high pressure capability using the 6/2 configuration, we have conducted experiments in the existing D-DIA using 6 mm first-stage anvils (DDIA-6). By introducing a pair of sintered diamond anvils (5 mm diameter with 1 mm truncation - see Figure 2), we were able to reach 30 GPa when compressing the cubic cell assembly isotropically to 40 tons. Then we advanced the deformation pistons to drive the sintered diamond anvils under constant confining load, to generate up to ~ 50 GPa. This test demonstrates the feasibility of the proposed technique.

Currently, engineering drawings are being done by GSECARS drafting personnel, based on the smaller D-DIA. The co-investigators have planned to meet during the COMPRES annual meeting in June of 2006 to discuss engineering details. Requests for quotation will be sent out as soon as the design is finalized. We expect to have the module built in early 2007; then a series of testing will begin.

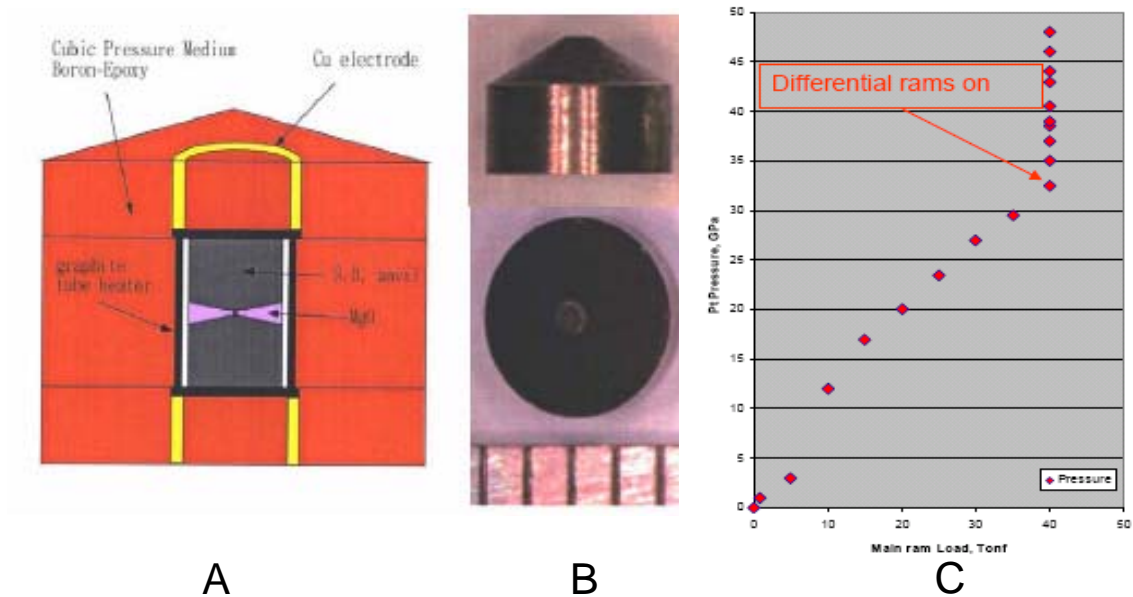


Figure 2. Cell assembly (A) used for the test in DDIA-6 using sintered diamond anvils (B). The sample was a Pt rod, 0.25 mm in diameter and 0.25 mm in height. The pressure generation curve is shown in (C).

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B.2.k Calorimetry-on-a-Chip

[A. Navrotsky, University of California at Davis, F. Hellman, University of California at Berkeley]

Funding for period 2006-2007: \$50K

Knowledge of heat capacities and standard entropies of mantle minerals is necessary for thermodynamic modeling of high P-T equilibria. However, many of these materials can only be prepared in milligram quantities in a multianvil apparatus or in microgram quantities in a diamond anvil cell. This eliminates traditional adiabatic calorimetry techniques for C_p measurements. Hellman's microcalorimeters¹ (Figure 1) have been used to successfully measure thin films², multilayers³, and magnetic single crystals⁴ in a magnetic field⁵ (when applicable) and in a wide temperature range⁶. Using these versatile "calorimeters on a chip", we have measured the heat capacity of the Fe_2SiO_4 olivine and spinel polymorphs from 2 K to room temperature. We have also measured a CoO single crystal to verify the feasibility of our measurement for materials other than thin films at the microgram scale.

We obtained a single crystal of cobalt oxide that had already been measured by our collaborators at BYU. This provides a base case for comparison. The 638 μg sample was attached to one of our thick nitride devices (for measuring large heat capacities) with $\sim 100 \mu\text{g}$ of silver paint. The antiferromagnetic transition is quite well-defined (Figure 2) and agrees with the adiabatic measurement at BYU to within $\sim 1\text{K}$. This difference is within the precision of our Cernox thermometer. The $\sim 8\text{-}10\%$ discrepancy at high temperature is related to the inaccuracy of our silver paint heat capacity addenda correction, which is being refined.

Having shown the viability of our technique on microgram quantities of material, we turned to measuring the heat capacity of the olivine polymorph of Fe_2SiO_4 , fayalite. Attaching the 933 μg sample obtained from the Oak Ridge National Laboratory to a thick nitride device, we observed the magnetic transition in fayalite (Figure 3). Again there is a high temperature discrepancy due to the silver paint. In focusing on the nature of the transition, though, it appears to be slightly suppressed from that of the bulk⁷. After structural analysis at UC Davis, it was discovered that the sample obtained had a significant amount of magnetite (Fe_3O_4) contaminant. This accounts for the slight suppression of the magnetic transition.

The UC Davis group synthesized a pure, quenched spinel Fe_2SiO_4 powder in the multianvil apparatus in Leshner's lab, using olivine starting materials prepared by Don Lindsley at Stony Brook, which is more pure and magnetite free than the Oak Ridge material. We mounted a 24.4 μg grain of this powder onto one of our thick nitride devices. However, because of the small sample size, the net heat capacity of our sample was only $\sim 16\%$ of the total heat capacity measured. This resulted in somewhat noisy data, especially at low temperatures. However, it is clear there is no evidence of any

magnetic phase transition in the spinel sample according to this preliminary data (Figure 4). However, quantifying this result is premature and additional measurements are planned

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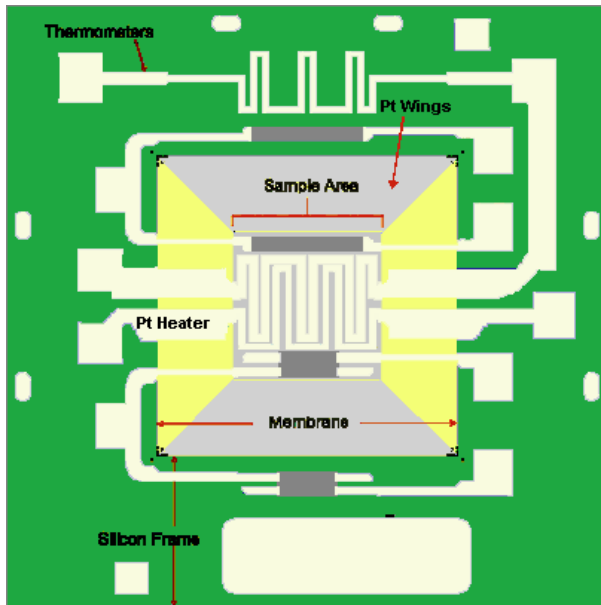


Figure 1. A diagram of our lithographically patterned, silicon-based microcalorimeter.

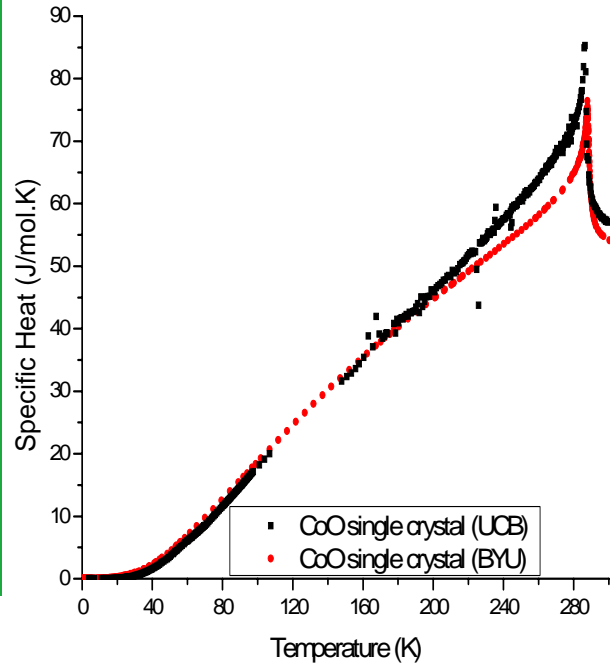


Figure 2. Heat capacity of a CoO single crystal as measured by our technique compared to that measured by our collaborators.

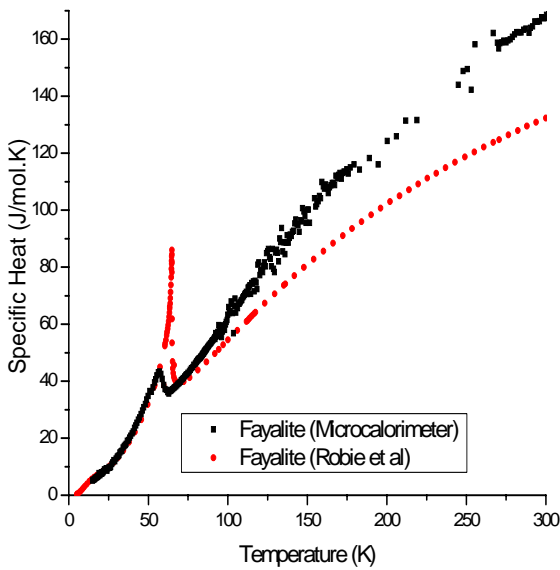


Figure 3. Measurement of a fayalite sample obtained from Oak Ridge National Lab and measured by our technique as compared to bulk.

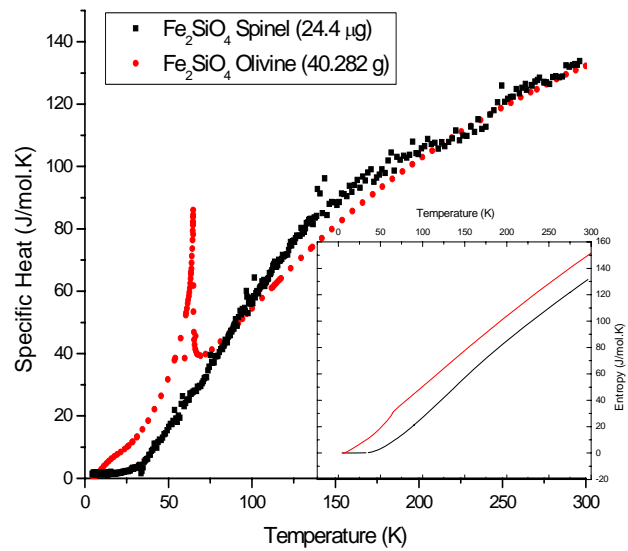


Figure 4. Preliminary results of the specific heat of the spinel polymorph of Fe_2SiO_4 compared to the olivine phase. Entropy information is shown on inset.

B.2.1 Monochromatic x-ray side-station at the X17B2 beam line of the NSLS

[Jiuhua Chen-Stony Brook University]

Funding for period 2006-2007: \$99K

This project is to develop and install a portable multi-anvil high pressure apparatus, deformation-Tcup (D-Tcup), at the monochromatic x-ray side-station at the existing multi-anvil high pressure beamline X17B2 of the NSLS.

COMPRES infrastructures at synchrotron x-ray sources have been playing a critical role in advancing the frontier of our scientific researches. As more and more scientists in the community begin to take the advantage of these infrastructures to enhance their scientific programs, limitation of the beam time available to high pressure research becomes a major issue of COMPRES infrastructure facilities. The monochromatic side-station operates simultaneously with the main multi-anvil station, and therefore increases the beam time for high pressure research by a factor of two.

The concept of the monochromatic side-station is to install a single bounced monochromator in the white x-ray beam at the beam entrance of the B2 hutch (Figure 1). This monochromator sends a side beam at a 2° angle to the white beam. A full time monochromatic station running simultaneously with the white beam system is therefore possible.

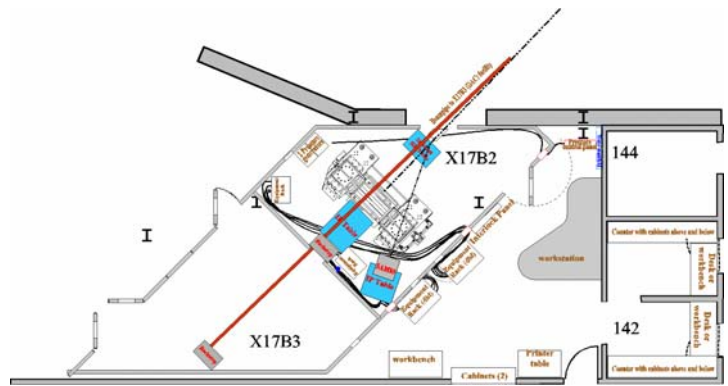


Figure 1. Floor plan of X17B2 and B3 hutches

Design of the monochromator is shown in Figure 2. A silicon single crystal is bent sagittally in one dimension which generates an anticlastic meridional bending, yielding a 2-D focusing.

A detector/press combo stage (see Figure 3), designed and manufactured by Advanced Design Consulting USA, Inc. is installed next to the existing main multi-anvil station in the X17B2 hutch. Figure 3 also shows an on-line imaging plate detector MAR345 installed on the stage.

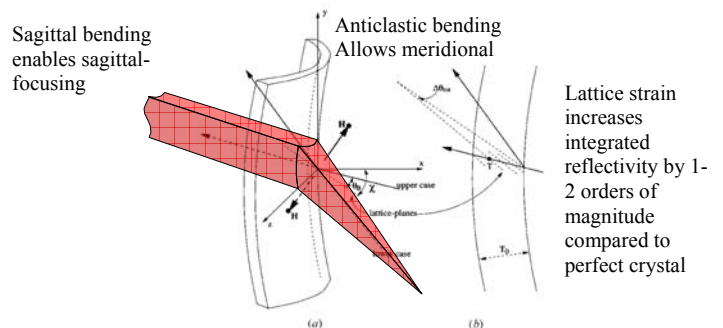


Figure 2. Sagittally bent 2-D focusing monochromator

Test experiments were conducted for measuring stress field in cylindrical symmetry and shear configuration deformation cells using a D-DIA pressure module of main station.

With the support of this COMPRES Infrastructure Project, we plan to develop and install a deformation-Tcup (D-Tcup) apparatus at the side-station. Design of the D-Tcup has been finished under the collaboration of Stony Brook (Don Weidner) and University College London (David Dobson). A modified Tcup module with an independent driving piston for each of the two corner second-stage anvils is equipped in a customized Paris-Edinburg (P-E) press. Two additional rams are added to the original P-E press design, serving as differential ram (similar to the differential rams in D-DIA). As illustrated in Figure 4, main ram of the P-E press delivers the force for generating confining pressure to the sample. At high pressures, the differential rams can be driven to advance the two corner second-stage anvils for deforming the sample. This apparatus is expected to conduct deformation experiment at pressure up to 20 GPa, and therefore nearly double the current maximum pressure of *in situ* deformation studies (~10 GPa with D-DIA). In addition, this new apparatus is as portable as the original P-E press which opens the possibility for transporting the press to some other beamlines for different measurements. Leaving the differential rams at home position, the D-Tcup can operate as a regular Tcup.

The D-Tcup is expected to be delivered to the beamline before the end of 2006 for installation, and we anticipate to have the press operated at the monochromatic station by April 2007 for experiments. The D-Tcup will not only operate for deformation experiment, but also serve as a major high pressure apparatus for general non-deformation studies.

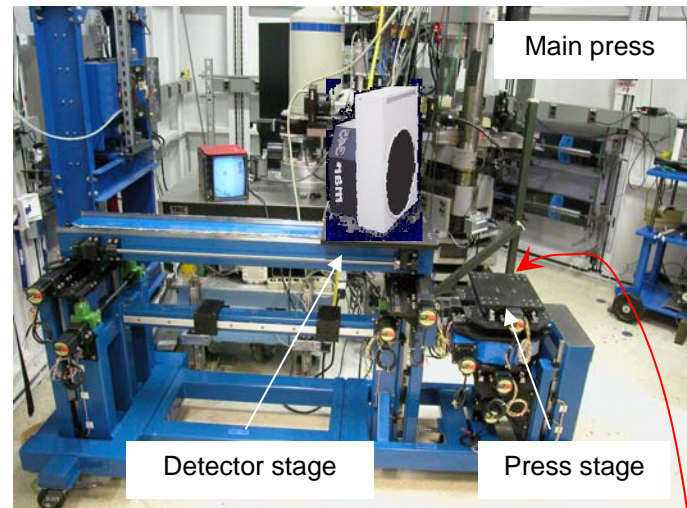


Figure 3. Mar345 detector and side-station x-y-z table in the X17B2 hutch of the NSLS

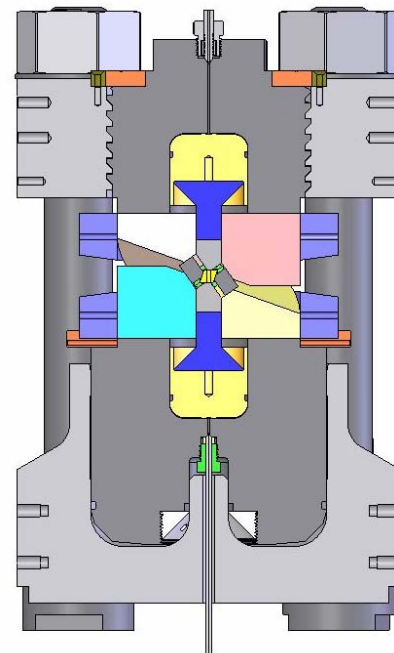


Figure 4. Design of the Deformation-Tcup: a modified Tcup module in a customized Paris-Edinburg press.

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